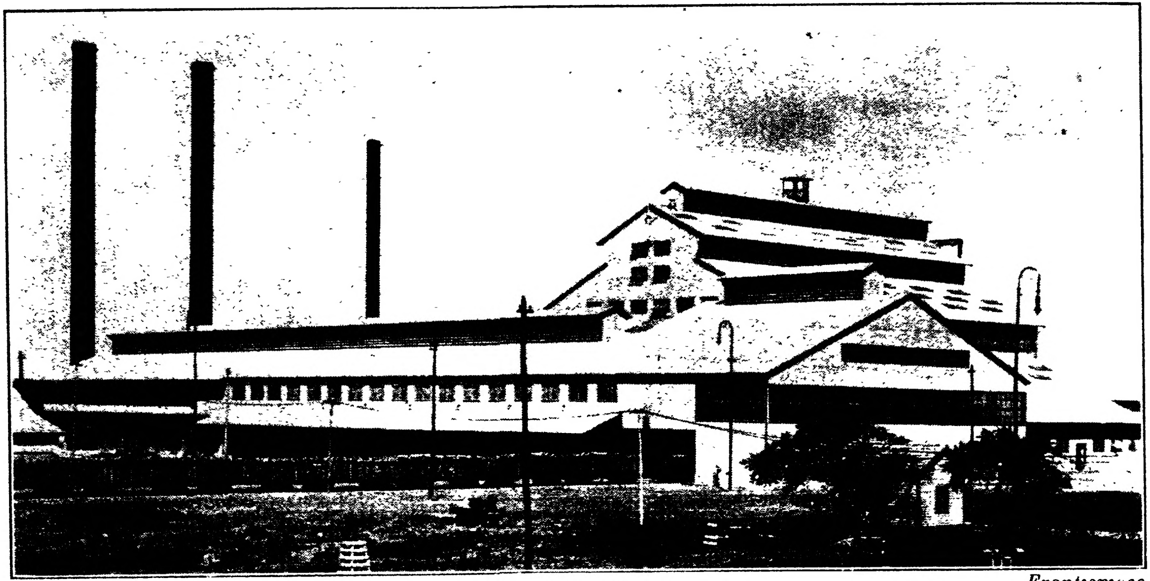

Frontispiece

Central Delicias, Cuba.

CANE SUGAR HANDBOOK

A Manual for Cane Sugar Manufacturers and Their Chemists

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PREFACE TO THE EIGHTH EDITION

The purpose of this book continues to be fourfold: (1) to afford a concise survey of the manufacturing and refining processes; (2) to provide selected analytical procedures and reference tables for the control chemist; (3) to explain and compare methods of chemical control; and (4) to indicate, by references, additional sources of information.

In this second revision by the present writer the scope of the book has been extended as in all previous editions. However, the publications relating to sugar processes have increased to such an extent that the treatment requires careful selection and drastic condensing. Chapter 6 on clarification has again been almost entirely rewritten to cover the many studies on this subject during the past fifteen years. The section on the carbonation process in this chapter is by Dr. Peter Honig, former Director of the Java Experiment Station. Evaporation and Heating, covered in previous editions by the late Professor W. H. P. Creighton of Tulane University, has been replaced by Chapter 9 by Alfred L. Webre, internationally recognized specialist on this subject. Mr. Webre also collaborated in the preparation of Chapter 11 on pan boiling, which has been greatly expanded. A new chapter on the theory and practice of crystallization in motion (Chapter 12) is largely by J. G. Davies, Sugar Technologist, of the Imperial College of Tropical Agriculture, Trinidad, B. W. I. Chapter 14 on blackstrap molasses and edible sirups reviews the chemistry and technology of these by-products. Methods of Cane Purchase (Chapter 37) has been rewritten from a different viewpoint to include the plans of payment in various cane-producing countries. Other subjects in the first part of the book which have been given greatly increased space are composition of cane and its products (Chapter 2), industrial uses of bagasse (Chapter 3), warehousing of sugar (Chapter 13), and vegetable carbon processes in refining (Chapter 16). Chapter 38 on Fermentation by William Ludwell Owen has been revised by him to include recent work on microorganisms in refined sugars.

The portion on analytical procedures has been revised to conform with the latest recommendations of the International Commission for Uniform Methods of Sugar Analysis, the International Society of Sugar Cane Technologists, the U.S. Bureau of Standards, and other recognized authorities. The Examination of Refined Sugars has been given separate attention in a new chapter (Chapter 30). Conductivity methods for ash determination (Chapter 22) and potentiometric methods for pH control (Chapter 23) are treated at much greater length than in the previous edition, and a section on Turbidity has been added to Chapter 24 on Color Determination.

The recommended terminology and methods of the International Society of Sugar Cane Technologists have been explained in Chapters 34 and 35 on Definitions and Chemical Control, respectively. Available sugar formulas are also discussed in detail in connection with these recommendations. The recommendations are based largely on Eastern Hemisphere practice, and it has therefore been necessary to include the older terms and methods in use in Louisiana and the West Indies and to explain and clarify the differences, advantages, and disadvantages of the two systems.

Reference tables based on the old standard temperature of 17½° C. have been discarded and others for 27½° C., the standard used in certain tropical countries, have been added. Revision of other tables to conform to the normal weight of 26 grams (instead of the older 26.048), the exclusion of obsolete material, and the addition of many new tabulations bring this section up to date.

This revision has been made possible through the kindness of friends in the sugar industry who have given freely of their time and thought whenever asked to do so. Acknowledgments for specific aid have been made in the text. Special thanks are due Dr. F. W. Zerban for his ever-ready and valuable assistance and suggestions; also to Henry G. Gerstner, Assistant Superintendent, Gramercy Refinery, for calculations and proofreading; and to Stark L. Davis and Henry D. St. Martin, Engineering Department, Gramercy Refinery, for charts and drawings.

George P. Meade

Gramercy, Louisiana
July, 1944

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MANUFACTURE OF RAW SUGAR

CHAPTER 1

ECONOMIC PHASES OF THE SUGAR INDUSTRY

1. Historical. Sugar cane is generally believed to have originated in northern India, the earliest mention of it being in some of the legends concerning Buddha about the fourth century B.C. There was no field culture and its use for several centuries was restricted to chewing the cane and drinking the juice. From India the planting of cane probably spread first into China. Neither sugar nor sugar cane is referred to in the Talmud or the Koran. Two references in the Bible to "sweet cane" (Jeremiah, vi:20, and Isaiah, xliii:24) very probably refer to sugar cane, but some authorities believe this "sweet cane" to be a perfumed grass or reed common in Palestine.

The earliest positive evidence of sugar in solid form seems to date from about A.D. 500 in Persia. The original Persian name for white sugar was *kandi-sefid* from which comes our word candy. *Shekar,* or *Shakar,* the East Indian word for sugar, shows the origin of our term.

Commercial manufacture and refining developed in Egypt during the ninth and tenth centuries, and the exportation of sugar was an important part of that country's commerce. The culture of the sugar cane had been spread by the Arabs through northern Africa and southern Europe at the same time that it was being carried into Java and the Philippines by the Chinese. The Crusaders brought sugar back to France in the eleventh century, after which time the commercial development and use became widespread in Europe.

During the middle ages sugar was a delicacy of so great cost that only the nobility used it. Marvelous curative powers were ascribed to its use. A study of the medieval medicinal formulas shows sugar as an ingredient of the majority of them, not for its sweetening properties but for the miraculous health-giving properties which it was then thought to possess.

Columbus introduced sugar cane into Santo Domingo on his second voyage in 1494, and from there it was carried to Cuba and other West Indian, Central and South American sections. By 1600 the production of raw sugar from cane grown in tropical America was the greatest industry in the world. The cultivation in Louisiana dates from 1750.

Sugar refineries were built in Germany, France, and England in the sixteenth century, but modern cane sugar refining dates from the early part of the nineteenth century in England.

The first commercial beet sugar was made about the middle of the eighteenth century, the culture and manufacture being largely developed through French initiative toward the end of that century. This branch of the

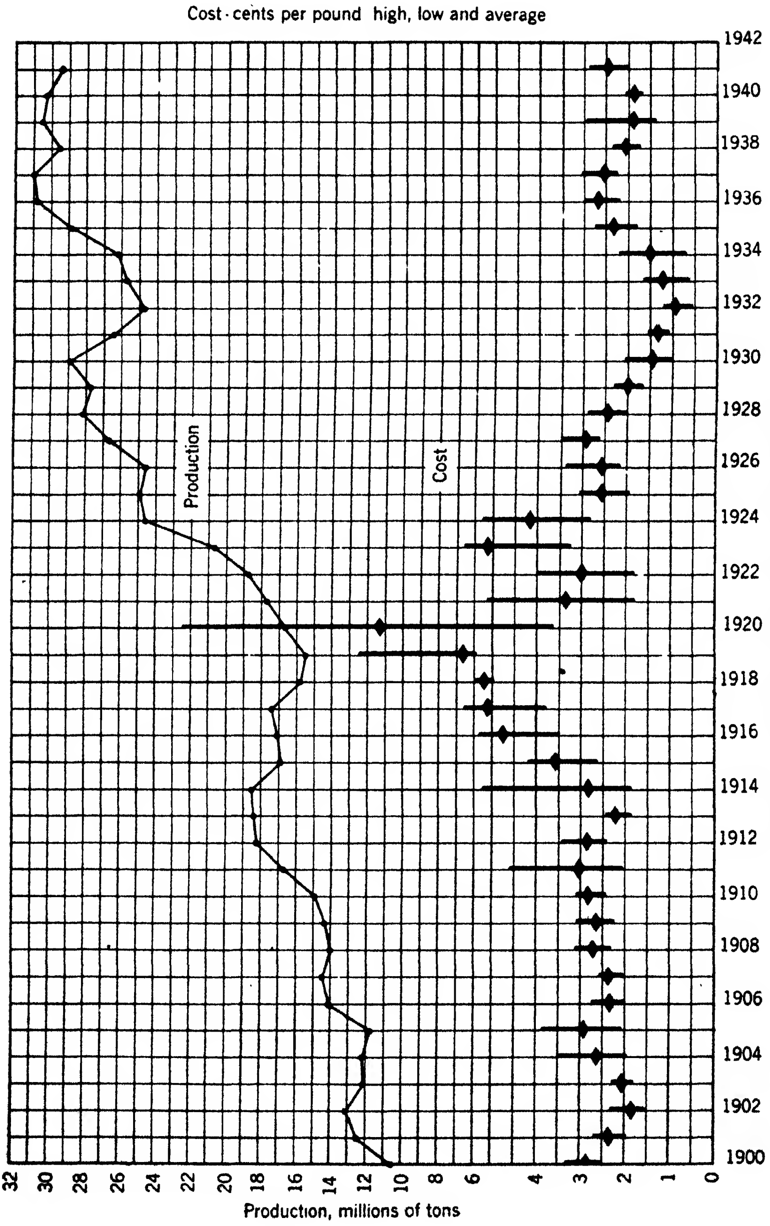

FIG. 1. Sugar Production and Price Range.
Note that divisions up to 5 are double those above 5.

WORLD'S SUGAR PRODUCTION

(Willett and Gray)

	Tons of 2,240 Lb.		
Cane Sugar	1940–41	1927–28	1913–14
Cuba	2,440,990	4,011,717	2,597,732
United States			
Florida	86,607	....	....
Louisiana	210,229	63,207	268,337
Puerto Rico	832,140	670,831	325,021
Hawaiian Islands	845,705	807,180	550,925
Virgin Islands, W. I.	6,853	10,562	5,800
Dominican Republic	400,711	368,196	105,778
Haiti	31,466	16,367	....
British West Indies	428,803	243,266	136,257
French West Indies	121,666	74,355	66,298
Mexico	295,259	175,214	127,944
Central America	104,650	104,845	22,000
Argentina	540,591	421,601	280,319
Brazil	1,172,864	832,931	158,038
Peru	431,055	370,724	228,054
Other South America	300,432	171,868	128,594
Total in America	8,250,021	8,342,864	5,001,097
British India	5,016,884	3,216,000	2,291,500
Java	1,678,107	2,939,164	1,272,417
Japan	980,544	692,932	157,050
Philippines	1,028,354	622,704	232,761
Total in Asia	8,703,889	7,470,800	3,953,728
Egypt, Mauritius, Reunion, Natal, and Mozambique	8,445,012	656,360	480,956
Australia	1,183,827	493,049	266,267
Fiji Islands	808,817	95,114	92,112
Spain	118,463	10,552	7,376
Total Cane Sugar	19,075,317	17,068,739	9,801,536
Beet Sugar			
United States	1,580,545	965,241	655,298
Canada	95,350	27,212	11,675
Germany	2,207,143	1,665,450	2,617,937
Czecho-Slovakia	644,289	1,253,163	....
Russia and Ukraine	2,700,000	1,501,986	1,687,799
France	461,516	863,206	717,400
Poland	517,857	566,515	....
Holland	292,950	259,966	229,257
Italy	536,607	284,276	304,977
Spain	165,773	259,964	169,355
Belgium	255,543	273,113	229,049
Hungary	176,785	187,600	....
Roumania	126,434	139,522	34,758
Gt. Britain and Eire	574,864	208,114	....
Denmark	249,355	142,800	145,700
Sweden	304,171	145,335	137,082
Austria	....	110,020	1,682,678
Other countries	348,831	170,844	11,977
Total in Europe	9,562,118	8,031,874	7,967,969
Total Beet Sugar	11,238,013	9,024,327	8,634,942
Grand Total—Cane and beet sugar	30,313,330	26,093,066	18,436,478

sugar industry extended to the United States about 1835. A review of the history of sugar machinery and processes is given on p. 72.

2. **World Production.** The total sugar produced in the world (both cane and beet) has about trebled in the last forty years (see Fig. 1). The production for representative years of that period is shown in the table on p. 3 (Willett and Gray).

3. **Price Range.** The maximum, minimum, and average yearly price of Cuban raws, c and f basis, i.e., cost and freight paid to New York but duty unpaid, for the past forty years is shown in the chart, Fig. 1. It is to be noted that the divisions on the chart up to 5 are twice as great as those beyond 5. The length of the vertical bar for each year represents the price variation, and the diamond the average price. The duty paid price or actual cost price paid by the refiners for raws may be obtained by adding the amount of the duty from Cuba in effect at the time as given in the tabulation in the following paragraph.

4. **United States Sugar Tariff.**[1] The following table shows the duties which have been effective since 1789 upon sugar entering the United States.

	FROM CUBA c. per lb.	OTHER FOREIGN COUNTRIES c. per lb.
July 5, 1789 to Aug. 11, 1790	1.000	1.000
Aug. 11, 1790 to May 14, 1800	1.500	1.500
May 14, 1800 to April 28, 1816	2.000	2.000
April 28, 1816 to July 15, 1832	3.000	3.000
July 15, 1832 to July 31, 1846	2.500	2.500
July 31, 1846 to March 3, 1861	30% Ad. Val.	30% Ad. Val.
March 3, 1861 to Aug. 6, 1861	0.750	0.750
Aug. 6, 1861 to July 15, 1862	2.500	2.500
July 15, 1862 to July 1, 1864	3.500	3.500
July 1, 1864 to July 15, 1870	4.000	4.000
July 15, 1870 to March 4, 1883	2.750	2.750
March 4, 1883 to Oct. 2, 1890	2.240	2.240
Oct. 2, 1890 to Aug. 28, 1894	Free	Free
Aug. 28, 1894 to July 25, 1897	40% Ad. Val.	40% Ad. Val.
July 25, 1897 to Dec. 27, 1903	1.685	1.685
Dec. 27, 1903 to March 1, 1914	1.348	1.685
March 1, 1914 to May 27, 1921	1.0048	1.256
May 27, 1921 to Sept. 22, 1922	1.600	2.000
Sept. 22, 1922 to June 17, 1930	1.7648	2.206
June 18, 1930 to June 7, 1934	2.000	2.500
June 8, 1934 to Sept. 3, 1934	1.50	1.875
Sept. 4, 1934 to Sept. 11, 1939	0.90	1.875
Sept. 12, 1939 to Dec. 26, 1939	1.50	1.875
Dec. 27, 1939 to Jan. 4, 1942	0.90	1.875
Jan. 5, 1942 to July, 1943	0.75	1.875

---

[1] Farr, *Manual of Sugar Companies*, 1942.

The variations in the present tariff (July, 1943) according to test are shown below.

ON SUGAR TESTING	FROM CUBA	FULL DUTY
	Cents per Pound	
100° *	0.795 *	1.9875
99°	0.78375	1.959375
98°	0.7725	1.93125
97°	0.76125	1.903125
96°	0.75	1.875
95°	0.73875	1.846875
94°	0.7275	1.81875
93°	0.71625	1.790625
92°	0.705	1.7625

* Refined sugar.

The table shows that both the Cuban duty and the full-duty rates increase or decrease by 1.50 per cent per degree above and below the 96° standard. Because of reciprocal trade treaties some countries besides Cuba have the benefit of tariffs lower than the full-duty rates.

5. Price Adjustment for Test. The Sugar Institute, Inc., adopted the following allowances in March, 1938, for the calculation of the price of raws purchased on a basis of 96° Test Cubas c & f New York.

96° to 97°	Add 1.50 per cent of the basis price.
97° to 98°	Add an additional 1.25 per cent of basis price.
96° to 95°	Deduct 1.60 per cent of the basis price.
95° to 94°	Deduct an additional 2.00 per cent of basis price.
94° to 93°	Deduct an additional 2.50 per cent of basis price.

Fractions of a degree in proportion.

No additional allowance is given above 98°. No sugar is to be delivered below 93° unless on terms mutually satisfactory to consignee and seller.

PRICE ADJUSTMENT ON 97.2 POLARIZATION CUBAN SUGAR PURCHASED ON BASIS 2.98 CENTS COST AND FREIGHT FOR 96°

	Cents
Basis 96° c & f	2.98000
Add for 96° to 97°, 1½ per cent	0.04470
Add for 97° to 97.2°, $\frac{2}{10}$ of 1¼ per cent	0.00745
Adjusted price c & f	3.03215
Add duty at 96°	0.75000
Add duty increment 96° to 97.2°	0.01350
Add insurance *	0.0200
	3.81565

* Insurance varies for different parts and rates may change rapidly owing to war conditions.

PRICE ADJUSTMENT ON 97.2 POLARIZATION PUERTO RICAN SUGAR PURCHASED ON BASIS 3.75 CENTS C.I.F. (COST, INSURANCE, FREIGHT) FOR 96°

	Cents
Basis 96° C.I.F.	3.75000
Deduct	0.77000
Equivalent cost and freight Cubas	2.98000
Add for 96° to 97°, 1½ per cent	0.04470
Add for 97° to 97.2°, $\frac{2}{10}$ of 1¼ per cent	0.00745
Adjusted equivalent, c & f	3.03215
Add duty at 96° and insurance *	0.77000
Add duty increment, 96° to 97.2°	0.01350
	3.81565

* Insurance varies for different ports and rates may change rapidly owing to war conditions.

6. Calculation of Raw Value. For tax and quota purposes the United States Treasury Department requires the calculation of all sugar to "raw value," which is defined in TD4441 as follows:

> The term raw value means a standard unit of sugar testing 96 sugar degrees by the polariscope. All taxes shall be imposed and all quotas shall be established in terms of raw values, and for the purposes of quota and tax measurements all sugars shall be translated into terms of raw value.

The Treasury regulations for the calculation of raw value are arbitrarily set up as follows:

> In order to obtain 100 pounds of refined sugar tested by the polariscope 99.8 sugar degrees and above, it is necessary to use 107 pounds of sugar raw value, i.e., sugar tested by the polariscope 96 sugar degrees, and the raw value of 1 pound of refined sugar tested by the polariscope 99.8 sugar degrees or above is, therefore, 1.07 pounds. The pounds of sugar raw value to be added for each degree (and fraction of a degree in proportion) of polariscope, above 96 degrees to 100 degrees, is to be determined by the formula 1.07 minus 1.00 over 100 minus 96 and is 0.0175 lbs.
>
> The most accurate method for translating any quantity of sugar tested by the polariscope less than 96 degrees in the terms of raw value is to find what weight of sugar raw value will have the same weight of total sugar content as such quantity of sugar. The total sugar content per pound of 96 degrees sugar (i.e., raw value sugar) is 0.972 pound. The raw value of any sugar testing less than 96 degrees by the polariscope is to be determined by dividing the number of pounds of the total sugar content thereof by 0.972 pound. [The "total sugar content" of sugars polarizing less than 96 is the sucrose (Clerget) plus the reducing sugars.]

7. United States Tariff on Blackstrap Molasses. The Tariff Act of 1930, paragraph 502, says, "Molasses not imported to be commercially used for the extraction of sugar or for human consumption, three one-hundredths of 1 cent per pound of total sugars." The Cuban preferential of 20 per cent is deducted from this amount for importations from that country. For the purposes of these regulations, blackstrap molasses is defined as "final molasses practically free from sugar crystals, containing not over 58 per cent of total sugars and having a ratio of

$$\frac{\text{Total sugars} \times 100}{\text{Brix}}$$

not in excess of 71. In the event of doubt, an ash determination may be made; an ash content of not less than 7 per cent indicates a blackstrap molasses within the meaning of these regulations."

8. Duties on Raw Sugars into Great Britain. Duties on raw sugars entering Great Britain depend upon the country of origin. Full-duty sugars are those imported from outside the British Empire; those from the Empire but not from the Colonies enjoy a preferential; and those produced in the Colonies enjoy a still further preferential as shown below. (Customs and Excise Act of 1941.)

POLARIZATION	FULL DUTY (Per cwt.)		PREFERENTIAL EMPIRE (Per cwt.)		PREFERENTIAL CERTIFIED COLONIAL (Per cwt.)	
	Shillings	Pence	Shillings	Pence	Shillings	Pence
99–100	23	4.0	17	6.0	14	0.7
98–99	23	4.0	15	9.2	12	6.3
97–98	19	3.8	15	4.5	12	2.6
96–97	18	9.7	14	11.7	11	10.7
95–96	18	3.7	14	6.9	11	6.9
94–95	17	9.6	14	2.0	11	3.1
93–94	17	3.6	13	9.3	10	11.3
92–93	16	9.6	13	4.5	10	7.5

9. Sugar as a Food. Cane sugar is a natural product occurring in the juices of various plants and fruits as a result of the action of sunlight on carbon dioxide and water by the process known as photosynthesis. The elaborate processes of manufacture and refining described in the later chapters do not change the chemical character of the sugar itself. The crystalline product on the market is the same sugar that exists in the juices of the cane.

Refined sugar is practically a chemically pure organic compound, sucrose, having the chemical formula $C_{12}H_{22}O_{11}$, i.e., it is a combination of twelve molecular weights of carbon and eleven molecular weights of water. It is therefore one of the groups of compounds known as the carbohydrates. The so-called impurities in a high-grade refined sugar are in such small proportions as to be negligible for practical purposes. (See Chapter 30.)

The carbohydrates are the energy producers in the diet, and sugar rates high in this respect. It is literally burned up in the body processes to produce this energy. Its value for athletes, soldiers, workers, growing children, and others leading an active life is therefore evident.

Lyle[2] discusses the comparative value of sucrose and dextrose, and the digestion, storage, and use of sugars in the body. He states that the levulose present, either in invert sugar or in sucrose after it has been inverted by the digestive juices, "is much superior to dextrose in relieving the symptoms that supervene after a period of starvation or prolonged muscular effort."

The extreme purity of refined sugar is the basis of most of the criticisms of it as a food. It contains no vitamins nor minerals, and this has been used as an argument against it, but the compactness, keeping quality, low cost, and other valuable properties argue strongly for its value as an energy producer to supply the needed calories in high-calorie diets.

Dr. Stroud Jordan[3] offers the following regarding the lack of vitamins and minerals in refined sugar:

> More than eighty percent of all refined sugar, consumed as food, carries from three to four times its own weight of other essential foods along with it.
>
> If we depended on refined sugar as the oriental depends upon polished rice, then it should contain all minerals and vitamins that are essential to the complete diet. But with less than fifteen percent of the entire caloric intake supplied by sugar, and the remaining eighty-five percent containing a large excess of minerals and vitamins such a contribution by sugar is neither indicated nor essential.

Dr. Howard S. Paine, of the U.S. Department of Agriculture, gives the following factual information as to the value of sugar in the diet.[4]

> As a carbohydrate food, which provides a high energy value per unit of weight, and which furnishes a degree of sweetness that is highly valuable for improving the palatability of other foods. In addition, by virtue of other important properties, sugar serves as a preservative agent for preserving other foods (for example, fruit and fruit products) and also provides a physical basis for fabrication of various food products (for example, confectionery). It is also valuable for making desirable modifications in the properties of various food products (for instance, controlling retention of moisture). Sugar has many accessory features of value (in addition to its specific nutritive value) in the preparation of other foods.

The Sugar Research Foundation, Inc., established in 1943 with headquarters in New York, has for one of its primary purposes the study of the food value of sugar and sugar products. All the cane and beet sugar interests which supply the markets of the United States have contributed to this enterprise, which will also investigate other uses for sucrose.

The U.S. Bureau of Labor Statistics in April, 1942, gave retail prices of various commodities which show the following cost relationship between the calories in refined sugar and those in other common foods. The calories in $1.00's worth of granulated sugar would have cost $1.10 in lard; $1.25 in rolled oats; $1.91 in bread (white); $2.01 in rice; $2.29 in potatoes; $3.29 in butter; $4.31 in cheese; $5.06 in rib roast (beef); $6.12 in lamb chops; $7.11

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[2] *Technology for Sugar Refinery Workers,* 1941, pp. 351–358.

[3] Address before Institute of Food Technologists, New York, Dec. 5, 1943.

[4] Lamborn, *Sugar Market Report* (New York), Jan. 19, 1943.

in pork chops; $9.07 in chicken; $12.52 in ham (sliced). It is to be understood that the comparison is on a basis of calories and not of general dietetic value. As stated previously sugar is a pure carbohydrate, and therefore it is an energy producer only.

10. Per Capita Consumption of Sugar (1936–1937).

Denmark	121 lb.
Great Britain	113
Australia	112
Sweden	108
United States	103
Canada	101
Switzerland	92
Cuba	91
Ireland	88
Argentina	86
Norway	70
Belgium	65
Holland	63
Germany	61
Czecho-Slovakia	61
Austria	59
France	58
British South Africa	54 lb.
Brazil	48
Algiers, Morocco, and Tunis	42
Japan	34
Mexico	31
Poland	27
Hungary	25
Spain	25
Russia	25
Egypt	24
British India	24
Italy	19
Persia	17
Roumania	13
Turkey	13
China	3

CHAPTER 2

RAW MATERIAL

11. Sugar Cane. Sugar cane is a large grass, belonging to the genus *Saccharum*. All the so-called noble canes which, up to fairly recent times, comprised all the cultivated varieties of the world are of one species, *S. officinarum*, and the endless varieties which occurred throughout the tropical and semitropical regions of the globe are the results of soil conditions, climate, and modes of cultivation. Four other species are now recognized: *S. spontaneum*, *S. sinense*, *S. barberi*, and *S. robustum*. The first species includes the wild canes of India and the Pacific Islands, which because of their vitality and disease-resisting characteristics have been used extensively for breeding purposes. The famous variety POJ 2878 is in part a descendant of this species. *S. sinense* includes the Chinese canes of which "Uba" is a type, whereas the last two species are of lesser importance.[1]

12. Seedling Canes. Until 1888 the flowers of the sugar cane were supposed to be sterile, but the discovery simultaneously in Java and in Barbados that the cane produces viable seed started a search for new varieties in experiment stations in various parts of the world which has resulted in the production of large numbers of seedlings. The seedlings are crossed with other seedlings and with existing varieties in order to develop certain characteristics. These experiments have resulted in numerous varieties which are now in broad culture. The new varieties are selected for some particular qualities such as richness in sucrose, resistance to disease, persistence of type, time of ripening, milling qualities, fuel value, and color. The first extensive use of seedling varieties was in Java where few of the old varieties are now cultivated.

The seedlings are designated by initials and numbers, the initials indicating the origin, the best known being the famous POJ series (Proefstation Oost Java, or East Java Experiment Station). Seedlings originating in Demarara (British Guiana) form the D series, and the initials PR for Puerto Rico, B for Barbados, H for Hawaii, etc., are easily recognized.

Less readily understood are such initials as CP for U.S. Experiment Station, Canal Point, Florida, Co for Coimbatore Experiment Station (India), BH for Barbados Hybrid, and EK, the designation used in Java for the POJ canes developed by E. Karthaus.[2]

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[1] For a concise review of the subject see "Sugar Cane and Its Varieties," by M. C. Alcantara, *Tenth Annual Proc. Assoc. Cane Sugar Tech. Cuba* (1936), p. 193.

[2] Arthur H. Rosenfeld, *J. Dept. Agr. Puerto Rico*, **11**, 1927.

13. Selection of Varieties. Because of the restricted land in Hawaii and Java a constant search for varieties giving higher yield of sugar per acre has resulted in the gradual replacement of old varieties for new,[3] but on this side of the globe except in the British West Indies and Louisiana dependence until recently has been largely on the so-called native canes, i.e., those that have been in cultivation in the locality for many generations.

A graph of the plantings of varieties in Hawaii each year during the period from 1913 to 1937 shows the slow but constant change taking place in the search for the perfect cane in those islands. At the beginning of the period there were 100 per cent of "native" canes, but the seedling canes have now risen to 85 per cent. The graph also shows that the plantings of the predominant varieties have undergone gradual changes.[4]

Earle[5] says, "It is only through calamities that threaten the very existence of the sugar industry that progress in the knowledge of cane and its culture usually occurs." The terrific inroads of the mosaic disease in the Argentine which threatened the extinction of the industry about 1910 were stopped by the introduction of the POJ varieties by Rosenfeld and others. These Java canes, though not immune to mosaic, are highly resistant to its effects, and the crops in the Argentine are now much greater than at any time in the history of the industry. Similarly in Puerto Rico mosaic developed to an alarming extent between 1915 and 1918, necessitating the introduction of new varieties by Earle and Rosenfeld. Uba cane, immune to mosaic, was found effective in checking the disease, but its low sucrose, high fiber, and other poor manufacturing qualities have caused its replacement by Java and British West Indies seedlings. In 1918 Puerto Rican plantings were 98 per cent of native canes (Rayada and Cristalina), but today the more progressive companies have changed almost completely to seedling varieties with notable increase in yield.

Louisiana used Demarara seedlings for many years, but mosaic, the cane borer, and other maladies reduced the yields per acre to an extremely low figure.

The POJ canes (234, 213, and 36) were tried out, beginning about 1927, and gave promising results at first, but by 1934 the yields both of cane per acre and sugar per acre for all varieties were reduced to one-half of what they were six years before. Denley[6] says:

> The complete failure of the old varieties Louisiana Purple and D 74 and the evidence which indicates that the POJ varieties were headed toward complete failure raise the question as to whether or not the Louisiana sugar industry must continue to change varieties periodically.

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[3] See Maxwell, *Economic Aspects of Cane Sugar Production,* Chapter XI, London, 1927.

[4] Mangelsdorf, *Proc. 6th Congr. Intern. Soc. Sugar Cane Tech.* (Baton Rouge, 1938), p. 726.

[5] *Facts About Sugar,* May 7, 1927.

[6] *Proc. 6th Congr. Intern. Soc. Sugar Cane Tech.* (Baton Rouge, 1938), p. 714.

There is already evidence that the Canal Point and Coimbatore varieties now being grown are not as resistant to disease as they were when first planted.

Cuba was the last of the cane-producing countries to turn to variety canes and even yet Cristalina, the so-called native cane, is grown on more than one-third of the acreage. Figures from the Cuban Department of Agriculture (1940) show the distribution of the acreage to be POJ 2878, 38 per cent; Cristalina, 36 per cent; other POJ canes (2726–2883–2727–2714), 13 per cent; Co 213, 5 per cent; and Co 281, 4 per cent. There is no doubt that here, as in other countries, the trend will continue away from the native canes and toward the varieties.

14. Mode of Growth. Sugar cane is propagated by means of the buds that are located at the nodes. Pieces or cuttings of the cane are planted with a very shallow covering of soil or in certain localities are only partly covered, but in this latter event are irrigated. Each bud produces a plant and from each of these there are several shoots or suckers. These form a clump or stool of canes. The cane under suitable soil and climatic conditions is usually planted but once in several years. New plants, termed ratoons, spring up. from the stubble after harvesting the crop and produce a second crop, and so on. Fiscal or soil and climatic conditions sometimes limit the crop to "plant cane" or to plant cane and one or two ratoons.

The length of time that the cane is allowed to grow before harvesting varies greatly in different countries. In Louisiana climatic conditions require that cutting begins after seven or eight months' growth, so that the cane never fully matures; Cuba and Puerto Rico average ten to twelve months, whereas in Hawaii the bulk of the crop is allowed eighteen, twenty, or even twenty-four months' growth. A plant crop and four or five ratoons constitute the average cycle in Hawaii, so that only about 10 per cent of the total area in cane is planted each year.

The ripening of cane depends on many factors, most important of which are the nature of the variety and amount and distribution of rainfall or irrigation. The plant matures with the approach of cool or dry weather, the highest sugar yields being found in countries with a pronounced dry season, and in irrigated countries the distribution of water is suspended for a few weeks previous to the cutting season in order to ripen the cane. The sucrose content of the stalks increases, and the reducing sugars decrease as the plant approaches maturity.

Some varieties of cane "arrow" or flower freely in the tropics but others seldom or never flower. Few arrows have been noted in Louisiana and then only in exceptionally mild years. It is generally believed in Cuba that a year in which the cane arrows freely is not usually very productive. Such cane, however, is often rich in sucrose and of low invert sugar content. It increases in its sucrose content for several months after flowering, and, as is true with other canes, deteriorates as regards the sugar when the rainy season begins

The yield of cane, however, may be small, since the plant grows little taller and heavier after flowering.

Normal sugar canes are never hollow or even partially so. They contain approximately from 87 to 90 per cent of juice and some water, in composition with certain plant constituents (colloid water), that contains little or no sugar. This colloid water has been estimated in Java to be 2.5 per cent of the cane, but some authorities believe it to be between 7.5 to 10.0 per cent.[7]

15. Harvesting. This may begin before the cane is considered to be ripe in order to obtain a longer working season. The stalks are cut off close to the ground in harvesting and should be topped just above the highest colored joint. These conditions vary somewhat with the country and for irrigated fields. Where irrigation is practiced, the two or three top joints are usually removed for use in planting new fields. Since these joints are of lower sucrose and higher glucose (invert sugar) content than the rest of the stalk, the raw material entering the factory is improved by their removal (see Sec. 29). The leaves are also removed at the same time that the stalk is cut.

In Cuba, the canes are often topped too high, thus giving the laborer a larger wage and the farmer more cane, but reducing the return to the manufacturer through a smaller yield of juice in milling and the necessity of grinding the poorer parts of the stalk. It is customary in harvesting in Java to dig the earth from about the plants and cut off the stalks a foot below the surface of the ground. The top joints of the canes are reserved for "seed" in planting.

In Hawaii it is now common practice to send the cane to the mill without topping since the ratio of cane to tops is much greater in crops grown eighteen to twenty-four months. However, difficulties are reported in clarification where this procedure has been adopted.[8]

16. Machine Harvesting. Hand cutting as described is still general practice, but machines for harvesting have been invented from time to time, none of which has proved practicable until recently. In Louisiana, two machines, the Munson-Thompson and the Wurtele, have been developed and are in practical use.

The Munson-Thompson machine is made up of a structural steel superstructure for carrying the operating mechanism, mounted on three pneumatic-tired wheels, one in the center at the front of the machine, and two at the rear opposite each other. The front wheel is somewhat smaller, and the tractor power is supplied to the rear wheels. The gage or span of the two rear wheels equals the spacing of two alternate furrows, bringing two rows of cane within the cutting range of the machine at the same time. The latest model (Fig. 2) cuts only one row at a time, but the general principle of operation is much the same.

The serrated cutting knives, located at the bottom of the machine just aboveground, are adjustable for raising or lowering to suit conditions. The

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[7] Behne, *Intern. Sugar J.*, January, 1938, p. 17.

[8] H. A. Cook, *Repts. Assoc. Hawaiian Sugar Tech.*, 1937, p. 59.

topping cutters on the order of a circular saw operate in a horizontal plane. Just ahead of these saws is a large, short scroll which serves to feed and hold the cane while the saws are operating, the cut tops falling to the ground through a metal chute or hopper.

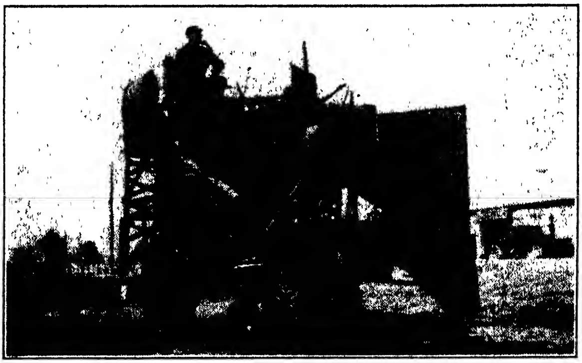

Fig. 2. Munson-Thompson Cane Harvester.

The machine is driven by a gasoline engine of the automotive type. The front of the machine is equipped with guides made into a framework with light pipe for guiding the cane into the path of the knives and saws as the machine advances.

Fig. 3. Wurtele Harvester and Loader.

Strictly speaking this machine does not completely harvest the cane, as the leaves remain on the stalk and go to the mill or must be removed by burning the cane after the stalks are on the ground. This practice is detrimental to the clarification qualities of the resulting juice and is not practicable during wet weather. In the experimental stage is an addition to the harvester by means of which the cut and topped cane will be chopped into short lengths and the leaves and trash will be freed and removed by brushes and a strong blower. This apparatus has not proved successful on a plant scale so far (1942).

The Wurtele machine (Fig. 3) is made up of a standard four-wheeled rubber-tired farm tractor and a steel superstructure made of steel pipe or tubing of welded construction. The machine is designed to cut only one row of cane at a time.

When operating, the canes first come in contact with a two-armed rotary cutter which cuts off the tops. The canes are then engaged by the chain with fingers which bring them in and hold them against a rubber-edged guide while a rapidly revolving circular saw blade is cutting the canes just above the ground, after which they are delivered to a carrier which throws the cane into a trailer drawn by the harvester. It therefore is a harvester-loader. Like the Munson-Thompson machine the Wurtele has no provision for removing the leaves. The Falkiner harvester cuts, cleans, and loads the cane, but trials in Hawaii have not been favorably reported upon.[9] It cuts the cane into lengths of 6 to 12 in.

17. Grab Harvesting in Hawaii. A decided innovation in cane cutting is "grab harvesting" as practiced in Hawaii.[10] The cane, which has always been burned on these islands, is picked up without cutting and is broken off at the roots at which point the stalk breaks easily. The apparatus is similar to the "grab loader" commonly used in Louisiana, but it is made heavier and larger to handle 2 tons of cane instead of 500 lb. Much trash and dirt are picked up in the cane by this method, and means must be installed for clearing the cane of trash, rocks, and dirt at the factory, except with irrigated canes which burn clean and can be grab harvested with relatively little dirt and trash.

At Ewa Plantation a cane-cleaning plant invented by Munson of Louisiana chops the cane in 12-in. lengths. These are passed through revolving brushes to remove adhering dirt and trash, which are then separated from the cleaned cane by a large suction fan. A rock eliminator precedes this cleaner and consists of a wide drag-type conveyor through which the rocks fall.[11] The operation is not entirely successful.

An extension of grab harvesting is "drag-line harvesting." In this method, the cane is torn by a heavy dray rake which is hauled in by a crane and back by a tractor. The cane which is thus hauled into piles is picked up by a grab on the crane and is loaded into trucks or other transport system.

It will be seen that the harvesting practices in Hawaii and the harvesting machines in Louisiana result in much trash and dirt going to the mills, whereas formerly the insistence was on "clean cane."

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[9] *Intern. Sugar J.*, May, 1939, p. 179.

[10] W. J. Maze, *Proc. 6th Congr. Intern. Soc. Sugar Cane Tech.* (Baton Rouge, 1938), p. 524.

[11] *Intern. Sugar J.*, May, 1939, p. 181.

18. Burned Cane. The lower leaves of the cane become quite dry as the harvest season advances. These often take fire through accident or intention. It is usual to burn cane fields that contain irritating weeds or small and trashy cane. This burning is to facilitate harvesting, and it is practiced especially where labor is expensive. It is general practice to burn all cane in Hawaii before harvesting. The cane is not injured by burning, but it must be harvested very promptly to avoid loss through deterioration, which is accelerated by the burning. The rate of deterioration is greatly increased if rain falls upon the burned cane. The manufacturer agrees in most Cuban cane contracts to receive burned cane up to and including five days without deduction from the price, but in the event of rainfall he may refuse it at any time. The purification of the juice is not usually so readily accomplished with burned as with sound cane, and the heating surfaces of the evaporator foul sooner. The fine particles of carbon sometimes persist through the manufacture and finally appear in the sugar. It is preferable to grind a mixture of sound and burned cane rather than burned cane alone, since the mixed juices are more readily purified.

19. Deterioration after Cutting. The sugar content of the cane deteriorates rapidly after the stalks are cut; therefore these should be conveyed to the mills as soon as possible and be ground immediately. The small factory, as compared with the large factory, usually has an advantage with respect to the freshness of its raw material, owing to its proximity to the fields. To insure regularity in the delivery of the raw material the large factory generally has a considerable quantity of cane cut in advance of grinding, which lies exposed to the sun in the fields. This is unquestionably a source of huge loss which is preventable in part at least by more systematic harvesting.

Spencer noted a fall of several degrees in the coefficient of purity of the juice from canes that had been cut and left lying three or four days in a hot climate, exposed to the sun in cars, and on the ground. He also stored cane under cover, during cool weather in Louisiana, with no appreciable deterioration in a period of more than two weeks.

An extensive series of experiments covering three recent crops in Louisiana showed conclusively that inversion and deterioration of canes in storage are intimately associated with loss of moisture and increase with the drying out of the cane. This investigation by the U.S. Bureau of Plant Industry[12] proved that sprinkling of cane in small piles prevents heavy loss of sucrose; that the storage of unsprinkled cane may result in heavy losses of sucrose by inversion; that wet weather tends to check inversion, whereas storage in the sun increases it. Stored in large piles and sprinkled, inversion is retarded during six to ten days. The temperature difference due to sprinkling does not appear sufficient to account for the reduced deterioration, which seems

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[12] Lauritzen and Balch, *Intern. Sugar J.*, March, 1935, p. 104.

to result from the difference in the turgidity of the cane, i.e., maintenance of normal turgidity by sprinkling during storage seems to account for the value of the sprinkling process.

The more mature the cane the less rapid the deterioration, and the variety is also a factor, although all varieties deteriorate more rapidly if unsprinkled. Co 281 deteriorates less rapidly under all conditions, whereas POJ 36-M and CP 807 appear to be the most susceptible. The cane in these experiments was sprinkled four times a day long enough each time to insure wetting all of it. Large-scale factory experiments with sprinkling corroborated the results. Similar experiments in India showed that moderate sprinkling was preferable to excessive wetting, but that the latter is better than no sprinkling at all.

Rosenfeld[13] in Egypt recommends sprinkling in the fields and covering with trash to prevent evaporation. He gives figures to show that during hot weather 11 per cent of cane weight can be lost through evaporation in four days and 17 per cent in eight days. Inversion and evaporation losses together may reduce the yield of sugar after eight days in the field to less than half that of fresh canes. The figures give further proof, if any were needed, of the great urgency, both to planter and manufacturer, of grinding freshly cut cane.

20. Windrowing. In those cane countries such as Louisiana where freezes occur during the harvesting season it is necessary to cut all the cane immediately after a freeze in order to prevent excessive deterioration and even complete loss. This is done by windrowing, which consists of cutting the cane without removing the leaves or tops and allowing the stalks to fall between the rows in such a way that the leaves of one stalk protect the next from evaporation and freezing. Lauritzen and Balch[14] show that whereas temperature, humidity, loss of moisture, and other factors have a marked effect on losses in cane after cutting and particularly in windrow, there is a decided difference in susceptibility in the different varieties in Louisiana. The most resistant cane was Co 281, with Co 290 and CP 29–116 showing less resistance, whereas a large group of canes show little resistance and therefore heavy losses during windrowing and in storage.

21. Transporting. The method of transport of the cane to the factory varies with local conditions. Small factories usually transport their cane in carts or small cars. Portable railways are largely used in the Hawaiian Islands and in Java, but in Cuba, Puerto Rico, and Louisiana the greater part of the haul is by permanent railroads, either public or private. The privately owned railroads are frequently narrow gage. The cane is brought to the factory or railway in Cuba in bullock carts. It is flumed to the factories in the Hawaiian Islands, and in British Guiana it is usually transported in punts.

[13] *Intern. Sugar J.*, January, 1937, p. 10.
[14] *Sugar Bull.*, Oct. 15, 1937.

In Louisiana, mule carts were almost universal in the fields up to a few years ago, but now motor tractors and carts equipped with very large rubber tires are becoming quite common. Such hauling equipment, besides being faster and of greater capacity, is able to enter the fields during wet weather when mule carts would be mired. Motor trucks and large trailers (10–15 tons capacity) are also replacing railroad haul in Louisiana. Barge transportation where waterways are available is also regular practice in Louisiana.

22. Manufacturing Season. The season begins at greatly varying dates in various parts of the world. In the almost rainless districts where irrigation is practiced, grinding may be carried on during nearly or quite the entire year. This is true in Peru and in parts of the Hawaiian Islands. The season of manufacture begins in October and November in Louisiana and lasts through December and often into January.

Before the advent of the quota system with curtailment of crops the grinding season in Cuba and the other West Indies was December into June. Since crops have been so greatly reduced, the Cuban season has begun in January and continued for sixty to ninety days. The Hawaiian season begins in November and continues about six months, though climatic conditions in many parts of the Islands permit a very much longer season. The factories of the Dutch East Indies grind during the dry monsoon, or from about May into November. This corresponds very nearly with the Argentine season.

The advent of the rainy season determines the close of the manufacturing period in the tropics. The rains not only interfere with the transportation of the cane but also cause it to renew its growth at the expense of its sucrose content.

23. Purchase of Cane. Payment for cane is made by a wide diversity of methods in the various countries of the world, and the details, advantages, and disadvantages of these are considered fully in Chapter 37. The methods may be divided roughly into three classes. (1) The cane is purchased on a flat basis irrespective of sucrose content. (2) The sucrose (pol) and the price are directly related. (3) A flat basis is paid for "normal" (average) cane, and penalties are imposed for cane of lower quality, with bonuses for cane of superior quality. Cuba is one of the few large countries which continues to pay on a flat price basis irrespective of quality. Louisiana is an example of the third type, and South Africa, Queensland (Australia), Puerto Rico, the Philippines, and Mauritius follow the second plan. In the Hawaiian Islands and Java most of the cane is grown by the factories that grind it.

No matter what system is adopted, more or less arbitrary allowances and deductions are generally made, and there are wide variations in the percentage of sugar value which goes to the grower. According to Moberly[15] the lowest percentage (48 per cent) is paid to the grower in Hawaii and the highest (70–75 per cent) in Australia. The average is somewhere close to 60 per cent.

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[15] *Proc. 5th Congr. Intern. Soc. Sugar Cane Tech.* (Brisbane, 1935), p. 145.

COMPOSITION OF CANE AND ITS PRODUCTS

24. Variation in Composition. Sugar cane varies greatly in richness in different countries, as well as in different localities in the same country, and in different years in the same locality. The sucrose content in Cuba rarely reaches 17 per cent, averaging between 13 and 14, whereas Louisiana considers 12 per cent as very rich. Most tropical countries such as Puerto Rico and the Philippines have figures corresponding to those of Cuba, but in Hawaii irrigation is practiced, and the percentage runs much higher.

The table on p. 20 showing the composition of the stalks of Louisiana cane at the time of harvesting, November-December, was made many years ago by Dr. C. A. Browne, of the Research Department of the Bureau of Chemistry and Soils and formerly of the Louisiana Sugar Experiment Station. The figures are condensed from many analyses of the purple variety of the cane.

As will be shown later the composition of the cane varies greatly on account of many conditions, and this typical analysis should therefore be taken as indicative only. In fact Louisiana juices today would show very different analyses from those shown in the table on p. 20.

A great many studies have been made on the composition of the juice expressed from the cane, and it has been found that the juice varies with the variety of the cane, its maturity, the part of the stalk from which the juice comes, and the climatic and soil conditions under which the cane has been grown. The degree of milling also has a great effect on the composition of the juice. Furthermore, not all the soluble substances in the cane are extracted to the same degree when the cane is crushed, as has been shown by Fort and Holmes[16] and many others. Still more variation in the juice composition is occasioned by the extent of the milling; the more extensive the milling the greater is the extraction of non-sugars in proportion to the sugars. This is evident from the successively lower purities of the juice from the first to the last mills in a train (see Sec. 53). However, these variations in composition are quantitative rather than qualitative, and all cane juices have more or less the same constituents but in varying proportions.

A recent study by Fort and McKaig of the U.S. Department of Agriculture on the "Comparative Chemical Composition of Juices of Different Varieties of Louisiana Sugar Cane,"[17] is possibly the most extensive yet undertaken. They found that a variety grown under identical conditions always shows the same relative content of non-sugar substances. Because of the differences in non-sugar content between varieties, they conclude that all studies to determine the effect of soil, climate, or other factors must use identical varieties.

The solids of the juice may be considered as divided into sugars, inorganic non-sugars (ash), and organic non-sugars, but as Fort and McKaig point out, there is no sharp division between the latter two. The amount of ash is not

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[16] *Sugar Bull.,* Nov. 15, 1937, p. 3.

[17] *U.S.D.A. Tech. Bull. 688,* October, 1939. 65 pages (64 references).

Composition of Louisiana Sugar Cane (about 1910)

	Per Cent			Per Cent
Water	74.50			74.50
Ash	0.50	Silica, SiO_2		0.25
		Potash, K_2O		0.12
		Soda, Na_2O		0.01
		Lime, CaO		0.02
		Magnesia, MgO		0.01
		Iron, Fe_2O_3		Trace
		Phosphoric acid, P_2O_5		0.07
		Sulfuric acid, SO_3		0.02
		Chlorine, Cl		Trace
Fiber	10.00	Cellulose		5.50
		Pentosans (Cane-gum)	Xylan	2.00
			Araban	0.50
		Lignin bodies, etc.		2.00
Sugars	14.00	Sucrose		12.50
		Dextrose		0.90
		Levulose		0.60
Nitrogenous bodies (Total N = 0.06 per cent)	0.40	Albuminoids		0.12
		Amides (as asparagin)		0.07
		Amido acids (as aspartic)		0.20
		Nitric acid		0.01
		Ammonia		Trace
		Xanthin bodies		Trace
Fat and wax	0.20			0.20
Pectin (gums)	0.20			0.20
Free acids	0.08	(Malic, succinic, etc.)	*	0.08
Combined acids	0.12	(Malic, succinic, etc.)		0.12
Total	100.00			100.00

* Recent work by McCalip and Seibert (*Ind. Eng. Chem.*, May, 1941, p. 637) shows that aconitic acid is the most important acid in cane and cane products. (See p. 24.)

a measure of the salt content since some salts are present in combination with organic non-sugars.

25. Sugars in the Cane. Sugar cane usually contains three sugars, sucrose, dextrose, and levulose (*d*-fructose, fruit sugar). The dextrose and levulose are present in the very immature plant in nearly equal proportions, i.e., in the proportions in which these sugars are formed when sucrose is inverted by acids. As the cane matures the levulose content decreases and sometimes disappears. Levulose again always appears, however, in the molasses. This reappearance is due to isomeric changes in the dextrose when its solutions are heated in the presence of salts of the alkalis and alkaline earths, notably potassium salts. The reducing sugar content of Cuban cane juice, lævulose and dextrose, termed the glucose in the cane industry, usually ranges between 0.4 and 1.35 per cent and these figures would also hold for Louisiana. CP 28–19 was found by Fort and McKaig[18] to average 0.48 in 1932 whereas Co 290 showed 1.19 and 0.97 per cent in 1931 and 1932 respectively. The reducing sugars are sometimes almost or quite absent from cane juices. This condition existed for several weeks one season in Spencer's experience at Magnolia Plantation, Louisiana.

Figure 4 shows data according to Browne on the ripening of cane in some experimental plots in the Louisiana Experiment Station. The percentage of sucrose obtained in this cane is quite abnormal for Louisiana and closely approximates best tropical conditions.

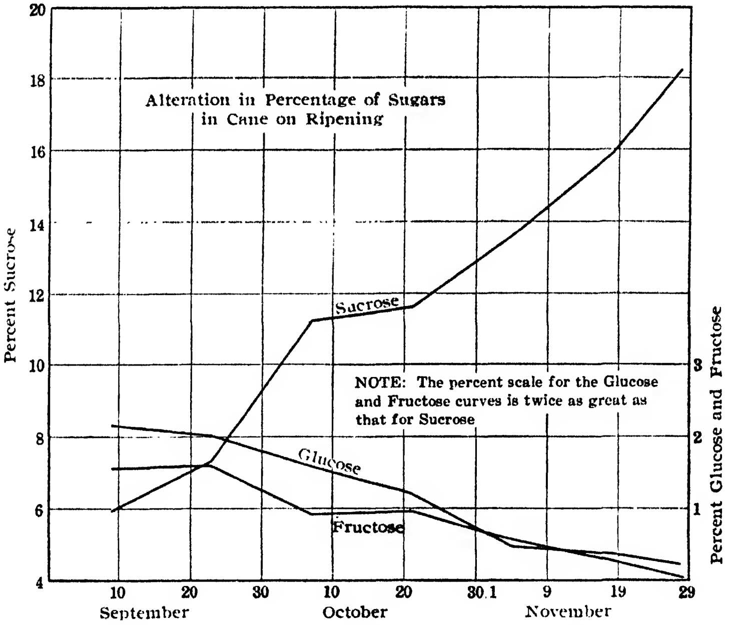

Fig. 4. Percentage of Sugars in Cane During Ripening.

26. Ash Content. The mineral or ash content of cane juice is quite variable both as to quantity of total ash and as to amount of the various constituents. In Cuba the ash in the juice varies from 0.25 to 0.80 per cent, and Fort and McKaig show averages in Louisiana of 0.40 per cent on the juice for POJ 36-M for 1931 and 0.72 per cent for CP 28–11 in 1932. These authors point out that the modern varieties of cane have a higher ash content than the older canes by at least a third and that throughout the world the percentage of ash is on the increase.

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[18] *Loc. cit.*

Average figures in the following tabulation show the variation in Cuba from one year to the next (Spencer) and for varieties in Louisiana (Fort and McKaig).

COMPOSITION OF CANE JUICE ASH

Percentage of Ash

	(Spencer) (Cuban)			(Fort & McKaig) (Louisiana)		
	Factory A (1913)	Factory A (1912)	Factory E (1912)	CP 2811 (1933)	POJ 234 (1932)	Co 281 (1933)
Silica (SO_2)	5.56	11.90	6.46	3.20	3.5	4.0
Iron (Fe_2O_3)	19.00	12.48	3.00	0.37	0.39	0.33
Alumina (Al_2O_3)	.....	.....	.....	2.00	2.20	2.60
Lime (CaO)	7.62	10.25	4.70	5.7	3.4	7.9
Magnesia (MgO)	6.55	6.83	5.01	6.7	8.4	7.5
Potash (K_2O)	25.15	27.14	46.28	49.5	51.1	43.9
Soda (Na_2O)	5.35	2.32	1.36	0.3	2.5	0.9
Phos acid (P_2O_5)	5.44	7.15	4.21	13.1	13.8	11.4
Sulf acid (SO_3)	15.02	9.84	4.08	17.9	12.6	16.2
Chlorine (Cl)	5.14	5.10	12.90	6.7	7.6	6.5
Carbonic acid (CO_2)	2.68	9.59	10.53	.....	.....	.....

These averages may be very deceptive as the individual variations are wide. For example, in Cuba the largest constituent, K_2O, varied from 25 to 46 per cent in different factories, whereas in Louisiana the minimum and maximum found by Fort and McKaig for K_2O in all juices tested were 36.6 per cent and 60.1 per cent respectively. A still greater variation was found in the CaO content, the lowest being 0.7 per cent (on ash) and the highest 13.8 per cent. The limits of P_2O_5 were 5.4 and 27.8 per cent, and for SO_3 4.3 and 31.8 per cent. Obviously no quantitative statement can be made about the composition of the ash of a cane juice unless analyses have been made.

The ash determination does not indicate the total salt content as it measures only the mineral content and leaves out the organic salts. In Louisiana Fort and McKaig showed that the total salts are from 58 to 72 per cent higher than that of the ash constituents, the quantity of organic salts being nearly equal to the sum of all the mineral salts. Here again the variations are wide. The organic salts are of practical importance because of their effect on molasses formation and sucrose recovery.

27. Organic Non-Sugars. The organic non-sugars are the so-called organic impurities of the juice and contain the fats and waxes, nitrogenous substances, gums, organic acids, hemicelluloses, pentosans, etc. The percentage of total organic non-sugars per cent solids can be obtained by subtracting the sum of the sucrose (Clerget), reducing sugars, and ash from the true solids. The use of apparent solids by Brix and sugar by polarization in this calculation gives a misleading figure. The total organic non-sugars in Louisiana juice

(1931) ranged from 5.69 to 6.42 per cent of the solids in the juice (Fort and McKaig). The average on the basis of the juice itself was close to 1 per cent.

28. Nitrogenous Constituents. The nitrogenous non-sugars in the juice are roughly only one-half those in the cane according to Honig,[19] and Fort and McKaig proved that about one-third more nitrogen is in the whole mill juice than in the crusher juice. The percentage is much higher in second and third mill juices,[20] showing that the degree of extraction is of great influence. Formerly all nitrogenous compounds were considered objectionable; but those which are coagulated by heat and lime (protein materials, also classified as albumin and albuminoids) are actually beneficial in that they aid clarification. Salinas [21] says that for satisfactory clarification at least 0.10 per cent albumin in conjunction with 0.30 per cent P_2O_5 is required. Fort and McKaig divided the nitrogen in Louisiana juices according to coagulability, classifying these divisions as protein and non-protein nitrogen. They found wide variations between varieties in the amount of non-protein nitrogen, the objectionable portion, the POJ canes being twice as high in this constituent as Co 290.

The exact composition of the objectionable nitrogenous compounds is not certain and may vary widely both quantitatively and qualitatively.

Zerban [22] isolated asparagin, glutamin, and tyrosin. A part of the asparagin and a still greater part of the glutamin are broken up in the manufacture with the result that aspartic and glutamic acids accumulate in the molasses along with undecomposed asparagin and glutamin. These amides are largely responsible for the ammonia given off during the evaporation of the juice. Acid amides and amino acids are positive molasses-makers. (Glycocoll, guanine, and lecithins are also nitrogenous substances which have been reported in juice.)

Quite recently, Browne and Phillips [23] in a study of the methoxyl and uronic acid content of the sugar cane gave the following analysis of bagasse (laboratory made) from Louisiana cane (Co 281):

Ash	2.37	
Pentosans	22.06	Calculated to moisture-free and ash-free basis
Methoxyl (OCH_3)	2.46	
Uronic acids (as anhydrides)	2.96	
Lignin	8.90	
Methoxyl in ash-free lignin	15.90	

They also give figures for cane juice and molasses showing that hot maceration and liming in the presence of particles of cane fiber greatly increase the methoxyl and uronic acid content of the juices, bearing out the observa-

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[19] *Intern. Sugar J.*, July, 1934, p. 272.

[20] Geerligs, *Cane Sugar*, second edition, p. 131.

[21] *6th Annual Proc. Assoc. Sugar Tech. Cuba* (1932), p. 116.

[22] *Rept. 8th Intern. Congr. Applied Chem.*, 8, 103.

[23] *Intern. Sugar J.*, November, 1939, p. 430.

tion of milling practice that juices so treated are richer in gums and hence more difficult to clarify. These authors believe that the methoxyl and uronic acid constituents of cane juice and molasses belong chiefly to that class of organic complexes known as pectins.

The nitrogen compounds, gums, and organic acid radicals constitute only about a third of the organic non-sugars of the juice, the remainder probably being largely hemicelluloses.[24] The role of cane wax in sugar cane and its products will be dealt with in detail below.

29. Variation in Composition in Different Parts of the Stalk. The sucrose content of the cane and also the coefficient of purity of the juice vary greatly in different parts of the stalk. The juice of the nodes is of lower sucrose content and lower purity than that of the internodes. A similar difference exists between the lower and upper halves of the stalk.

Noël Deerr analyzed whole canes, the pith, rind, and node, and has referred the sucrose in each part to that in the whole cane on a percentage basis. His figures are given in the following table for three varieties of Hawaiian canes. In using these figures it should be considered that the separation into pith, rind, and node can be made only approximately.

VARIETY	ROSE BAMBOO	YELLOW CALEDONIA	LAHAINA	LAHAINA	YELLOW CALEDONIA
Origin of Sample	Oahu	Oahu	Oahu	Maui	Kauai
Pith	83	77.3	82.4	72.2	76.6
Rind	4.7	8.7	6.9	8.8	10.3
Node	12.3	14.0	10.6	19.0	13.1

King[25] has made an elaborate study of the distribution of the sugars and the ash in Philippine canes which shows the variation in the bottom, upper bottom, middle, lower top, and top. In general the two bottom sections have the richest juice and the top the poorest, but this is not invariably true.

Fort[26] analyzed the tops and the mill cane of whole cane of varieties Co 281 and Co 290. As would be expected the objectionable constituents (fiber, reducing sugars, ash, and organic non-sugars) are all much higher in the tops, whereas the sucrose is largely concentrated in the mill cane (85 to 90 per cent in the mill cane, 10 to 15 per cent in the tops). Honig, in the study already referred to, determined non-sugars in juice, bagasse, leaves, and tops. In one example, he found 37 per cent of the total ash was in the juice, 22 per cent in the bagasse, and 41 per cent in the leaves and tops.

30. Acids and Gums in the Juice. Acids other than amino acids are aconitic, oxalic, succinic, glycolic, and malic all of which probably enter into the formation of the organic salts already referred to. According to Yoder[27] malic and oxalic are present only in small amounts, and he could not detect tartaric, succinic, or citric acids. It has been stressed recently that aconitic

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[24] Hardy, *The Planter*, 79, 445 (1927).

[25] *Ind. Eng. Chem.*, August, 1931, p. 957.

[26] *Sugar Bull.*, December, 1937.

[27] *Ind. Eng. Chem.*, 3, 640.

acid is a much more important constituent of cane juice, sirups, and molasses than has formerly been supposed; in fact it constitutes 80 to 90 per cent of the non-volatile acid in the cane. McCalip and Seibert [28] showed that the sediment occurring in sirup in first and second molasses tanks in Louisiana is principally calcium aconitate. Refinery pan and evaporator scales were found to contain aconitic acid. Two samples of Louisiana final molasses showed 1.80 and 2.52 per cent aconitic acid respectively. The presence of this acid in cane products in relatively large amounts may prove of commercial importance as a source of aconitic acid.

The so-called gums of the sugar cane and juice are generally taken to mean the amount of material precipitated by acidified alcohol,[29] and the precipitate is sometimes referred to as alcogel. Fort and McKaig used alcohol without acid and then determined the gums by hydrolyzing the precipitate and determining the reducing sugars so formed. They found the gums so determined to be from 2.0 to 2.5 per cent of the non-sugars, but Hardy [30] found 1.5 per cent. Browne and Blouin [31] found soluble gums much higher in some varieties than in others and stated that "the gums of the juice are derived entirely from the hemicelluloses of the cane fiber and are composed of xylan and araban, with some galactan."

31. Coloring Matter. The cane contains coloring matters such as chlorophyll, anthocyanin, and the saccharetin [32] of Steuerwald. Chlorophyll is insoluble in water and is therefore readily removed in the purification of the juice. Anthocyanin is precipitated in the presence of excess of lime; hence it is removed in the carbonation process. It is partially bleached by sulfurous acid. Anthocyanin [33] is very soluble and decomposes rapidly, and is much more prevalent in dark canes than in light varieties. Saccharetin is present in the fiber of the cane. According to Steuerwald it turns yellow and is soluble in the presence of lime and other alkalis, and it is not altered in the carbonation and sulfitation processes. Saccharetin unites with iron to form an intensely black compound. Such saccharetin as is present in the raw juice passes through all the processes of manufacture and finally appears in the molasses. Saccharetin should be prevented from entering the manufacture by thoroughly straining the juice as it flows from the mills and by the avoidance of alkaline imbibition water.

These results of Steuerwald have recently been corroborated by Narsinger Rao.[34] He concluded that saccharetin does not belong to a definite class of natural coloring matters, and that it may not be a single substance, though it certainly has some relationship to tannins and lignins.

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[28] *Ind. Eng. Chem.*, May, 1941, p. 637.

[29] Ruff and Withrow, *J. Ind. Eng. Chem.*, 1922, p. 1131.

[30] *Loc. cit.*

[31] *Louisiana Exp. Stat. Bull. 91* (1907).

[32] *Intern. Sugar J.*, 14, 53.

[33] C. Muller, *Bull. Assoc. Chim. France*, 31, 849.

[34] *Intern. Sugar J.*, May, 1942 (abs.).

Schneller[35] showed that the most objectionable sources of coloring matter in cane are the water-soluble polyphenols or tannin-like compounds which combine with the iron dissolved from the mills to give the green or brown color to the juice. Later Zerban and Freeland[36] proved that certain oxidizing enzymes in connection with the polyphenols and iron explain all the different colors that a raw juice may assume. This relationship between iron and color has been corroborated by Fort[37] in recent work on clarification of Louisiana juices. He also found color in the clarified juices to be correlated with the CaO and residual nitrogen of the juice after clarification.

32. Reaction of the Juice in the Cane. The hydrogen-ion concentration (expressed as pH) of the juice in mature canes has been found to be a fairly constant figure for any given variety in any given locality, but the variations are much greater than was supposed when the earlier investigations were made. Fort and McKaig found Louisiana crusher juices to average 5.38 pH in 1932 but individual samples ranged from 5.22 to 5.63 pH. The average for Florida was found to be 5.17 pH by these same workers, although the titrated acidity was approximately the same as for Louisiana. Paine and Balch[38] found crusher juices in Puerto Rico averaged 5.09 pH with a range from 4.73 to 5.29. In the Philippines the average was 5.39 pH with a range of 5.26 to 5.57,[39] figures which are practically identical with those for Louisiana, in spite of the fact that Louisiana is a sub-tropical and the Philippines a tropical region.

In the earlier investigations of the pH of normal cane juice it was believed that the pH value could be used for determining the degree of maturity, extent of souring due to burning, evaluation of frozen cane, etc. Later work showed that the variation of pH in normal canes is greater than at first had been supposed, although average figures are close for any given variety and locality. The variations from the average figure therefore make the pH value of any particular juice of doubtful value if only small differences are found. A large difference from the normal average is naturally indicative.

The titrated acidity, expressed as the number of milliliters of tenth-normal alkali required to neutralize 10 ml. of juice is of less interest than the pH, but it is of value under certain conditions. The variations of acidity in normal canes in Louisiana (according to Fort and Lauritzen)[40] are much wider than the pH variations. For a given locality the normal acidity may vary from 1.5 to 3.0 ml., and for the entire sugar belt the extremes are 0.7 to 4.5. The use of total acidity as a measure of the souring of frozen cane is therefore not of great value, according to these writers, and they recommend the use of

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[35] *Louisiana Exp. Stat. Bull. 175.*

[36] *Louisiana Exp. Stat. Bull. 165.*

[37] *Facts About Sugar*, September, 1939.

[38] *Sugar*, **29**, 206 (1927).

[39] Gomez, *Philippine Agr.*, **19**, 609. Also King, *Ind. Eng. Chem.*, August, 1931, p. 955.

[40] *Ind. Eng. Chem.*, May, 1938, p. 251.

"excess acidity" for the measure of souring. Excess acidity is the difference between the acidity of sound cane (before freezing or before souring started) and the acidity of the frozen cane under consideration. Thus a cane having an acidity of 3.0 originally and 4.0 after souring would have an excess acidity of 1.0, whereas another cane having 1.5 acidity before souring and 3.0 after souring would have an excess acidity of 1.5 and would therefore have deteriorated more than the first cane because of the freeze.

The acid formed when juice sours is largely acetic which is volatile, whereas the acids of normal cane juice are non-volatile. A simple measure of the excess acidity suggested by these authors is to distil 25 ml. from 100 ml. of juice and then titrate the distillate. This method is particularly recommended where the excess acidity is less than 1.0 ml.

CANE WAX

33. Occurrence and Importance. A thin layer of wax occurs on the outside of the cane, more noticeably near the nodes, but a portion of the wax complex (mostly the low-melting point fats) is distributed through the inner part of the cane. The wax complex has been the subject of much interest and study for two reasons: (1) it is a disturbing factor in clarification, and (2) the hard wax has commercial possibilities as a substitute for Carnauba and other waxes.

A portion of the wax complex finds its way into the juice, although 60 to 80 per cent remains in the bagasse.[41] Of the portion of the fats and waxes which go into the juice, by far the larger part is trapped or precipitated in the clarification and is found in the press cake. The small fraction which enters the juice has been shown to persist to some extent throughout the entire process of manufacture and refining.[42]

Studies by Fort[43] show that Louisiana canes averaged 0.225 per cent (1937) or 4.5 lb. per ton of cane with variations from 0.19 to 0.26 per cent as the extremes in individual samples. These figures included the combined fats and waxes extracted by carbon tetrachloride. A continuation of this work separated the fats and waxes in canes from the 1938 crop and proved that the low-melting point fats (from the internal part of the cane) greatly exceed the commercially valuable wax from the outside of the cane. The average wax per cent cane was about 0.08 per cent, combined fats and waxes about 0.18 per cent. In this work Fort concluded that three or four million pounds of the external wax (1 lb. per ton of cane) could be obtained from each crop in Louisiana if some method could be devised for removing the wax directly.

Balch (see below) in his extractions with toluene found that the hard wax exceeded the fatty fraction, and there is no doubt that these proportions are widely variable, depending on the kind and method of extraction and many

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[41] Honig, *Intern. Sugar J.*, July, 1934.

[42] Bardorf, *Ind. Eng. Chem.*, March, 1928, p. 258; April, 1929, p. 366.

[43] *Sugar Bull.*, Nov. 15, 1938, p. 4.

other conditions. Because of the complex character of the extracted material all statements as to the composition of "cane wax" are misleading. Wijnberg[44] made a most exhaustive study which should be consulted if the chemical properties of the complex are desired.

34. Commercial Extraction of Wax. For extraction from press cake hot alcohol, ether, kerosene, carbon tetrachloride, carbon disulfide, toluene, and other organic solvents have been advocated, the extract varying greatly, depending on the solvent. The lower-boiling point solvents give soft greasy residues on evaporation, whereas the less volatile solvents yield more of the hard wax in the crude extract. Many patents have been published for the extraction from the cane itself or from the bagasse. No successful process had been developed in the western hemisphere until World War II, but present indications are favorable with pilot plant operation. A commercial project was put in use in Natal, South Africa, some years ago, but it was later abandoned.

The commercial possibilities of cane wax have again been given extensive consideration by R. T. Balch of the U.S. Department of Agriculture,[45] and he has published detailed methods and flow sheets of a pilot plant. He concludes that a solvent having a higher boiling point than the melting point of the wax (78°–80° C.) is preferable and his laboratory work was done with toluene. The crude wax ranged from 5 to 17 per cent of the dried press cake, including the fatty fraction (17 to 42 per cent, average 30 per cent) which must be extracted with a suitable solvent, acetone being selected by Balch as best for this purpose. It has been suggested that the fatty fraction may also have commercial value.

The previous history of commercial cane wax extraction has been that interest revives during wartime but dies down when other better-known waxes are available. Present commercial experiments are more extensive than heretofore and indicate that extraction, even in peacetime, may be profitable.

[44] *J. Soc. Chem. Ind.*, **28**, 991 (abs.).

[45] *Sugar J.*, November, 1941, p. 24.

CHAPTER 3

EXTRACTION OF THE JUICE

35. Unloading the Cane. The cane should be delivered to the mills as promptly as possible after cutting. The loss of sugar every year due to the exposure of the cut cane to the sun in the fields and in cars is unquestionably very large, although it is difficult to estimate with any degree of accuracy (see Sec. 19).

The cane is unloaded from the cars and carts by mechanical devices in the well-equipped modern factory. Where the climate is cool and the cane deteriorates but little in storage, as in Louisiana, derricks are often used. These are usually arranged to lift the cane from the cars or carts and deposit it upon endless conveyors, termed carriers or elevators according to their form, or in large piles for the night work, or in case of interruption in the cart or railway service. When the cane is placed in piles, the same derricks are again used in picking up the cane and in placing it upon the carrier.

Raking devices are used in many Louisiana factories and to some extent in Java for pulling the cane from the cars or a platform onto the carrier.

Hoists and dumping devices are very generally employed in Cuba in unloading cars and carts. The hoists usually have a lifting capacity of one-half or one-third carload of cane at a time and drop it into the hopper from which it is carried to the mills by elevators. In using a hoist, chains or cables are passed under the load and are attached to a yoke, provided with a tripping device. The breakage of bundles often occasions loss of time in this method. However, the use of hoists greatly simplifies the arrangement of the railway tracks in factories which use three milling plants.

Dumping devices are in great favor in Cuba and other tropical countries. They discharge the cane quickly and with a minimum of labor. Further, there is little loss of cane in transit. There are two general types of dumping devices. In the one, the car is run upon a platform, which is then tilted, and the load is discharged endwise through a swinging door into the hopper of an elevator. In more general use, is a second type, with its platform tilted sidewise. The stakes or sides of the car are hinged at the top and fastened at the bottom with latches, which are released when dumping the load. Hydraulic power is usually employed in tilting the platform. The platform is hinged at such points that the weight of the load causes the platform to tilt when the water is released from the hydraulic cylinders. With this arrangement very little water pressure is required to return the platform and car to a level position.

After being unloaded the cane is carried from the hoppers to the mills by heavy endless chain elevators, or slat conveyors, which are fitted at intervals with arms to drag the cane with them.

MILLING PROCESSES

36. Milling Machinery. Multiple mills are used exclusively in crushing the cane and in expressing its juice. These usually consist of a crusher or shredder, or a combination of these, and from three to seven sets of three-roller mills. Electric drives for mills are now in quite general use, a separate motor for each three-roller mill and crusher being the rule. The electrification of milling machinery has resulted in great fuel economy and frequently in an excess of bagasse (Sec. 73). A modern mill installation consisting of a Fulton double crusher and fifteen-roll mill, all electrically driven, is shown in Fig. 5. A heavy duty "tandem" of mills such as this, with driving machinery, bed plates, carriers, and other accessories makes one of the largest and heaviest machinery combinations in use in any industry. Three such tandems of mills are not uncommon in the larger factories.

Fulton Iron Works Co.

Fig. 5. Motor-Driven Fifteen-Roller Mill and Crusher.

A great difference of opinion exists among engineers on the value of electric drives for mills. Tromp[1] in an extensive study of the advantages and disadvantages of total electrification of cane sugar factories, gives the following summary:

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[1] *Intern. Sugar J.*, July, 1940, p. 244; August, 1940, p. 277.

With "partial electrification" there are great advantages in operation, and the cost of installation and the ability to carry out the change gradually in existing factories are the deciding factors.

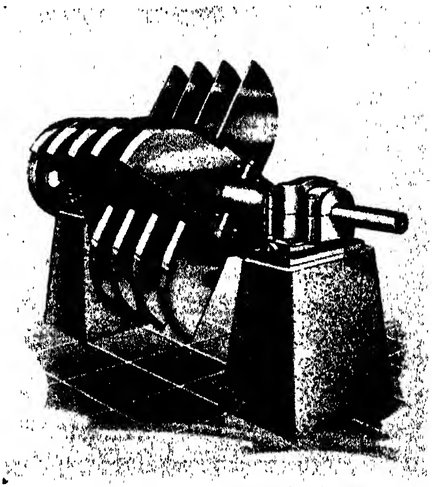
Electrical mill drive (total electrification) may mean lower running costs where new factories are concerned, but for existing factories having good reciprocating mill engines, there are opposing factors of considerable magnitude.

37. Preparation of the Cane for Milling. The milling process may be separated into two steps: (1) the preparation of the cane by breaking down the hard structure and rupturing the cells and (2) the actual grinding of the cane. The preparation of the cane is accomplished in several different ways:

(1) By revolving cane knives which cut the cane into chips but extract no juice.
(2) By shredders which tear the cane into shreds but extract no juice.
(3) By crushers that break and crush the structure of the cane, extracting a large proportion of the juice.
(4) By combinations of any or all of the above means.

38. Revolving Cane Knives. These are the most recent addition to cane-preparing machinery, but as they are always located ahead of any other apparatus in a grinding system they will be described first. Revolving knives first came into general use in Hawaii, but during the past fifteen years they have been widely adopted in Cuba and Puerto Rico and are also in use in the larger Louisiana installations. In fact they are standard equipment in most large milling plants throughout the world.

The earliest cane knives were comparatively slow-moving single sets with blades set about 6 in. apart placed at the base of the carrier with a view to leveling the cane mat. To these were added later a second set higher up on the carrier revolving at a speed of 450 to 500 rpm. with knives spaced 2 in. apart. In some countries multiple sets of knives are still used but in Cuba the practice is toward a single set revolving at the speed mentioned above and placed near the middle or toward the head of the carrier. The knives are generally so placed that they reach to within 4 to 6 in. of the carrier slats. They revolve in the same direction as the cane travels.

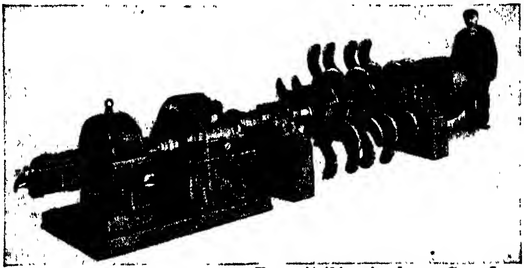


Squier Mfg. Co

FIG. 6. Swinging Type Cane Knives.

In Cuba where only one set of knives is used a cane "kicker" for leveling the cane is placed before the knives. This is a revolving shaft with rigid arms which have a clearance of about 2 ft. above the carrier.

Many different knife blades have been patented with the idea of modifying forms to facilitate replacement, sharpening, and to reduce breakage of blades. In general, the knives are placed spirally on a shaft which is driven by a separate motor, steam turbine, or high-speed engine. The Ramsay type swinging knives shown in Fig. 6 are bolted on the shaft in such a way that they are free to swing laterally so that they may turn aside if a hard object is encountered in the cane. The hinged arrangement also avoids vibration of the blades and consequent fatigue of the metal. A reversible type of blade has the front and back edge identical so that while the front edge is cutting, the back edge is being sharpened by the action of the cane sliding past it. The blades are reversed each week, and the self-sharpening action is thus continuous. Knives with serrated edges (Fig. 7) and some with the ends turned at right angles to form a hook are other forms which have found favor.



Farrell-Birmingham Co., Inc.

FIG. 7. Serrated Cane Knives.

The power required to drive the knives is much greater when the blades are dull. For average blade conditions a power consumption of about 1 hp. per ton of cane per hour is reported,[2] but Tromp[3] gives 2 hp. as a conservative figure.

According to Semple[4] the total power to drive knives and mills is no greater per ton of cane than for mills alone without knives.

The revolving knives cut the cane into small chips and therefore cannot be used with ordinary wooden-slat cane carriers. Overlapping steel-slat conveyors must be used to avoid the loss of chips, so that the installation of knives in existing mills is made more costly because of this need of carrier replacement.

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[2] *Proc. 2d Congr. Intern. Soc. Sugar Cane Tech.* (Havana, 1927), pp. 162–163.

[3] *Machinery and Equipment of the Cane Sugar Factory,* London, England, 1936, p. 121.

[4] *The Planter,* 80, No. 23 (1928).

Tromp[5] has given a detailed study of various blade designs, the extent of wear, and the power required for operation.

Knife sets are not used as the sole means of preparing cane for mills but are generally supplementary to crushers or shredders. They are particularly effective in supplying an even feed to the crusher or shredder and mills. They also give increased capacity to the mill train, the reported increases varying from 12 to 20 per cent. More effective use of maceration water and increased sucrose extraction are further advantages that users of knives agree upon.

39. Shredders. The first successful machine for preparing cane for milling was the National cane shredder, invented by Samuel Fiske and first used in Louisiana. This machine consists essentially of two shafts carrying conical cutting disks which dovetail or mesh into one another. These shafts are rapidly revolved in opposite directions and at different rates of speed. The cane is torn into shreds by the disks. The little juice that is separated in the shredding process is immediately reabsorbed.

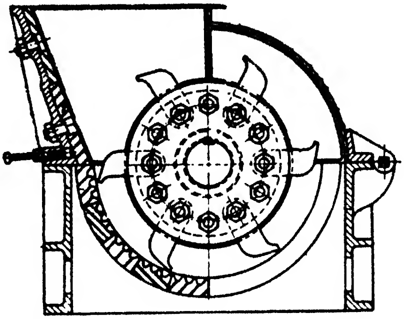

FIG. 8. Gruendler Cane Shredder.

The National type has been largely discontinued because of operating difficulties. It is still standard equipment, however, in Australia where it is generally preceded by two sets of slow-speed knives.[6] A recent study by Behne indicates that the National shredder performs very little useful work and could well be discarded in favor of the Searby shredder[7] or high-speed knives.

The Searby shredder is in rather general use in Hawaii and consists of a set of rapidly revolving swing hammers (1200 rpm.) which pass between anvil bars on which the cane is beaten and disintegrated into a fluffy mass. The Searby is generally used in conjunction with a crusher, the cane going from the crusher to the shredder and thence to the first mill. Tramp iron does not affect the Searby's operation. Most of the high sucrose extractions reported from Hawaii (98 per cent and above) are with mills having cane knives, crusher, and Searby shredder in conjunction with twelve-roller mills.

Another swing hammer type shredder which has recently come into quite general use in Louisiana and elsewhere in the western hemisphere is the Gruendler shown in Fig. 8. It is sometimes installed between the crusher and the first mill, though most installations are ahead of the crusher. The layout is such that it may readily be fitted into existing mill trains.

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[5] *Intern. Sugar J.*, December, 1937, and January, 1938.

[6] Behne, *Intern. Sugar J.*, November, 1940.

[7] *Intern. Sugar J.*, January, 1942, p. 12.

Increased extractions and increased capacities are reported, and the shredder is said to be particularly well adapted to improving the performance of old installations. High fiber cane and trashy cane are more easily handled than where the shredder is absent.

The Maxwell shredder is a single cast-iron roll with short hard-steel knife blades or teeth held in dovetails in the roll, which operates in conjunction with a crusher or first mill. The toothed roller revolves at a speed of 300 to 400 rpm., and the cane is combed into shreds as it issues from the crusher or mill.

The Maxwell shredder has been favorably reported on from Java[8] by disinterested observers. The shredder roll can readily be installed in existing equipment and is reported as giving the equivalent improvement of an additional three-roller mill.

The Morgan disintegrator (or shredder), designed to dispense with the crusher, has not been generally adopted. The shredder is very like an ordinary centrifugal pump with a solid steel disk some 5 ft. in diameter in place of a rotor. The cane is prepared for this disintegrator by two sets of revolving knives as it cannot be fed except in relatively small chips. The first of these Morgan installations was at Central Hershey, Cuba, where it operated in conjunction with a fifteen-roller mill at a capacity of 150 tons of cane per hour.[9] Another installation at Central Aguirre, Puerto Rico, is described by Norman Kay[10] as giving excellent results with a twelve-roller mill and 90 tons capacity per hour.

40. Crushers. Crushers, in general, consist of two or more deeply grooved rollers which crush or break the cane, expressing a large part of the juice (from 40 to 70 per cent, depending on type and rates of grinding). They are the main dependence for the preparation of cane for milling and with few exceptions constitute a part of all modern installations. The revolving knives and shredders already described are usually supplementary to crushers. Cuban factories formerly depended upon crushers only in their grinding trains, but with the increasing emphasis on higher sucrose extraction, together with the large capacities always desired in Cuba, revolving cane knives as preliminary preparation for the cane mat entering the crusher are now standard equipment.

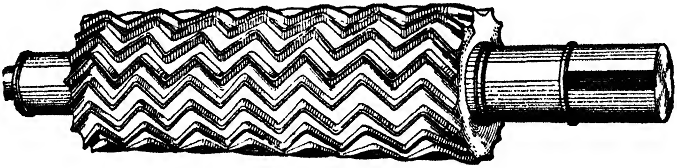

Fig. 9. Krajewski Crusher Roll.

The Krajewski crusher was invented by Krajewski in Cuba after the introduction of the shredder into this island. A crusher roll is shown in Fig. 9.

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[8] *Intern. Sugar J.*, January, 1929, p. 24.

[9] *Facts About Sugar*, 1930, No. 28, p. 786.

[10] *Facts About Sugar*, December, 1935, p. 471.

The rolls are driven slowly by gearing, but the recent tendency is toward faster rates of speed than for mill rolls. In the Hawaiian Islands Krajewski rolls have been grooved with Messchaert grooving (Sec. 43) to permit the free flow of juice.

A second type of crusher is the Fulton, which differs in its cutting or crushing surface quite radically from that of the Krajewski crusher. The cutting teeth are V-shaped and are arranged spirally ranging from 1.75 in. to 3 in. pitch, with the spirals working from opposite ends of the rolls. This is well shown in Fig. 10. There are also grooves separating the teeth into groups, and scrapers are provided to prevent clogging the teeth.

Fig. 10. Fulton Crusher Rolls.

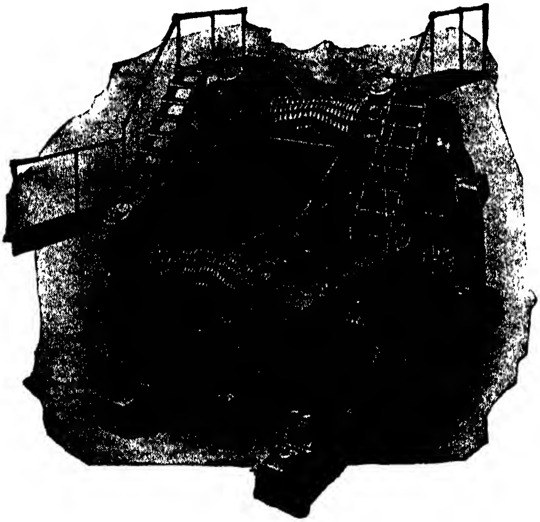
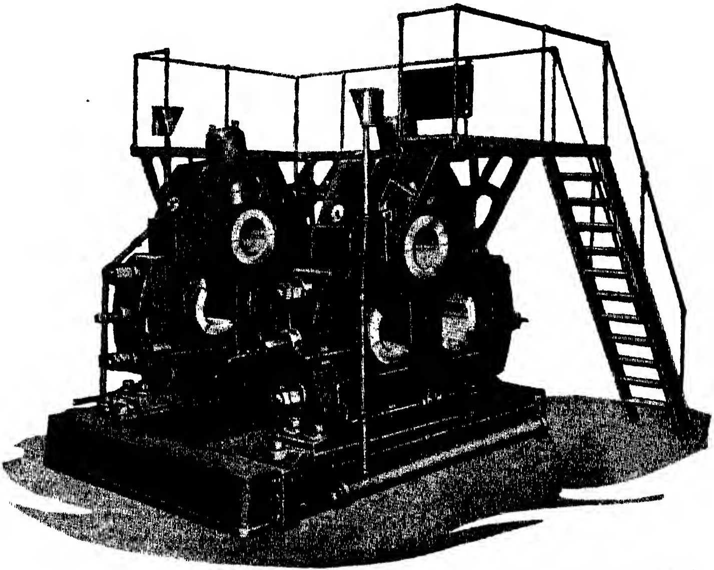


Fig. 11. Double Crusher.

A further improvement is the double crusher shown in Fig. 11, in which the cane passes through two crushers before entering the mills. The pre-crusher has much larger teeth, 3 in. or even 4 in. pitch being used with 42-in. diameter rolls. Triple crushers are in use in some large factories in Cuba. The double and triple crushers were generally installed before revolving knives were prevalent in Cuba. A marked tendency toward increase in peripheral speed of crusher rolls has been under way so that speeds of 45 or 50 ft. per minute are common as compared with 25 to 30 ft. in 1910.

Three-roller crushers are in use in Hawaii and are favorably reported upon. Such a crusher is essentially a three-roller mill with deep V-shaped annular grooving (1¼ in. to 1½ in. pitch). According to McAllep[11] about 75 per cent of the juice can be extracted by one three-roller crusher preceded by cane knives compared with 40 to 50 per cent for single crushers and 65 per cent for double crushers.

41. Mill Trains. Present-day mills always have three rolls, as is shown in Figs. 12 and 13. The bottom roll, where the cane enters, is termed the "cane roll," and that opposite, the "bagasse" or "discharge-roll." The two bottom rolls are usually rigidly fixed in position, and the top roll is controlled by a hydraulic ram and is so arranged that it may rise and fall or "float" with variations in the feed of the cane. Hydraulic pressure is applied to the bagasse roll by certain builders. A ram is shown in cross section in the top-roll cap in Fig. 13. The crushed cane, now called bagasse, is passed from one



Fulton Iron Works Co.

FIG. 12. Heavy-Duty Inclined Mill Housing.

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[11] *Proc. 2d Congr. Intern. Soc. Sugar Cane Tech.* (Havana, 1927).

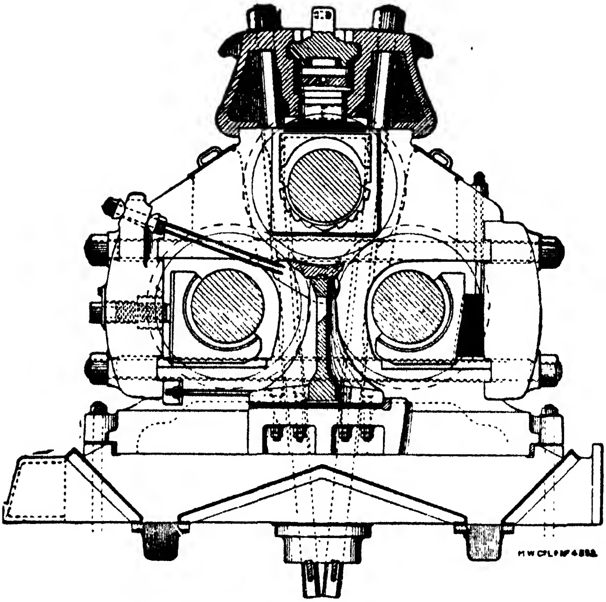

Fig. 13. Cross Section of Mirrlees-Watson Mill.

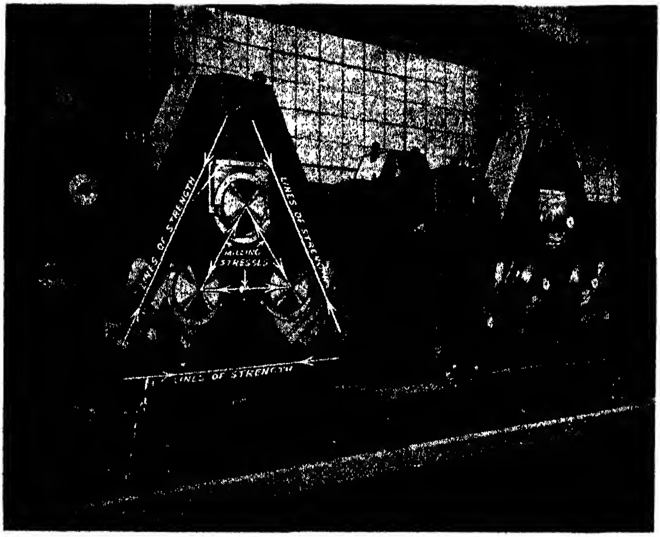

Fig. 14. Squier Housing with Stress Diagram.

pair of rolls to the next by a curved plate also shown in cross section in Fig. 13 variously termed a turnplate, knife, dumb turner, trash turner, etc., according to the country in which the mill is used. This is supported by a heavy steel turnplate bar.

The mill rolls are supported in massive castings termed housings or mill cheeks. The more recent models are shown in Figs. 12 and 14. The crown wheels by which the bottom rolls are driven from the top roll are shown in Fig. 5. The driving engine is connected through flexible couplings and gearing with the top roll of the mill.

A recent Fulton housing shown in Fig. 12 dispenses with the king bolts which one may note projecting above the top-roll caps of the Mirrlees-Watson mill shown in Fig. 13.

42. Recent Improvements in Mills. The past fifteen years have not seen as great changes in milling machinery as occurred during the years 1910–1925. The earlier period saw the steady increases in weight, size, and strength of mills and housings; the extension of mill trains from 6 or 9 rolls to 15 or more rolls with double and triple crushers; the adoption of heavier pressures and higher mill speeds; the electrification of mills; the addition of knives and other accessory apparatus. Recent steps have been largely refinements of these improvements.

One of these is the Squier triangular-stress steel housing shown in Fig. 14 for which many advantages are claimed. One feature is that the housing slopes from top to base in unbroken lines. It has no caps, and is divided into two pieces opposite the two lower journals so that the top may be readily lifted, exposing all three rolls and their bearings at once. The angle at the apex of the triangle is 70° instead of the usual 80°. This narrower included angle permits a much narrower turnplate; side pressures are reduced, and less danger of housing breakage is said to result. The freedom from ledges and other projections makes for greater cleanliness around the mills.

A further advantage of this type of housing is that it allows sufficient space around each of the shafts to mount roller bearings. This improvement, not yet in practical use, but a feature of some experimental mills, will result in power savings up to 50 per cent, will reduce lubricating costs, and will eliminate water cooling of bearings.

43. Mill Grooves. Mill rolls are cast of an iron mixture that will remain rough or acquire a "grain" with use to facilitate the feeding of the cane and bagasse. In general, all rolls have peripheral V-shaped grooving and the size of the grooves has been greatly increased to reduce the slipping of the roll upon the bagasse. Where formerly the grooves ranged from four to six to the inch, modern practice now prescribes a minimum of ½-in. grooves on the rolls of the last mills. The first and second mills carry grooves of 1 in. and the top roll is grooved longitudinally in the same way as the Fulton crusher roll (Fig. 10). The contained angle of the annular grooves is usually 55°. In some very large installations working to high capacities 1½-in. and even 2-in.

grooves are used on the earlier mills of the train. Such heavily grooved mills are practically the equivalent of the three-roller crusher used in Hawaii.

The Messchaert juice grooves shown in Fig. 15 are now in general use on feed rolls throughout the world. The grooves are usually ¼ in. wide, 1¼ to 1¾ in. deep, the pitch varying from 2 in. to 4 in., with 3 in. the general practice. The recent tendency has been toward deeper grooves and sharper angles. Special scrapers are used to keep the grooves free of bagasse. These grooves were developed in Hawaii about 1912 and have been the means of improving sucrose extractions and increasing mill capacities, since they provide a very free exit for the juice and practically eliminate slipping and the consequent mill vibration. A larger quantity of saturation water may be used without slipping or reduction of grinding capacity, and the mill inlets and outlets may be materially reduced in size, in some instances nearly one-half.

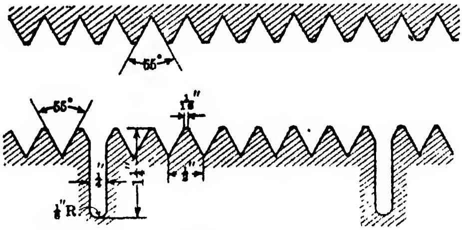

Fig. 15. Messchaert Mill Grooves.

Hawaiian practice, as well as that in some Cuban mills, also provides the discharge or bagasse roll with Messchaert grooves ⅛ in. wide, the moisture content of the bagasse being materially reduced by this grooving of the discharge roll.

Another invention is the Hind-Renton grooving which is in favor in Hawaii. The pitch is two grooves per inch and the groove angle is 30° instead of the usual angle of about 55°. It is claimed that the juice flows out through the lower part of the groove and that the bagasse wedges itself above it as a boot in a bootjack.

44. Pressure on Rolls. The pressure on the top roll is usually regulated by means of hydraulic rams (see Fig. 13), except in Java and Australia.

The hydraulic pressure applied on the top roll varies with the length of the roll, the strength of the mill, and the quantity of cane to be ground in a given time. The pressure is also varied with the position of the mill in the series or tandem.

Practice varies as to the loading, but this approximates 150 to 250 tons hydraulic pressure on the crusher and up to 500 tons or above on other mills. A Cuban mill consisting of cane knives, single crusher, and seven three-roller mills (rolls 7 ft. in length) with a record of nearly 250 tons of cane per hour (Central Vertientes) reports pressures as follows:[12] crusher 170–250 tons;

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[12] Scharnberg, *Facts About Sugar*, Jan. 14, 1928.

first mill, 500; second mill, 450; third mill, 480; fourth and fifth mills, 500; sixth mill, 520; and seventh mill, 550 tons. There is much difference of opinion and practice as to whether higher pressures should be put on the last mills (as in the instance cited) or whether the earlier mills should get the heaviest pressure.

Many engineers advocate the heaviest possible pressure on the first mill in order to rupture and disintegrate the cane as thoroughly as possible to prepare it for the maceration water, but others insist that the last mill pressure should be highest to insure as low a moisture content in the bagasse as is practicable. Still others carry full pressure throughout the whole train.

The hydraulic regulation of the top roll has a twofold purpose: (1) the protection of the mills from serious damage if a piece of metal should fall into them or if the feed of cane were too heavy, and (2) the regulation of the opening between the rolls to suit variations in the quantity of cane or bagasse passing through them. The hydraulic pressure now carried is so great that a piece of metal may sometimes bury itself in the shell without raising the roll sufficiently to afford protection.

The use of very strong cast-steel housings or mill cheeks has enabled manufacturers to dispense with king bolts, Figs. 12 and 14, or to use very short bolts that extend only part way through the housing (Honolulu Iron Works). This arrangement permits the use of hydraulic rams of large diameter with consequent increase of life in the packing leathers.

45. Mill Settings and Speeds. The mill setting, or the adjustment of the openings between the rolls and the relation of the turnplate to the rolls, varies greatly in different factories and with the rate of grinding, the quality of the canes, and the grooving of the rolls. The setting is also somewhat modified when hydraulic pressure is not used on the top roll, or when it is applied to the bagasse roll.

It is outside the scope of this chapter to give figures on mill settings since these vary so widely with differing conditions that figures would be valueless without all correlative data. Sharnberg[13] gives full details for the single crusher and seven three-roller mill combination referred to above showing openings varying from 2½ in. on the crusher (3 in. pitch) to about ⅜ in. on the discharge roll of the last mill, these openings being measured from the tip of one tooth to the base of the corresponding groove. It may be noted that the opening between the turnplate and the top roll is gradually enlarged from the inlet to the outlet end. This enlargement permits the bagasse to expand after the first pressure and facilitates the passage of this material and the escape of the juice.

The speed at which a system of mills is driven is usually expressed in feet per minute of the periphery of the rolls. Practice varies greatly in different countries in regard to the speed of the rolls. It is usual to have the speed increase successively from the first to the last mills, but this is not invariably

[13] *Loc. cit*

the rule. The speed ranges from as low as 12 ft. or less in Java to over 40 ft. per minute.

The Cuban practice has been toward higher mill speeds with 30 ft. per minute the general rate in good modern mills. As already stated, crusher speeds range above 40 ft. per minute, peripheral speed.

46. Cane and Bagasse Carriers. The cane elevator is generally driven by independent engines or motors rather than from the milling machinery. This results in greater uniformity in the delivery of the cane to the crusher and thus gives a more even feed throughout the whole set of mills.

Between the different mills are "intermediate" carriers, the commonest in Cuba being an endless-chain "apron" or wooden-slat conveyor. These conveyors tend to foul through the souring of juice on chains, slats, etc., and are a source of mill stops because of breakage. This type has been entirely superseded in Hawaii, and to some extent in Cuba, by either the Meinecke chute or Ewart drag conveyor. The Meinecke type (Fig. 16) consists of a covered steel chute rising at an angle of about 35° from the discharge roll of the mill and then dropping sharply to the feed opening of the next mill. The bagasse is forced up the chute by the succeeding bagasse that is discharged from the mill and falls into the next mill by gravity. The maceration water was formerly applied at the top of the chute, but it is now put on just as the bagasse is emerging from the mill. An objection to the Meinecke chute is that the feed to the following mill is irregular. The blanket of bagasse breaks and falls in lumps causing choking.[14]

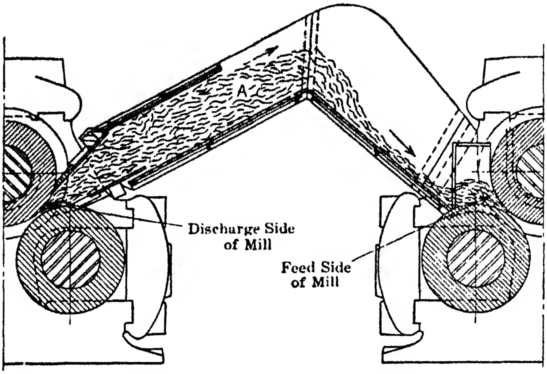

FIG. 16. Meinecke Intermediate Bagasse Chute.

The Ewart carrier is a steel trough to which the bagasse is moved by an endless-chain drag traveling above the trough. Both the Ewart and Meinecke methods make for better mill sanitation and are easier to maintain than the older type slat conveyors.

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[14] *Intern. Sugar J.,* July, 1930, p. 383.

47. Fulton Force-Feed Intermediate Carrier. An intermediate carrier of recent design is shown in Fig. 17. The carrier is on short centers and is of the pushing type, with the downward slope forming a tangent to the curve of the first roll. The under frame of the carrier has a drip pan for improved cleanliness under the mills.

Feed rollers and pushers as accessories to bagasse carriers are used to avoid difficulties in the feeding of mills due to heavy imbibition and finely disintegrated bagasse. The feed rollers consist of steel drums with angle irons attached longitudinally which are placed immediately in front of the feed roll above the carrier. Feed rollers are widely used in Java. A pusher is a bar driven by an excentric or crank. The function is not to push the bagasse into the mill, but to make it available in the right place. They are common in Australia and South Africa.[15]

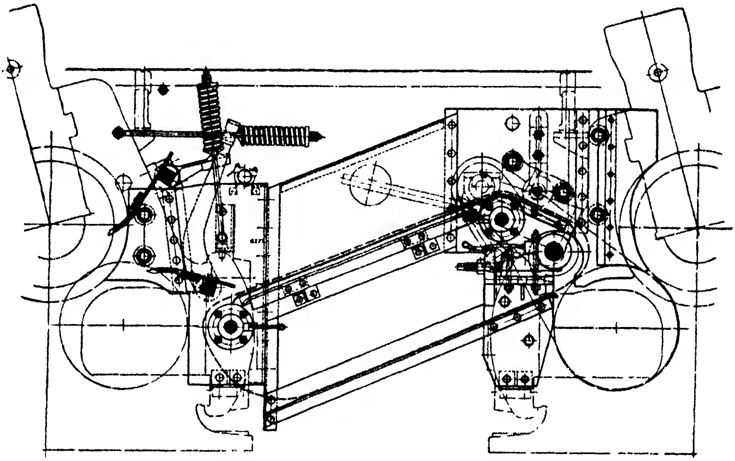

Fig. 17. Fulton Force-Feed Intermediate Carrier.

48. Extraction of the Juice. The mechanical equipment of the milling plant has been described, and the crushing of the cane and the extraction of the juice will now be considered.

Under the usual conditions, the cane parts with more than 60 per cent of its weight of juice in its passage through the crusher and the first mill. In other terms, upwards of 70 per cent, sometimes more, of the sucrose in the cane is extracted. The woody residue of the cane from the first and subsequent mills is termed "bagasse" by the Americans and "megasse" by the English. As the bagasse progresses through the mills it is compressed more and more, each time parting with some juice, until finally under good working conditions it leaves the mills containing 50 per cent of woody fiber.

[15] *Intern. Sugar J.*, February, 1932, p. 62.

Few factories conduct their mill work as above without adding water to the bagasse after each mill to dilute the remaining juice and thus increase the extraction. This use of water is termed saturation, imbibition, or maceration.

49. Maceration, Imbibition, Saturation. The maceration process, properly speaking, is one in which the bagasse is passed through a bath of thin juice or water to dilute the remaining juice before regrinding. This process is little used except in Australia, and except in that country the word "maceration" has come to have the same meaning as saturation and imbibition.

The International Society of Sugar Cane Technologists' adopted the term "imbibition" as standard for the process in which water or juice is sprayed on the mills. However, the terms "saturation" and "maceration" are more common in the western hemisphere, and it is doubtful whether the newer term will replace the commoner terms promptly. In this chapter imbibition and saturation are used interchangeably, and the term maceration is reserved for "true maceration." The saturation may be single, double, or compound. Single saturation uses water only; double saturation uses water and the thin juice from the last mill or mills, whereas compound saturation uses water and the juices from two or more of the last sets of mills, applying these juices separately to the earlier mills of the train.

The water is sprayed upon the bagasse, in single saturation in a nine-roll plant, as it emerges from the second mill. In double saturation, the water is applied as above, and the thin juice extracted by the last mill is pumped back upon the bagasse as it emerges from the first mill. Compound saturation may be practiced when four or more mills are in the series or tandem. This method is illustrated in the diagram, Fig. 21. The thin juice extracted by the third mill is pumped back upon the bagasse from the first mill and that of the fourth mill upon the bagasse from the second mill. Water is applied to the bagasse from the third mill. The juice from the crusher and the first and second mills enters into the manufacture. The application of the water is modified to meet the needs of other combinations of rolls exceeding twelve in number.

Saturation water is sometimes applied to both the upper and lower side of the blanket of bagasse. The application to the upper surface of the bagasse does not usually penetrate the lower layers. When the water is applied from above, it is preferable to do so just as the bagasse emerges from between the rolls so that it will absorb the water in expanding. In this way it acts as a sponge that has been compressed.

General practice is to apply the water to the bagasse as it issues from the two mills next from the last one. A method of double saturation in vogue in Cuba is shown in Fig. 18 for a double crusher and fifteen-roll mill. It will be seen that the water is sprayed on the bagasse coming from the third and fourth mills; that the juice from the fourth and fifth mills is mixed and sent back to the bagasse coming from the first and second mills. Such a system is simpler than compound saturation as it requires only one set of pumps, strainers, etc., for the thin juice, but it is not so efficient as compound saturation in which the juice from the fourth mill would go to the first mill bagasse and the juice from the last mill to the bagasse from the second. Compound saturation is general in Hawaii and Java and many Cuban factories also make use of the system. The introduction of strainerless juice pumps has greatly simplified the process of compound saturation.

There is always danger of fermentation in the rehandling of thin juices, and great care must be taken to prevent souring and fouling of juice channels where these methods are used. The mills must be shut down at frequent intervals for a thorough cleaning. Few Cuban factories can spare the time to wash down mills and tanks thoroughly more often than once a week.

The percentage of saturation water used varies widely with the country, the capacity of the mills, the character of the cane, and the relative costs of fuel and sugar. The introduction of juice grooves and heavier grooving on Cuban mills has permitted the addition of more water with larger capacities, whereas various fuel economies, notably through electrification, have made the use of this increased saturation economical. Twenty-five per cent water is now common practice in the larger mills where only 10 or 15 per cent was used formerly. The average for the island is still about 15 per cent. Hawaii and Australia range up to 35 and 40 per cent, and Java uses 15 to 20 per cent.

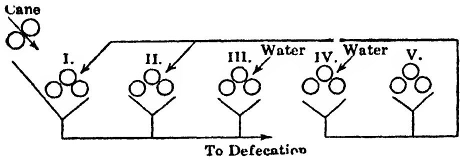

Fig. 18. Diagram of Double Saturation.

50. Bath Maceration. Australia uses "true maceration" or "bath" maceration. A description of the process by Harmon[16] follows:

> The system of hot maceration fluids and of diffusion baths or boots is one of the most distinctive features of Australian practice. The mills are spaced up to 60 feet apart and the bagasse from any one mill falls down a chute into a circular boot, kept overflowing with the macerating fluid. A large drum in this boot takes the chain of the intermediate carrier which is of the rake type with fingers above as well as below the chain attachment. A compound maceration system is employed but the maceration fluids are heated almost to boiling point before being sprayed on at the rollers. Varying quantities of maceration water are added, but in an efficient quadruple mill about 40 gallons water are added per ton cane (i.e., 18 per cent on cane). Steam is also added direct to the diffusing boots and the temperatures of the mill-expressed juices other than the first are about 170° F. The saturated bagasse takes in most cases up to 10 minutes to travel from mill to mill and, although the crushing pressure is relatively low, as compared with Hawaii, this combination of diffusion (really lixiviation) and crushing results in an extraction of about 95 per cent on the average, but ranging up to 98 per cent in the best cases. The "lost juice" per 100 fiber is about 25.

[16] *5th Cong. Intern. Soc. Sugar Cane Tech.* (Brisbane, 1935), p. 45.

A full description and flow sheet of a twelve-roller mill using bath maceration in a compound system shows the rather complicated set of tanks, heaters, pumps, and strainers required for this method of maceration.[17] The system has also been tried in Java with good results, and at least one factory in Natal (South Africa) has adopted it.

51. Hot or Cold Saturation Water. The saturation water may be used hot or cold. The matter of the quality of the water and some fuel economy usually dictates the use of warm water. The hot water is derived from the surplus of return water, from evaporator coils, etc. (so-called "sweet water") over the requirements of the steam plant, and it is therefore distilled water. Those of the heat units that pass with the saturation water into the juice are largely economized. There is also a slightly increased evaporation of moisture from the bagasse in transit to the fires, as compared with that obtained with cold saturation.

The rupturing of the cells may be caused by two means, by pressure and by heat. The cells are killed by heat (70° C.), and the enveloping membrane then becomes permeable so that when the cell dies the juices may diffuse and mix with the macerating fluids. Therefore hot maceration water should theoretically give greater extraction than cold but tests by many investigators lead to the conclusion that the mill extraction is practically the same whether the maceration water be used hot or cold, assuming that the water is sprayed in the ordinary way and bath maceration is not applied.

Behne[18] has shown that the time factor and the insulating nature of bagasse both tend to decrease the effectiveness of the influence of the heat, and he finds that quantities of intact cells can be found in the bagasse in spite of mill pressures and high temperatures. Another factor which has been found to hinder the imbibition is the presence of bubbles of air in the broken cells.[19] All these factors combine to prevent the complete penetration of the added water.

An argument against hot imbibition is advanced by Browne and Phillips[20] since "it causes a large increase in both methoxyl and uronic acid compounds (pectins) in the dry juice solids . . . These results bear out the observation of milling practice that cane juices obtained by hot maceration are richer in gums and hence harder to clarify." These writers also corroborate the adverse effect (long known) of lime or alkaline water on cane fiber. However, in spite of these theoretical arguments against hot saturation it is the more common practice throughout the world.

52. Disintegration of the Cane. Modern mill practice is toward disintegration of the cane as completely and as early as possible with a view to preparing it for the penetration of the maceration liquids. For this reason the use of cane knives, shredders, multiple crushers, and combinations of these has increased so rapidly. As stated under the section dealing with hydraulic

[17] Marriott, *Intern. Sugar J.*, December, 1935, p. 468.

[18] *Intern. Sugar J.*, January, 1938, p. 17.

[19] Khainovsky, *Proc. 3rd Congr. Intern. Soc. Sugar Cane Tech.* (Java, 1929).

[20] *Intern. Sugar J.*, November, 1939, p. 430.

pressures a large proportion of engineers use the heaviest possible pressure on the first mill of the tandem in order to break up the cane thoroughly for the maceration process.

If all the juice cells of the plant are ruptured in the shredding or crushing process and the first grinding, it is evident that when the water is applied to the bagasse, if the time element be sufficient, it will penetrate it and dilute all the juice it contains. The time element, however, in practice is so short, and so many of the cells escape rupture, that only the superficial portions of the juice are diluted.

Spencer demonstrated by laboratory experiment as follows, that very long contact with water is required for the dilution of all the residual juice in the bagasse. A sample of bagasse from thoroughly crushed cane was heated with water in the proportion of 5 parts of bagasse to 45 parts of water, and the temperature was maintained near the boiling point one hour. The water and bagasse were then thoroughly mixed and the dilute juice was strained off, using moderate pressure. The residual juice was expressed with a laboratory cane mill, using very heavy pressure, and the two samples of juice were separately analyzed. The percentage of sugar in the juice extracted by the mill was very perceptibly larger than that in the juice obtained by straining. This experiment was repeated several times with like results. These experiments show that it is not practicable in milling to dilute all the juice in the bagasse with the saturation water, and that a factor depending upon the time element and the efficiency of the mills must be applied in estimates of the water actually utilized.

53. Composition of the Mill Juices. The juices from the crusher and each of the succeeding mills differ according to the pressure and the extent of saturation. With continued crushing there is a reduction in the Brix, in the polarization, and in the purity, with a consequent increase in the non-sugars both organic and inorganic. The following figures are from actual milling with compound saturation on a fifteen-roller mill and double crusher:

Source of Samples	Brix	Polarization	Purity
Double crusher	17.16	14.50	84.50
First mill			
Front roll	17.08	14.12	82.67
Back roll	16.13	13.06	80.97
Second mill			
Front roll	7.63	5.83	76.41
Back roll	9.37	7.31	78.01
Third mill			
Front roll	5.04	3.73	74.01
Back roll	6.14	4.54	73.94
Fourth mill			
Front roll	3.00	2.18	70.60
Back roll	4.52	3.26	72.12
Fifth mill			
Front roll	1.31	0.88	67.18
Back roll	2.55	1.78	69.80

In a comparison of crusher juices with whole-mill juices with saturation Fort and McKaig[21] found that there was a 20 per cent increase in total non-sugars, but that the non-sugar constituents were not all increased to the same degree. Ash increased only 14 per cent and organic non-sugars 23 per cent. Non-protein nitrogen, one of the most objectionable constituents, increased 57 per cent. Similar results have been reported by other observers. King[22] shows graphically the increase in colloids (by dye number) in an eighteen-roller mill with and without saturation. The saturation causes an increase in the dye number from 400 to 2400, whereas without saturation the dye number increases from 400 to 600 from crusher to sixth mill. Salinas[23] gives results for a double crusher and fifteen-roller mill which show the much greater reduction in purity where saturation is used. He also shows that by separating juices of the crushers and first two mills (on which no dilution water has been sprayed) [A juice] from the last four mills which contain all the dilution water [B juice] there is a great difference in purity (87.2 for A juice and 80.2 for B juice). Also there is twice as much reducing sugars (dry basis) and three times as much ash (dry basis) in the B juice. Dye numbers indicate more than twice the colloids in B than in A.

Summing up the subject of the composition of the extracted juice, the more sucrose that is extracted (by ordinary means) the greater the proportion of undesirable material that will accompany the sucrose. It is not only a question of lower purities but also of the character of the impurities which go to reduce the purity.

All the soluble constituents in the cane are not extracted to the same extent during milling. Honig[24] found sugars, potash, and phosphates are extracted to approximately the same extent. Lime, magnesium, and soda are also largely extracted though not to the extent that sucrose is. Of the silica, iron, and alumina, the nitrogenous compounds, and the waxes, a greater proportion remains in the bagasse than goes into the juice. Fort and Holmes making a similar study of Louisiana juices[25] found much the same general relationship, but in no case did they find any constituent extracted to the extent that sucrose is. They grouped the extraction of the non-sugars as follows: potash, phosphates, and combined organic acids about 80 per cent; lime magnesia and sulfates about 70 per cent; nitrogen 48 per cent; iron 38 per cent; and silica 8 per cent. These workers found considerable difference in the extractibility of the mineral constituents in different varieties.

54. Influence of the Structure of the Cane on Milling. The structure has a marked influence on the mill results. With very efficient milling certain canes yield bagasse containing 50 per cent fiber and 45 per cent of moisture; others, when ground with the same mills and mill setting and apparently the same efficiency, give bagasse containing 45 per cent of fiber and 50 per cent

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[21] *U.S.D.A. Tech. Bull. 688*, 1939, p. 4.

[22] *Ind. Eng. Chem.*, August, 1931, p. 963.

[23] *10th Annual Proc. Assoc. Cane Sugar Tech. Cuba* (1936), p. 63.

[24] *Intern. Sugar J.*, July, 1934, p. 272.

[25] *Sugar Bull.*, Nov. 15, 1937.

of moisture. These conditions have been observed in Java when grinding the varieties 247 and 100.

In his study of "The Influence of the Structure of the Cane on Mill Work,"[26] Noël Deerr says:

> It not infrequently happens that while the fiber remains of constant percentage, the extraction varies largely, the milling conditions remaining the same. Such variation can be readily understood on the assumption that while the total amount of fiber remains the same, its distribution between the pith and rind varies, an increase in the proportion of the latter being accompanied by a decrease in the extraction.

The percentage of fiber has a marked effect on the extraction, the greater the amount of fiber the less the extraction for the same milling efficiency. In Louisiana during the past fifteen years the fiber percentage has risen from 9 or 10 per cent to 15 or 16 per cent because of the change in cane varieties. In consequence Daubert[27] has shown that average extraction figures for mills having knives, crusher, and nine-roller mills have reduced from 93.76 per cent for the 10 per cent fiber canes to 89.17 per cent for the 16 per cent fiber canes.

Tromp[28] gives an interesting tabulation showing the effect of fiber on extraction where milling efficiency, as measured by lost juice per cent fiber, is the same. For 10 per cent fiber the extraction would be 95 per cent, and a reduction of about 0.6 per cent occurs for each 1 per cent increase in fiber so that for 16 per cent fiber the extraction with the same milling efficiency is 91.40 per cent. These theoretical figures by Tromp exactly bear out those given from actual practice in Louisiana by Daubert.

Adhering leaves and the immature tops of the stalks, which remain because of careless harvesting, increase the quantity of fiber that must be passed through the mills and adversely affect the extraction. Increased saturation is necessary to overcome this influence.

55. Efficiency of Milling. The efficiency of milling is generally expressed in terms of the percentage of the total sucrose in the cane that is extracted in the juice (sucrose in juice per cent sucrose in cane). This figure is known as the sucrose extraction or more briefly the extraction.

(Strictly speaking the figure is the polarization extraction, since it is generally based on polarization, not on true sucrose figures. However, for practical purposes it approximates sucrose extraction and will probably continue to be called that in most parts of the world, although the International Society of Sugar Cane Technologists rightly condemns the use of "sucrose" for "polarization.") Hawaii used to average 97.5 per cent with higher figures reported by many factories, but grab harvesting (see Sec. 17) with the resulting dirty cane has brought this figure down to 95.5 per cent, which is also the average for Australia.[29] Java averages about 94 per cent and the better mills

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[26] *Hawaiian Sugar Planter's Expt. Stat. Bull. 30,* **41.**

[27] *Sugar Bull.,* No. 1, 1938.

[28] *Intern. Sugar J.,* 1940, p. 69.

[29] **Doolan,** ***Intern. Sugar J.,*** **October, 1939, p. 382.**

in Louisiana about 93 per cent. The average for Cuba is 93 per cent with more efficient mills reporting 95 per cent and even 96 per cent.

Formerly the percentage of "normal" juice obtained from the cane was considered the criterion of the efficiency of milling, but it is now universally recognized that the sucrose (polarization) extracted per cent sucrose (polarization) in cane is much more valuable. With accurate cane and saturation water weights and the exact methods of juice analysis so generally in use, the sucrose (polarization) extraction figure is extremely reliable. The only estimated figure used in the calculation is the weight of the bagasse, and it has been hoped that weighing this material would prove practicable. Several methods have been advocated, none of which has been put to practical use. A recent idea, described by Waddell,[30] is made up of a tilting arrangement carrying two balanced hoppers into which the bagasse from the carrier is alternately fed. When the weight of the bagasse in one hopper overcomes the weight of some steel balls in tubes the whole system tilts, the bagasse is fed to the second hopper and discharged from the first while the steel balls roll to the other end of the tubes. This device is too recent to have been tested thoroughly.

The sucrose (polarization) extraction is adversely affected by the fiber content (see Sec. 54), but makes no allowance for this in the reported figures. Noël Deerr suggested that all extractions for comparative purposes be corrected to a fiber content of 12.5 per cent (to be called "reduced extraction," see Sec. 794) and this was adopted by the International Society of Sugar Cane Technologists.[31]

Another figure which takes into account the fiber is the "lost juice per cent fiber" which was developed in Java. Noël Deerr in a detailed study [32] developed a system for comparing the operating efficiency of different mills, in which he took into consideration the quantity of added water and the number of milling units as well as the fiber. These control calculations will be considered in the analytical section of this book (Chapter 35).

56. "Juice Curves": Juice Brix Diagram. B. P. Luce [33] and W. E. Smith [34] have suggested a diagram of the Brix of juices from the different mills as a guide to the performance and this has been found to be of value. The usual practice is to take samples of juice from the discharge rollers throughout the mill train, although some prefer to take samples both of the front rolls and discharge rolls, making two diagrams. Sampling is timed so that the same part of the blanket of bagasse is passing through the successive rolls as each sample is taken, and conditions during the test should be as uniform and normal as possible. In general, the diagram should show a down-

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[30] *Sugar News,* **20**, 187 (1939); *Intern. Sugar J.,* August, 1939, p. 314.
[31] *Proc. 5th Congr. Intern. Soc. Sugar Cane Tech.* (Brisbane, 1935), p. 850.
[32] *Int rn. Sugar J.,* October, 1931, p 480.
[33] "Audit the Tandems," *The Planter,* June 1, 1929.
[34] *Hawaiian Sugar Tech.,* 1929.

ward sloping line from crusher to last mill (see Fig. 19). Any zigzag tendency is obviously due to incorrect work by one or more mills.

Practical interpretations of the diagrams have been given by Elliott[35] and Haldane.[36] Elliott suggests checking the density of the juice from the last mill with the density of the theoretical residual juice in the bagasse (Brix in bagasse divided by Brix plus moisture in bagasse). The ratio should be 55 to 80, but may be as low as 25 with poor milling. Haldane gives methods used in India for comparison with basis curves calculated by formulas of Noël

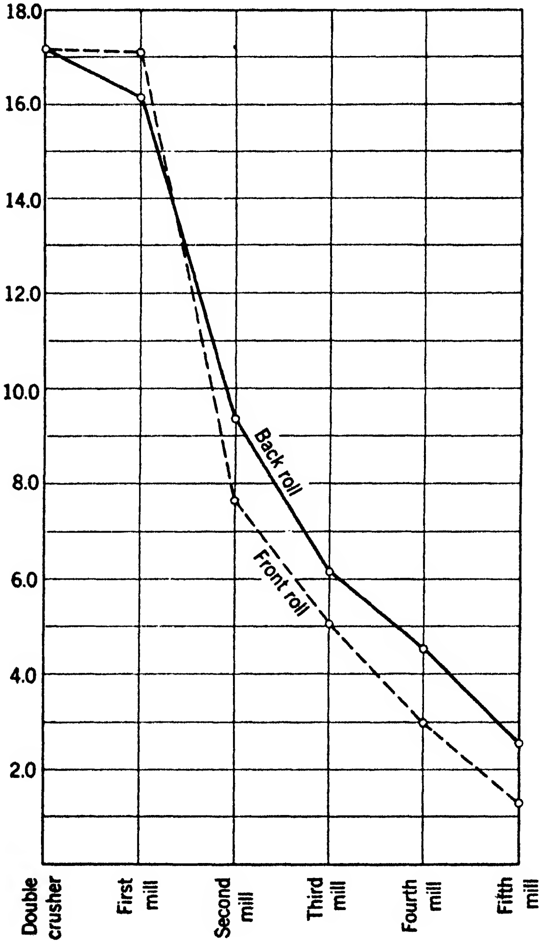

Fig. 19. Juice Brix Diagram.

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[35] *Intern. Sugar J.*, June, 1934, p. 230.
[36] *Intern. Sugar J.*, August, 1934, p. 306.

Deerr. In this method the total juice from each mill is sampled, not that from the back rollers. Jackson[37] gives the technique of "Wet Milling Tests" in Hawaii by which not only the Brix from each mill is determined, but the bagasse is also sampled and the extraction of each mill determined. The opinion is given that the extraction figures are of more value than the Brix diagram.

Figures for juice density curves in Mauritius[38] and in Jamaica[39] have also been published.

57. Straining the Mill Juice. The juices as they come from the mills must be strained free of bagasse and larger particles of bagacillo (fine bagasse) first, in order that the ordinary style pumps may handle them, and secondly, so that as much of the bagasse fiber may be kept out of the process as possible, because of the solvent action of lime on this fiber.

Browne and Phillips[40] showed that liming unstrained juice caused a considerable increase in both uronic acid and methoxyl content (pectins) which they supposed to be due to the action of the lime on the suspended fiber particles in the unstrained juice.

58. Coarse Strainers. The commonest form of juice strainer in use in the principal cane countries consists of perforated brass plates whose surfaces are kept clean by means of moving slats or flights similar to a squeegee. The flights are attached to an endless link belt. They brush the particles of bagasse from the surface of the plates and elevate and deliver them onto the bagasse carrier from the first mill. The holes in the strainer plate are round and about 0.04 in. in diameter, or the plate is perforated with about 324 round holes per square inch. If the holes are smaller the plates must be correspondingly thinner and are likely to break.

This type of strainer has certain advantages such as capacity and compactness, but it also has some very marked disadvantages. The wear of the perforated sheets is very great, and in order to use thick sheets the perforations must be larger than would otherwise be necessary. The link belt soon becomes very foul, and undoubtedly this is a source of sugar losses which escapes consideration, since the chemical control begins with the strained juice. The belt and flights must be frequently cleaned and steamed. The juice canal and tanks under this strainer cannot usually be located conveniently, thus making it difficult to keep them in a thoroughly sanitary condition. The elevator also fouls quickly from juice drippings.

Another type of strainer, the "grasshopper," is not generally used outside of Java. This strainer patented by Van Raalte, a Dutch engineer in Java, is a modification of the grasshopper sugar conveyor (see p. 238). In this modification, the bottom of the conveyor is composed of brass plates perforated with round holes of approximately 0.02-in. diameter. A canal under the

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[37] *Intern. Sugar J.*, January, 1935, p. 19.

[38] *Avice Revue Agricole*, 1935, p. 19.

[39] Nurse, *Intern. Sugar J.*, January, 1938, p. 36.

[40] *Intern. Sugar J.*, November, 1939, p. 431.

strainer collects the juice. The fine bagasse, "cush-cush" of the English factories, is discharged at the end of the strainer, in a thoroughly drained state, and is returned by an elevator to the mills for regrinding. A very marked advantage of this strainer is the ease with which it may be kept in a sanitary condition, thus reducing the loss of sugar. Thin sheets with very fine perforations may be used. There is very little wear on the sheets. A straining surface of approximately 2 ft. by 16 ft. is sufficient for 1000 tons of cane per twenty-four hours.

Recently much attention has been given to the certainty of sugar losses in and around the juice strainers to try to determine the extent of these losses and to correct them if possible. This subject of mill sanitation will be dealt with in a later section (60), but it may be mentioned here that modern improvements in straining equipment and the use of strainerless pumps have aimed at cleanliness and the prevention of souring in and around the mills.

Recently built mills in Cuba have located their strainers and juice troughs in large-sized, tile-lined pits which permit free access to all parts of the strainer and mechanism for cleaning. These installations can unquestionably be kept in much better sanitary condition than the older inaccessible types. The so-called Tuinucu strainer has screens that are removable from the sides, permitting easier replacement and readier cleaning.

59. Fine Straining of the Juice. The strainers so far described have been of a type that removes only the coarser particles of fiber. With the introduction of strainerless juice pumps it has become possible to omit all straining of juices returned to the mills and to strain only the juice going to process. Many mechanical screens have been developed for this purpose and these can be located above the mills so that the strained fiber can fall directly on the bed of bagasse. The strainerless juice pumps (also called chokeless pumps) are especially designed centrifugal pumps of the type used for drainage and dredging purposes. A reciprocating pump of the design used for sewage has also been used. Unstrained juices returned to the mill by this type of pump are distributed on the bagasse by a special trough fitted with a screw conveyor to spread the juice evenly. The Peck revolving juice strainer (Fig. 20) consists of a motor-driven drum revolving at 2 rpm. covered with woven wire cloth (100 by 100 mesh) through which the strained juice falls to the tray, *R*, and is thence conducted to the strained juice compartment, *K*. A part of the raw juice flows around the cylinder into the outer compartment, *K*, where it rises to the level of the overflow pipe, *M*, and forces itself through the screen because of the slight head. This strained juice is then also discharged into *K*. As the drum revolves the strained material on the outside of the screen falls into the tank and is caught by the paddles, *H*, and carried to the discharge lip, *P*, where suitable scrapers, *O*, engage the paddles and remove the strained material from the system. Arrangements are provided for steaming out the strainer while in operation.

Other mechanical strainers used by some factories after the coarse straining are the Hummer and Mitchell screens of the type used in screening refined sugar. These consist of inclined screening surfaces which are either mechanically or electrically vibrated to keep the screening surface clear. These screens and the Peck strainer require too much headroom to be used for direct flow of the juice by gravity from the mills, but as stated above they may be placed at any convenient point above the mills.

A rather different type of strainer, which has no moving parts, was invented by Coulter in Hawaii. The juice flows down over an inclined screen (64 mesh) and the bagacillo is retarded on its way down this screen by a friction blanket made of canvas, the ends of which are cut vertically for about 9 in. into 2-in. straps which are weighted with lead. Because of this loaded blanket the bagacillo builds up to a depth of 3 or 4 in. before discharging over the lower end of the screen.[41] The screen is made double, back to back. The same device as a single screen is used as a juice distributor for saturation purposes.

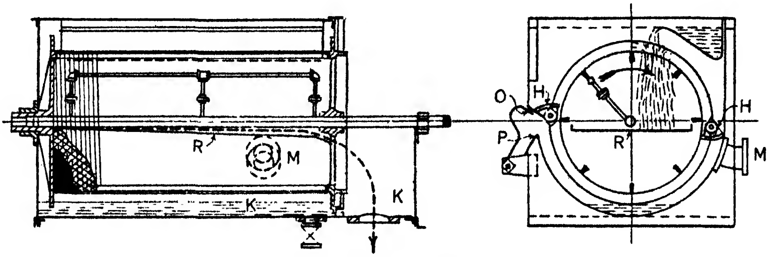

Fig. 20. Peck Revolving Juice Strainer.

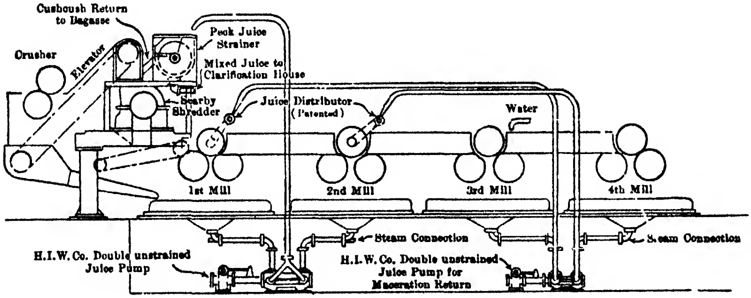

Fig. 21. Installation of Strainerless Juice Pumps and Compound Saturation.

This fine straining of juice has the advantage of removing the bagasse fiber before it has undergone the solvent action of heat and lime, but it also has the disadvantage of making the cachaza or mud scums harder to filter-press because of the lack of this fiber which acts as a filter aid. The addition of more lime has been suggested but this is known to be detrimental to the character of the press juices. An artificial filter aid, such as kieselguhr, or the

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[41] McCleery, *Intern. Sugar J.*, July, 1936, p. 256.

return of some of the "cush-cush" or bagacillo direct to the mud tank have been advocated as a solution of the problem. This latter method is used in conjunction with the Oliver-Campbell filter (see Sec. 145) as routine procedure.

An installation of strainerless juice pumps, of the reciprocating type, is illustrated in Fig. 21. Compound saturation is taken care of by having one end of a pump handle third mill juice, and the other end fourth mill juice. A Peck strainer is placed just above the mills to screen all the juice going to process, this location being chosen to permit the easy return of the strained material to the bagasse on the mills (in the present instance, to the shredder). The juice for saturation purposes is spread on the bagasse by means of a patented distributor. Centrifugal pumps are now in more general use for this purpose than reciprocating pumps, but the arrangement in the diagram would be much the same.

60. Mill Sanitation—Sugar Losses Around Mills. The certainty of sugar losses caused by old-style juice strainers has been mentioned, but it is equally certain that losses of large proportions occur in and around the mills, particularly if proper attention is not paid to cleanliness and sanitation. These losses are undetermined, in fact unrecorded, because the control of the sugar entering the factory begins at the weighing tanks after the juice has left the mills.

Bacteriologists have shown that a handful of sour bagasse can infect the whole stream of warm juice flowing over it. The more modern mill designs now avoid all projecting bolts and dead corners in juice troughs. So-called self-cleaning troughs are now used which have such a slope that the bagasse does not hold up but flows out with the juice stream. Bagasse conveyors, carriers, and all moving parts about the mills are made more accessible for cleaning and in general, recognition is given to the fact that warm cane juice is an ideal medium for the growth of microorganisms and that such growth must be avoided by moving the juice as rapidly as possible to the clarification station where it will be sterilized by heat.

W. L. McCleery[42] showed that in several Hawaiian mills increases in acidity from crusher juice to last mill juice (expressed as acidity per 100° Bx) were much more marked at the end of the week than immediately after cleaning. The natural increase in acidity in clean mills was about 100 per cent and after a week's time without cleaning the increase was frequently 500 per cent. He recommended the use of a high-pressure hot-water hose with a small nozzle to dislodge accumulations in and around the juice strainers, elevators, etc., as this practice was found to reduce the acidity increase in those mills which adopted it. McCleery estimated the loss of sugar around the mills at 1 per cent of the sucrose in the juice, but admitted that it was difficult to determine. The use of antiseptics around those parts of the mill where souring may occur has been recommended by others but is not in general use.

[42] *Facts About Sugar,* May 30, 1925.

The Java Experiment Station found about twenty different microorganisms in the interior of apparently sound cane. First mill juice showed 1,500,000 organisms per cubic centimeter; fourth mill juice 5,000,000 before washing down, 1,875,000 after washing down. They found that the pH is no indication of the extent of bacterial activity in the juice but titrated acidity affords some guide to such activity.[43]

Sarkar[44] recommends the use of electrolytic chlorine (called E.C.) generated on the spot by a small convenient plant which is on the market. He suggests a strength of 1 : 25,000 for washing down mills, gutters, mill beds, etc., before starting up and each time that operations are interrupted for more than four hours.

King[45] says that numerous tests in the Philippines convince him that neither pH nor acidity per 100° Brix can be relied on as indicators of acidity increase during the milling process. He recommends the use of hot water for cleaning at three-hour intervals and advises against the use of lime or disinfectants.

It is obvious from what has been said previously in this chapter on the action of lime on cane fiber that the excessive use of lime in and around the mills is of doubtful expediency. Not only is there likely to be a great increase of color and reversible colloids by the action of the lime on the bagasse but reducing sugars may also be destroyed, giving further color and acid formation.

61. Processes Involving Diffusion; Naudet System, Egypt. The diffusion process as applied to cane direct is not practiced, but bagasse diffusion is in use in several factories in Egypt and offers some interesting features. The system consists of two milling units which prepare the bagasse for diffusion,[46] a diffusion battery similar in all respects to those used in beet factories, and two more mill units to reduce the water in the bagasse so that it may be burned. The Naudet part of the system consists of adding the limed juice expressed by the mills to the cell just filled with fresh bagasse from these mills and circulating the juice at 105° C. under pressure through this bagasse. The bagasse acts as a filter, and after the juice has been forced through it several times, the juice is bright and clear and goes to the evaporators. This cell then becomes part of the diffusion battery, a new cell becomes the Naudet cell, and the process continues. The diffusion juice also goes to the evaporators without further treatment so that all settlers and filter presses are eliminated. A capacity of 3000 tons per day and extraction of 98 per cent (which includes all losses up to the evaporator) with higher purity juice than is obtainable by milling are reported. Henri Naus Bey[47] describes the same process but says that the bagasse from the diffusers is dried to 52 per cent moisture by stack

[43] *Intern. Sugar J.*, May, 1930, p. 252.
[44] *Intern. Sugar J.*, March, 1929, p. 165.
[45] *Sugar News*, 1930, p. 640.
[46] Maxwell, *Intern. Sugar J.*, April, 1929, p. 186.
[47] *Intern. Sugar J.*, November, 1936, p. 431 (abs.).

gases. He also says that 80 per cent of the juice reaches the evaporators in thirty minutes after the cane starts through the mill.

62. Vascane Process. A process invented by Anton Vazquez of Cuba, but which has never come into commercial use, involves the simultaneous manufacture of fiber board and sugar from the cane. The cane is disintegrated by shredding it against an abrasive wheel consisting of a silica carbide cylinder revolving at 1200 rpm. The disintegrated fiber goes to a continuous diffusion process which extracts 99 per cent of the sucrose with 30 per cent dilution. No defecation station is necessary, lime being added to that section of the juice (to 6.8–7.0 pH) which is returned to the abrading cylinder for lubrication. The precipitate is absorbed by the fiber, and the juice is clear and brilliant.[48] The exhausted pulp from the diffusers is treated with 2 per cent resin which is precipitated by small amounts of copper sulfate and aluminum sulfate. The precipitated copper-aluminum resinate is a preventive of fungous growth. The board is then formed on a traveling screen to a depth of 1 in. after which it is compressed to ½-in. or ¼-in. thickness, depending on the use.

63. By-Product of Milling. The by-product of milling cane is bagasse or megasse (English colonies), the woody fiber of the cane with the residual juice and moisture derived from the saturation water. This material supplies a very large part and in many instances all the fuel required by the factory.

The fuel value of bagasse is considered in the next chapter. In many modern factories there is an excess of bagasse because of electrification and fuel economies. In some factories the handling of this excess has become a problem, and baling machinery has been installed to facilitate handling it for such purposes as locomotive fuel and idle season usage in boilers. In the Philippines bales which are stored for six months lose about half their moisture (24 per cent) and the baled bagasse is then suitable for locomotives. It is also used by laborers for domestic fuel. Even so there is still an excess which is burned in incinerators, and the ashes are used for fertilizer.[49]

UTILIZATION OF BAGASSE

64. Paper Manufacture. Many attempts have been made to utilize this material in paper manufacture, but in general they have not been commercially successful. For several years in Hawaii bagasse has been made into a low-grade paper which is used to mulch the ground between the cane plants to keep down the weeds. It is used more extensively in the pineapple plantations for a similar purpose.

Havik[50] was commissioned by the Government of the Dutch East Indies to investigate the fibers of Java and the other islands with a view to paper manufacture. He obtained 32 per cent of the weight of dry bagasse in dry

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[48] Seidel, *Ind. Eng. Chem.*, July, 1930, p. 765.

[49] Pitcairn, *Proc. 6th Congr. Intern. Soc. Sugar Cane Tech.* (Baton Rouge, 1938), p. 925.

[50] *Intern. Sugar J.*, **14**, 52.

unbleached paper. The bagasse was from Cheribon cane. The frequently stated yield of 52 per cent of paper is disproved by Havik's experiments.

The most recent report on the subject[51] says:

> No one of the four reported commercial attempts in the production of paper from bagasse has gained success. Long fibers are necessary for good felting. Although bagasse cellulose contains some long fibers, the greater part is composed of short fibers.

65. Wall Board and Insulating Board. The successful use of bagasse fiber in the manufacture of a building and insulating board sold under the trade name of Celotex has superseded its use as a fuel in many factories in Louisiana. The bagasse is baled as it comes from the mill, and the bales are stored in the open in large piles covered by roofing paper. When taken to the manufacturing plant the bales are broken, and the bagasse is fed through a shredder to a cooker which removes the resins, waxes, and pectocellulose and at the same time renders the fiber tough and flexible. From the cooker and washers it goes through paper mill refiners to separate the bundles of fibers. Waterproofing material is added at this point as well as special chemicals to make the board termite-proof. The mixture of fiber and water is fed to a machine which forms the board by the process known in pulp handling as felting. No adhesive is used, the strength of the board being due solely to the interweaving and entangling of the fiber. As the wet board comes from the machine it is fed into a continuous hot-air drier 800 ft. long from which it emerges dry in a continuous sheet 12 ft. wide to be cut by saws into convenient sizes.

The material is made in several forms, the thickness depending on whether it is to be used for building or insulating purposes. A special form drilled with 441 holes per square inch is used for sound-deadening and sound-proofing. The pith washed out of the fiber is washed and dried and sold to the explosives industries. The output of the factory in Louisiana increased from 19,000,000 sq. ft. of board in 1922 to 460,000,000 sq. ft. in 1930.[52] The process has also been working on a commercial basis in England.

A similar process operates in Hawaii[53] where the main differences are found in the separate treatment of coarse and fine fiber and in the type of boarding machine and drier used. A plant for making bagasse wall board is also in operation in Australia. The Vascane process for making board and insulating material has already been described. A method by E. A. Gastrock and D. F. J. Lynch for calculating equivalent fuel values and costs of bagasse in relation to other fuels, together with a chart for evaluating bagasse for industrial utilization, has been published.[54]

66. Alpha-Cellulose. There have been many attempts to utilize the finer portions of bagasse for the production of alpha-cellulose, which in turn is used

[51] Lynch, *Hawaiian Sugar Tech.*, 1934, p. 159.

[52] E. C. Lathrop, *Ind. Eng. Chem.*, May, 1930, p. 499.

[53] *Facts About Sugar*, 1932, No. 7, p. 274.

[54] *Facts About Sugar*, June, 1931, p. 37.

in the manufacture of rayon, high explosives, etc. One of the successful attempts on a commercial scale is described by De la Rosa.[55] A white alpha-cellulose of 97 per cent purity was produced by means of an alkaline cooking process at Central Tuinucu, Cuba (details not given). Later articles[56] state that this is a process in which sulfurous acid (6 per cent SO_2 at 110° C. for two hours) is used for the first digestion, then 10 per cent caustic soda at 140° C. for two hours. Bleaching with hypochlorite is in two stages. These two later articles indicate that the process has not been continued on a commercial scale.

Laboratory experiments in Hawaii gave a high-grade cellulose pulp by treating all the bagasse that passes a ½-in. mesh as follows.[57] Digest for one hour at 90°–100° C. with 2 per cent nitric acid; drain, wash, and recover acid; boil forty-five minutes with 2 per cent caustic soda; drain, wash, and recover the caustic soda; bleach; digest for thirty minutes in 5 per cent caustic soda. The results were: pulp recovery 32–33 per cent; alpha-cellulose 95–96 per cent; soda soluble 6–8 per cent; ash 0.3 per cent.

Further studies of Hawaiian canes showed that wide differences exist between varieties of canes in the alpha-cellulose content.

Variations in crude cellulose per cent bagasse were from 50 to 55 per cent but the alpha-cellulose (the valuable portion) per cent bagasse ranged from 27 to 37 per cent.[58]

A detailed study of pulping bagasse and other fibrous materials with nitric acid together with a complete bibliography of all investigations and patents on the subject has been made by D. F. J. Lynch and others.[59] They conclude that "as evidenced by the data on bagasse, the cost of production of pulp from fibrous plant materials by the nitric acid process is not prohibitive."

67. Pith. The separation of the pith by screening or in some other way has been practiced. Lathrop[60] says that in the manufacture of Celotex a certain amount of the pith is washed out of the fiber. This is collected, washed, and put through a rotary drier, yielding several thousand tons of an extremely light, porous product for the explosives industry.

The pith is also separated by fine screens in some Cuban factories. A further separation of the pith is made in one plant in Louisiana by means of gravity separators such as are used for classification of boneblack (see p. 320).

68. Agricultural Mulch and Cattle Bedding. At least two factories in Louisiana have elaborate plants on a commercial scale for the separation of the bagasse into various-sized fractions for different uses. The bagasse from the cane mill goes to gas-fired Heil driers where it is heated to 1400° F., losing about 40 per cent of moisture during the drying process. The dehydrated

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[55] *4th Annual Proc. Assoc. Sugar Tech. Cuba* (1930), p. 152.
[56] De la Rosa, *Intern. Sugar J.*, December, 1938, p. 473; September, 1939, p. 364.
[57] Payne, *Ind. Eng. Chem.*, December, 1934, p. 1339.
[58] Williams, *Repts. Assoc. Hawaiian Sugar Tech.*, 1934, p. 162.
[59] *The Paper Industry and Paper World*, April, May, and June, 1939.
[60] *Loc. cit.*

bagasse then goes to a set of Rotex screens which divide it into three classifications. The coarest fibers are used as a horticultural mulch around flowers and plants to retain the moisture and keep down weeds, and this mulch has been reported to be superior to a mulch of peat moss. The second portion, also fibrous, is used for chicken litter and cattle bedding. The third or finest fraction is the pith. This is further separated by the gravity separator as described above; the lightest part, a fairly high-test alpha-cellulose, is used for explosives, and the remainder is used as fiber in a cattle feed mixed with molasses.

69. Distillation. The dry distillation of bagasse in experiments in Brazil did not yield favorable results.[61] Laboratory tests showed: coke 34.5 per cent; condensable gases 8.5 per cent; non-condensable gases 31.5 per cent; water 25.4 per cent. The condensable gas fraction yielded: acetic and homologous acids, 10.55 per cent; methyl alcohol 0.62 per cent; oily substances 14.22 per cent; soft pitch 63.25 per cent; water and losses 11.36 per cent. Actual runs on a semi-commercial scale failed to give these yields.

More recent experimentation by Orthmer and Fernstrom[62] resulted in a more favorable report on the destructive distillation of bagasse. Yields per short ton of dry bagasse were 1050 lb. of charcoal, 1.2 gal. of methanol, and 53 lb. of acetic acid, besides tar and combustible gases sufficient to carry out the carbonization. The charcoal was formed into briquets with molasses. A continuous retort such as is used for wood-waste distillation is recommended.

70. Plastics from Bagasse. The manufacture of plastics from lignin in bagasse has been the subject of many studies and much publicity in recent years. The lignin is the constituent of bagasse (and other vegetable products) which acts as a binder for the cellulose structure. The methods, as worked out in government laboratories, consist essentially in hydrolyzing or digesting the bagasse at high temperatures and pressures with dilute acid or aniline. It is then washed, dried, and ground, after which the ground powder is mixed with a suitable plasticizer such as furfural or furfural and aniline.[63] The furfural can be produced from bagasse pith by acid digestion at high pressures in the presence of aluminum chloride. A pilot plant at Valentine Sugars, Louisiana, is reported as operating successfully, but commercial scale operations are not yet under way. The same processes which would be used for bagasse plastics are applicable to many forms of waste products containing lignin, such as cornstalks, straw, sawdust, and waste liquors from papermaking processes.[64] The plastics are dark colored and do not mold as readily as some other forms of plastics, and they are best suited for shaping into flat sheets or tiles. The development is still on a relatively small scale.

[61] Freise, *Intern. Sugar J.*, May, 1931, p. 225.

[62] *Ind. Eng. Chem.*, March, 1943, p. 312.

[63] Willcox, *Sugar*, September, 1941, p. 20; Armovsky and Clark, *Intern. Sugar J.*, December, 1941, p. 371.

[64] Plunguian, *Ind. Eng. Chem.*, October, 1940, p. 1399.

CHAPTER 4

STEAM PLANT AND FUEL

71. Bagasse Feed to Boilers. The bagasse is carried directly from the mills to the boilers by carriers of the drag type and is fed to the boilers mechanically except in Java where hand feeding is practiced to some extent. The simplest mechanical device consists of a hopper fitted with a counterbalanced trap door which opens to permit the bagasse to fall into the furnace when a sufficient amount has accumulated in the hopper and closes again to prevent the entrance of air. Rotary feeders are mechanically driven drums which seal the opening at all times while revolving and delivering the bagasse to the furnaces. These mechanical feeders supply the bagasse to the boilers at a constant rate, adjustments being made by hand to take care of changes in fuel requirements. Automatic feeding devices in use in a number of mills in Australia and in Java regulate the quantity of bagasse fed to the furnace, depending on the amount of bagasse burned. According to Jenkins[1] these devices (generally used with step grate furnaces) save labor and improve boiler efficiency.

STEAM BOILERS

72. Boiler Capacity. The boiler capacity required in a cane sugar factory varies with the manufacturing equipment, the quality of the cane, the quantity of saturation water, and the grade of sugar produced. More boiler capacity is necessary for poor than for rich cane, and for the manufacture of plantation white sugar than for raw sugar. If the factory equipment includes multiple application of the vapors generated in evaporating the juice, in juice heating and for other purposes, as is customary in modern plants, its boiler requirements are lessened. It is customary to state the boiler capacity required by a factory in nominal or rated boiler horsepower per cane capacity-ton, i.e., the total rated boiler horsepower divided by the capacity in tons for twenty-four hours. (Nominal or rated boiler horsepower is the heating surface of the boiler divided by 10.) A large Cuban factory having good equipment requires from 1.2 to 1.5 nominal boiler horsepower per ton of cane in twenty-four hours or about 12 to 15 sq. ft. of boiler heating surface. Tromp[2] gives the average for raw sugar factories as 15 sq. ft. of heating surface, but he says that he knows of factories where the area is only 11 sq. ft. This average figure of 15 sq. ft. of heating surface per ton per day is undoubtedly high for

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[1] *Intern. Sugar J.*, July, 1938, p. 269.

[2] *Machinery and Equipment of the Cane Sugar Factory*, London, 1936, p. 294.

modern boiler plants. Lopez-Ferrer[3] gives the average for 60 Cuban factories as 13.8 sq. ft. with a range from 11.2 to 16 sq. ft. A modern installation in Queensland, Australia, has two boilers of 8650 heating surface for a mill grinding 1900 tons of cane per day, or approximately 9 sq. ft. per ton-capacity. The moisture in bagasse is reported as 45 to 47 per cent, and the boilers are equipped with automatic bagasse feeders, forced and induced drafts, and large combustion chambers.[4] At Clewiston, Florida, an elaborate arrangement for steam economy whereby vapors from effects are used for boiling massecuites permits the grinding of 4000 tons of cane per day with 2780 bhp. or about 0.7 bhp. (7 sq. ft. of heating surface) per ton of cane per day.[5]

As stated above the manufacture of white sugar with its attendant increase in steam consumption for evaporating wash waters, etc., may increase the steam requirements by 15 per cent or more. Carbonation factories require from 25 to 30 per cent more boiling capacity than a simple defecation raw sugar house.

Both fire and water-tube boilers of many different designs are used in cane sugar factories. The fire-tube boiler was formerly considered to have an advantage because of its large water capacity which makes it suitable to meet the irregular demands of the factory for steam. A recent tendency, however, has been toward water-tube boilers of from 600 to 1200 hp. The straight tube types seem to be more popular. Modern boiler control instruments, large units, superheaters, economizers, and improved furnace designs have greatly increased boiler efficiencies.[6]

73. Fuel. The usual fuel of the cane sugar factory is bagasse, the residue from milling of the cane itself. In Louisiana those factories which sell bagasse for the manufacture of insulation board (Sec. 65) use oil or natural gas either entirely or in part.

Bagasse contains a large percentage of moisture which varies with the grinding speed and the efficiency of the milling. In Cuba and Puerto Rico the average is 48 to 50 per cent with figures as low as 45 per cent in the more modern mills. In Hawaii the moisture content is frequently 40 per cent, or even lower, because of the much slower grinding rates and consequent higher efficiency of the milling. Similar figures are reported for the Philippines. Many small mills of the older type in Louisiana turn out bagasse with 55 per cent moisture, but the more modern installations average 50 per cent or below. Australia reports figures from 45 to 47 per cent in the more modern factories.

Where cane is of moderate cost and labor and fuel are expensive, the factory may bring in larger returns by grinding a large quantity of cane less efficiently. Large grinding rates produce more fuel in proportion to radiating surfaces. The improved boiler conditions, greater fuel economies in the fac-

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[3] *13th Annual Proc. Assoc. Cane Sugar Tech. Cuba* (1940), p. 235.

[4] *Proc. 6th Congr. Intern. Soc. Sugar Cane Tech.* (Baton Rouge, 1938), p. 256.

[5] *Intern. Sugar J.*, October, 1933, p. 388.

[6] See Hedley, *Intern. Sugar J.*, July, 1937, p. 264, on South African practices.

tory, electrification of machinery and higher fiber content of variety canes, together with more efficient milling, have all combined to give the modern plant an excess of bagasse which has become a problem to take care of. Formerly the majority of factories had to resort to the use of extra fuel which constituted an appreciable addition to the cost of manufacture, but these conditions now apply only to the smaller and less efficient plants.

74. Drying Bagasse. The partial drying of the bagasse by the waste heat in chimney gases to increase the net fuel value has been advocated by many and is actually practiced in a few small plants in some European colonies. It offers many mechanical difficulties, together with considerable danger of fire in the driers. A successful drier is described as in use in Jamaica in which the bagasse conveyor is converted into a continuous drier, with flue gases utilized as the drying agent. The bagasse is drawn over a perforated deckplate by a drag conveyor, the hot flue gases under pressure being introduced at the bottom of the housing enclosing the conveyor. The gases pass through the perforations and thence to the atmosphere and the partially dried bagasse goes to the furnaces or storage.[7]

75. Preheating Air. It can be shown that preheating the air admitted to the furnace for combustion purposes will effect as great a saving as drying the bagasse and requires much simpler equipment. The simplest of the air preheaters are those of the tubular type in which the flue gases pass through tubes and the air to be heated around the tubes. (A preheater of this type is part of the boiler installation shown in Fig. 25.) The Ljungstrom rotary preheater has a rotor which turns slowly and whose elements pass alternately from the flue gases to the air and back again. This type is gaining in popularity throughout the world because of its compactness and ease of keeping clean. Savings of 15 and 20 per cent in fuel are reported, but the most recent figures[8] by the Australian Bureau of Experiment Status show fuel savings of 6.8 and 8.2 per cent respectively on two boilers tested. Most modern installations include preheaters in some form.

76. Steam Accumulators. To take care of the fluctuating steam consumption in both factories and refineries, steam accumulators have come into fairly general use throughout the world. The accumulator is, in effect, a large tank of water into which steam is automatically introduced when there is an excess available from the boilers and from which steam is automatically drawn when there is an extra demand for the pan-boiling house. Thus peak loads are taken care of and the boilers operate at a more even rate. The Ruths accumulator is probably the commonest form, and large fuel savings and other advantages have been reported from several installations.[9]

77. Bagasse Furnaces. The first furnace for the burning of green bagasse for generating steam was patented in 1886 by Samuel Fiske, the inventor of the cane shredder. It was first used in Louisiana and then in Cuba at

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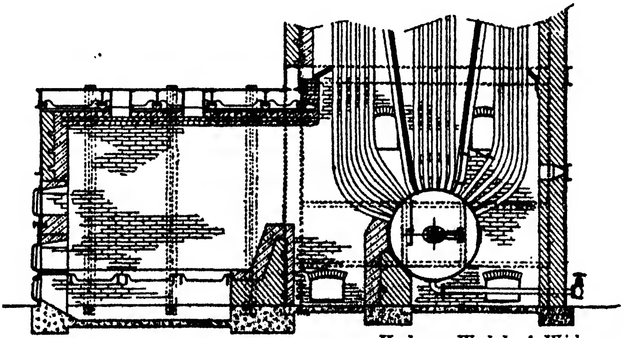
[7] Holgate, *Intern. Sugar J.*, January, 1930, p. 32.

[8] Clayton, *Intern. Sugar J.*, October, 1939, p. 386.

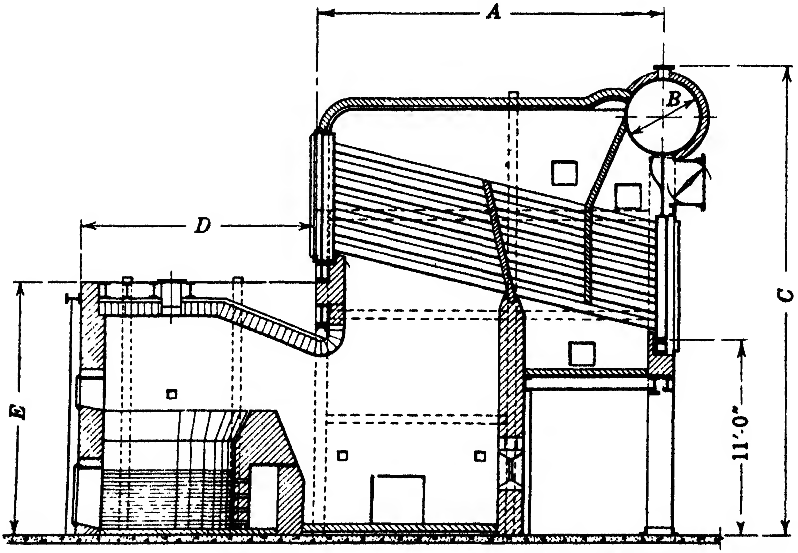
[9] Wilson, *Intern. Sugar J.*, August, 1938, p. 322.

"Soledad," Cienfuegos. This furnace consists of an oven fitted with horizontal grate bars upon which the bagasse is burned (Fig. 22). A single furnace was often connected by means of flues with several boilers, though preferably in



Hedges, Walsh & Widener.

Fig. 22. Flat Grate Bagasse Furnace.

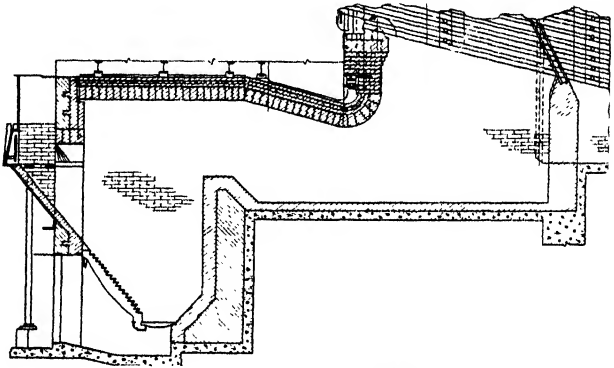


Combustion Engineering Co., Inc.

Fig. 23. Hearth Bagasse Furnace.

entirely new installations with but two. In the latter case the furnace was under the front end of the boilers. Forced draft was used in the early installations, since the bagasse often left the mills with as high as 60 per cent moisture. Almost simultaneously with Fiske, Frederick Cook introduced his green bagasse burner into Cuba at "Hormiguero," also after using it in Louisiana. In this type of furnace the bagasse is burned on a hearth (Fig. 23) in an oven placed between two water-tube boilers. Air is forced into the burning bagasse through tuyères. Prior to the introduction of the Fiske and Cook burners in Cuba, all bagasse was sun dried before burning it. The step grate furnace is the third and most recent type (see Fig. 24). In this type there is usually a furnace in front of each boiler. The grates are inclined and resemble a stepladder, and in fact they are often termed "stepladder" grates. These grates are very long and narrow. The bagasse falls upon the top steps of the ladder and gradually works its way to the small flat grate at the bottom.

Modern installations of all three types of furnaces are very efficient. The flat grate and hearth types are generally used in Cuba and Puerto Rico, but the step grates are favored in those countries where the moisture content of the bagasse is lower (Hawaiian Islands, the Philippines, and Java). Forced draft, which as stated before was used in the earlier installations, was later abandoned until its use was almost entirely discontinued. In the more recent furnace installations, however, forced draft has been generally readopted and with its use ratings as high as 200 per cent of the rated boiler horsepower have been obtained and constant operation at 175 per cent rating is not at all unusual.

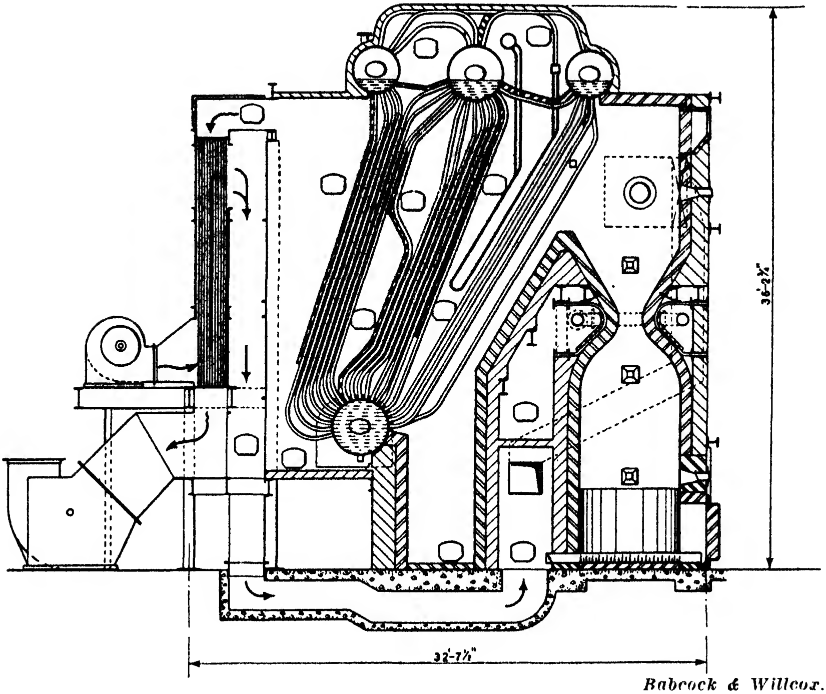


Hedges, Walsh & Widener.

Fig. 24. Step Grate Bagasse Furnace.

The problem of furnace design for bagasse has been given much study in the past two decades, and the essential factor in burning such a wet fuel is recognized to be the maintenance of high furnace temperatures. In burning such dry fuels as coal or oil the boilers are placed immediately above the fires to absorb as much heat as possible and reduce furnace temperatures in order to protect the brick settings, whereas with bagasse the opposite is true. Combustion must be completed in a separate compartment before the gases strike the cool surface of the boiler, otherwise temperature may be lowered to a point where losses will occur because of carbon monoxide or unconsumed carbon.

The tendency is toward much larger furnace volumes. In Hawaii where 1 cu. ft. per rated bhp. was formerly the practice, furnaces now have 2½ cu. ft. or more. These larger combustion volumes with modern furnace designs frequently show temperatures of 2300° F. where 1700° F. was formerly considered the best attainable. CO_2 readings up to 16 per cent are reported.[10] This enlarged combustion chamber in relation to boiler size with attendant higher furnace temperatures seems to be general throughout the sugar world.



Babcock & Wilcox.

Fig. 25. Ward Single-Pass Bagasse Furnace.

The most recent furnaces have suspended flat tops (so-called flat arches) which, besides having many advantages of construction, distribute the hot gases to the boiler more evenly than the circular brick arch formerly used. (See Figs. 22, 23, 24.) Another important detail in most recent designs is the mixing wall, above the bridge wall (Figs. 23, 24) which directs the gases at the top of the furnace downward to promote mixing and prevents stratification of the gases, thereby aiding in securing complete combustion. The use of forced draft also aids in mixing the gases and avoiding stratification. The downward slope of the roof reflects the heat back on the fuel bed, promoting the drying of the incoming bagasse.

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[10] Stone. *Rept. Assoc. Hawaiian Sugar Tech.*, 1930.

An improvement in furnace design which has received very favorable comment is the Ward single-pass furnace which is shown in Fig. 25 in connection with a Sterling type boiler. It consists of a cast-iron hearth with a bottle-shaped furnace, from which the name has been derived. Air is admitted at the lower rim of the hearth, and secondary air is induced from tuyères at the bottleneck. Because of the contraction and expansion of the combustion gases a torch effect is obtained. There is no combustion chamber needed and multiple hearths are used, which need to be cleaned only once a day, according to local conditions.[11]

78. Fuel Value of Bagasse. The fuel value of dry bagasse shows great uniformity throughout the world. Numerous calorimeter combustion tests in Cuba, Louisiana, Hawaii, Natal, and other sugar countries show that bagasse entirely free of moisture contains between 8300 and 8400 heat units (Btu.). Dr. R. S. Norris[12] found a value of 8100 in Hawaii, but later investigators there corroborate the figures given above (8350).

Recent investigations on the fuel value of the dry bagasse of newer varieties also agree closely with this figure. E. P. Hedley[13] in South Africa analyzed samples of POJ 2725 and 2878, Co 290 and 281, and Uba canes and found an average Btu. of ash-free dry bagasse of 8394, the extremes being 8294 and 8487. Further work by this same writer[14] gave average figures of 8310 Btu. per lb. of ash-free dry bagasse. Hawaiian figures for H 109 and POJ 2878 showed 8310 and 8330 Btu. respectively.[15] The average figure of 8350 Btu. per lb. of dry ash-free bagasse (4640 calories per kg.) is sufficiently accurate for all practical purposes and agrees within about 1 per cent with determinations made throughout the world. In considering the actual fuel value of bagasse as it is burned upon the grates, it must be taken into account that a certain number of heat units are absorbed in evaporating its moisture and heating the resultant vapor to the temperature of the chimney. Further, a part of the fuel is consumed in heating the excess air which is drawn through the grates, as well as in heating the products of combustion, this heat loss being dependent upon the temperature of the flue gases passing up the stack.

The table on the next page shows the actual number of Btu. per pound of bagasse, with varying moisture content and different percentages of excess air.

Heat value per pound of dry bagasse, 8350 Btu.

Bagasse assumed to have the following composition:

$$C = 46.5; \quad H_2 = 6.5; \quad O_2 = 46.0$$ [16]

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[11] *11th Annual Proc. Assoc. Cane Sugar Tech. Cuba* (1937).

[12] *Hawaiian Sugar Planters' Assoc., Bull. 40.*

[13] *Intern. Sugar J.*, September, 1936, p. 349.

[14] *Intern. Sugar J.*, July, 1937, p. 266.

[15] *Intern. Sugar J.*, March, 1934, p. 126.

[16] Other ultimate analyses of the pure fiber of bagasse show Kelly: $C = 49.48$; $H_2 = 6.20$; $O_2 = 44.32$. Average Geerligs and others: $C = 48.45$; $H_2 = 6.20$; $O_2 = 45.53$. (*Intern. Sugar J.*, February, 1939, p. 69 (abs.).)

Heat Value per Pound of Bagasse Burned

(Stack Temperature, 500° F.)

Per Cent Moisture in Bagasse	Theoretical Amount of Air	Per Cent Excess Air			
		50	100	150	200
40	3747	3572	3400	3225	3054
41	3664	3494	3321	3151	2985
42	3579	3412	3244	3377	2902
43	3494	3329	3167	3004	2840
44	3400	3249	3089	2928	2769
45	3326	3169	3012	2854	2697
46	3241	3087	2933	2778	2624
47	3160	3007	2856	2704	2553
48	3077	2928	2779	2631	2482
49	2993	2851	2700	2557	2409
50	2910	2767	2624	2481	2338
51	2829	2689	2548	2416	2275
52	2744	2607	2469	2332	2194
53	2664	2528	2393	2259	2124
54	2582	2450	2319	2188	2057

These figures represent the actual number of Btu.'s available at the burners when conditions as regard excess air, stack temperature, and moisture are as indicated.

The amount of excess air actually being used may be arrived at by determining the CO_2 in the flue gases (Chapter 40) and using the curve given in Fig. 206 showing the relationship between CO_2 by volume and percentage of excess air. One hundred per cent excess air was formerly taken as good average practice, but with modern installations this has been reduced, so that 50 per cent is closer to the average for recently built boiler plants.

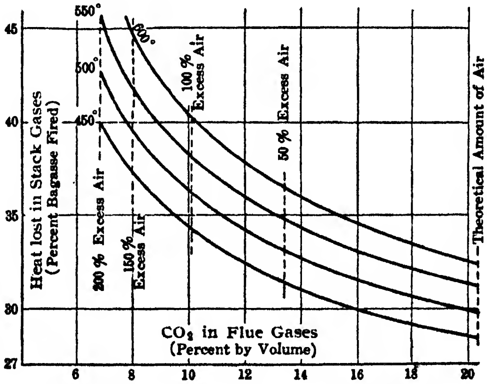

Fig. 26. Heat Lost in Stack Gases.

The effect of variations in stack temperatures on fuel losses is shown in the chart (Fig. 26) for a bagasse of 49 per cent. It will be noted that approximately 6 per cent more bagasse will be burned with a stack temperature of 600° F. than with one of 450°, using 100 per cent excess air, all other conditions being equal.

For comparative purposes, 1 ton of bagasse under average Cuban conditions (49 per cent moisture, 100 per cent excess air) closely approximates the fuel value of one barrel (335 lb.) of crude oil and of ¼ ton of coal, using the same firing conditions.

79. Molasses as Fuel. The gross calorific value of molasses is given by Behne[17] as 5380 Btu. per lb., and he figures the available heat under average Australian boiler conditions (50 per cent excess air) at 3419 Btu., with an evaporation from and at 212° F. of 3.53 lb. water.

Geerligs[18] gives the calorific value of molasses with 9.28 per cent ash and 19.4 per cent moisture as 5515 Btu. Naturally the composition of molasses, particularly the moisture content, would affect its fuel value.

According to Kress[19] molasses as fired in Australia has a fuel value one-fifth that of coal.

These figures show that molasses has a fuel value about equal to that of bagasse of 42 per cent moisture or about 26 per cent better than bagasse at 49 per cent moisture. To be economically used as a fuel, molasses must therefore be worth less than 1 cent per gal. or other fuels must be exceptionally high priced. In Java and Australia molasses is burned to a considerable extent and 25 per cent, or 4,000,000 gal. were reported as burned in the latter country in 1929.[20] In Java the heated molasses is sprayed over the bagasse as it leaves the last mill, or it is sometimes burned alone in special furnaces. When burned with bagasse the ash tends to fuse in the furnaces. According to Van Harreveld[21] the molasses must be thoroughly absorbed by the bagasse to avoid excessive trouble with slag. The recovery of potash salts in molasses by ashing it in a separate furnace is reported on recently by Bogstra and Klokkers.[22]

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[17] *Proc. 1st Congr. Queensland Soc. Sugar Cane Tech.*, 1930.

[18] *Cane Sugar Manufacture,* second edition, 1924, p. 328.

[19] *Intern. Sugar J.*, February, 1932, p. 62.

[20] Kress, *loc. cit.*

[21] *Proc. 2nd Congr. Int. Soc. Sugar Cane Tech.* (Havana, 1927).

[22] *Intern. Sugar J.*, April, 1942, p. 108 (abs.).

CHAPTER 5

OUTLINE OF METHOD OF MANUFACTURING RAW CANE SUGAR

80. Introductory. The purpose of this outline is to minimize repetitions in future descriptions, to define technical terms, and to provide a condensed description of raw sugar manufacture for those who may not care to study the detailed processes given in the separate chapters devoted to the various stations in the raw sugar factory. To aid in this feature, a flow diagram is given (Fig. 27) showing the complete process of raw sugar manufacture as carried out in modern plants. For purposes of simplicity only general practice is included without indicating refinements which may be in use in many plants, and no attempt has been made to indicate the correct number of units of each piece of apparatus.

81. Extraction of the Juice. This has already been described in detail in Chapter 3. In the process universally used the cane is crushed by passing it between heavy rollers which squeeze out the juice. The cane is prepared for the grinding in the mills by revolving knives, crusher rolls or shredders, or a combination of all three. Water and thin juices are sprayed on the crushed cane as it leaves the later mills in the series or tandem. This is called imbibition (or saturation or maceration) water and aids in the extraction of the juice. More than 90 per cent of the sugar in the cane goes into the juice, the remaining portion being held by the bagasse (or crushed cane waste) which generally goes to the boilers as fuel (see Chapter 4). The flow diagram shows a fifteen-roller mill with revolving cane knives and a crusher.

82. Purification of the Juice: Clarification. The defecation or clarification of the juice as it comes from the mills is accomplished by means of lime and heat. The acidity of the juice is neutralized by milk of lime and its temperature raised to the boiling point. The precipitates formed by the action of heat and lime are allowed to settle in either open tanks or closed continuous clarifiers and the clear liquid or "clarified juice" is decanted from the "muds," "scums," or "slops" and goes to the evaporator station. The muds are filtered in filter presses or vacuum filters and the filtrate or "press juice" is mixed with the clarified juice or returned to the raw juice for reclarification. The precipitates retained by the filter press (or vacuum filter) are called filter press cake or press cake (cachaza in Cuba) and are discarded or used as fertilizer. The flow diagram shows single clarification in a tray-type continuous clarifier with filter presses for the mud waters. There is a wide variety of

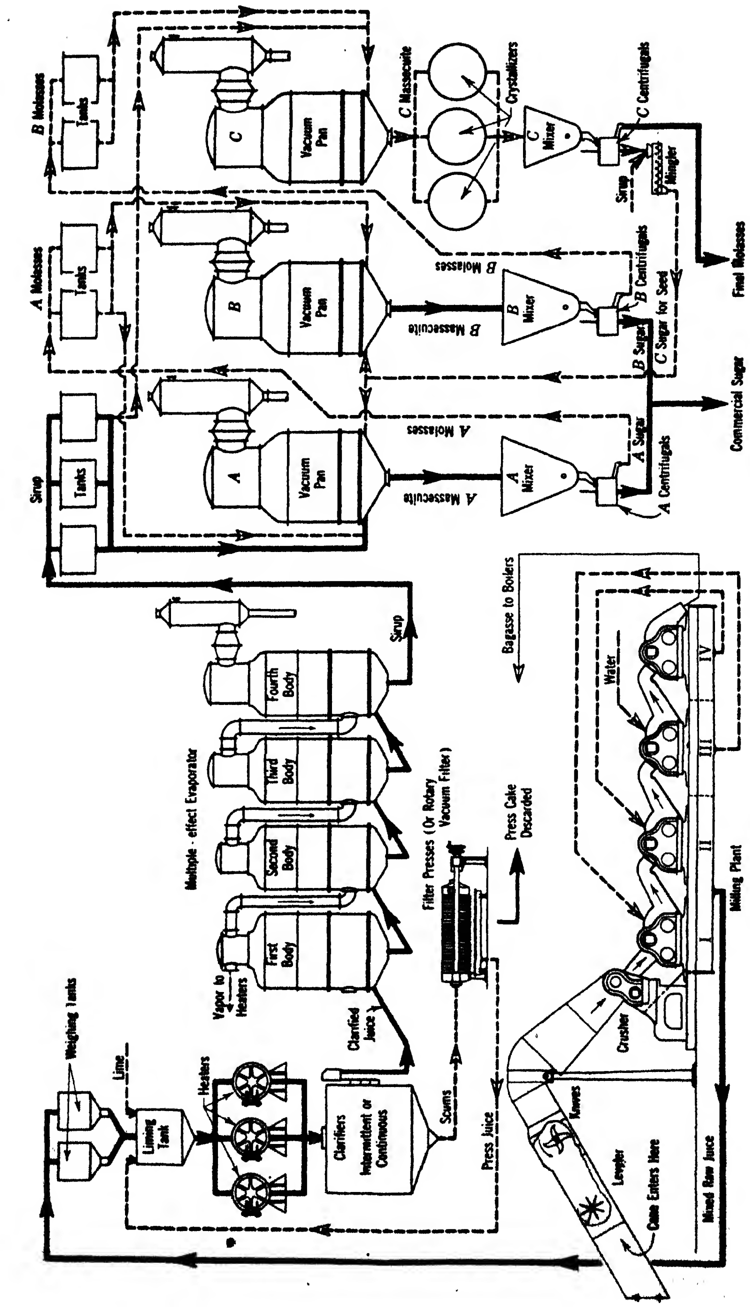

FIG. 27. Flow Diagram of Raw Sugar Factory.

systems and modifications in the clarification process, as described in the succeeding chapter.

83. Evaporation of the Juice. The clarified juice goes to the multiple effect evaporators without further treatment. The evaporators consist of a series of vacuum-boiling cells or "bodies" arranged in series so that each succeeding body has a higher vacuum than the one before it, and therefore boils at a lower temperature. The vapors from one body can thus boil the juice in the next one. By this system the steam introduced into the heating element of the first body does multiple duty or "multiple-effect evaporation." The evaporator in the flow diagram is a "quadruple effect," and 1 lb. of steam evaporates nearly 4 lb. of water. The sirup (meladura in Cuba) which leaves the last body of the evaporator continuously contains 30 to 40 per cent of water. (Chapter 9).

84. Crystallization. The crystallization is accomplished in single-effect vacuum pans under reduced pressure. The sirup is evaporated until saturated with sugar. At this point crystals or "grains" separate in the boiling mass or "seed grain" is added to serve as nuclei for the crystals. The pan is "charged" with sirup from time to time, as the water evaporates, and the sugar it contains is largely deposited upon the grain present without the formation of additional crystals.

The growth of the crystals is so regulated that they are of sufficient size when the pan has been filled, at which point the mixture of crystals and sirup is concentrated to a dense mass, "massecuite" (beet industry, *fill mass*) and the "strike" is then discharged from the pan.

The boiling of massecuites and the reboiling of the molasses (see next paragraph) are very carefully controlled and carried out by predetermined boiling systems (see Chapter 11). The flow diagram shows a three-boiling system which is common practice in tropical countries.

85. Centrifugaling; Purging; Reboiling Molasses. The massecuite is conveyed into a mixer and from this is drawn into revolving machines called centrifugals. These machines have cylindrical perforated metal "baskets," lined with wirecloth and perforated bronze sheets. The basket spins at high velocity on a suspended vertical spindle. The sugar crystals are retained by the lining and may be washed with water if desired. The mother liquor, "molasses," passes through the lining by reason of the centrifugal force exerted. The machine is stopped after the removal of the molasses, and the sugar is "cut down," leaving the centrifugal ready for another charge of massecuite.

The molasses is returned to the vacuum pan for reboiling with a portion of high-grade sirup to obtain a second crop of crystals and this "second massecuite" is centrifugaled as before, yielding a sugar which is about the same quality as that from the first massecuite but a "second molasses" of much lower purity. When successive reboilings have reduced the molasses to such a low purity that it will no longer yield any more sugar by crystallization, it is sold as "final molasses" or "blackstrap." The final low-grade massecuite is retained in crystallizers for several days before being purged in the centrifugals (Chapter 12). The modern factory produces only one grade of raw sugar and final molasses. The development of the production of high-grade sugar from all massecuites of whatever purity has greatly complicated the pan-boiling systems.

86. Historical Data. *Machinery and Apparatus.* Much of the apparatus used in cane sugar manufacture and refining was developed specifically for the industry and was later adapted to other chemical and food industries. The sugar industry was the earliest food process to employ chemists and anticipated by many years the modern ideas of technical and chemical control in present use in most large industries.

The milling of cane developed from upright wooden rollers, driven by animals, waterpower or windmills. Smeaton is credited with being the first to arrange three horizontal rollers in the triangular form now used, the first steam-driven mill of this type being used in Jamaica in 1794. Six-roller mills, i.e., two sets of three rollers each, were first used in Cuba in 1883, and the first nine-roller mill in the world was at Cora Plantation in Louisiana in 1898. Crushers with zigzag grooving were developed in 1883, and the shredder, invented by Fiske, was first used in Louisiana in 1887.

The vacuum pan was invented by Howard in England in 1813 and was in use in ten English refineries by 1827. The development of filtration of sugar solutions through columns of boneblack, credited to Derosne, took place in these refineries at about the same time. Rillieux of Louisiana propounded the principle of multiple-effect evaporation in 1834, and his first double-effect evaporator was erected at Packwood, Louisiana, in 1844. The centrifugal machine for purging sugar massecuites has been variously credited to Schöttler[1] in 1848 and to Dubrunfaut[2] in 1849, but it is generally agreed that the suspended centrifugal of the type now used was patented in 1852 and used in Hawaii by David Weston in 1867. For many years the overhead or suspended type was known as the Weston centrifugal. The first crystallizers were introduced by Bocquin and Lipinski about 1880. The idea of the filter press was advanced by Howard as early as 1820, but the first successful filter press for sugar work (constructed with wooden plates) is credited to Needham in 1853.

Processes. Priority in the use of processes is more difficult to determine than the invention of machinery. Lime as a defecant is mentioned in 1685. References to the value of carbonic acid and sulfurous acid are numerous in the early part of the last century (see Tables 51 and 51*a*) but the carbonation process for clarification of cane juice seems to have been first successfully employed by Schatten in 1843 although full commercial development did not result until 1880 in Java. The sulfitation process was introduced by Melsens in 1849. Soluble phosphates were suggested for both refining and raw sugar processes previous to 1850, but that seems to be the earliest date in which they were commercially employed. Phosphate defecation in refining did not

[1] Horne, *Ind. Eng. Chem.*, September, 1935.

[2] Noël Deerr, *Cane Sugar*, Manchester, England, 1911, p. 372.

completely replace blood albumin until the end of the nineteenth century, and the extended use of phosphates in raw sugar manufacture is even more recent.

87. Capacity of Equipment. Many factors enter into the selection of adequate equipment in the cane factory and average figures may be quite misleading. Local conditions, the character and richness of the cane, the desired quality of the output, the type of process, and many other considerations will influence the size and capacity of the equipment at the various stations. Formulas have been published for the different cane-producing countries, many of which are empirical.

Salinas[3] has listed in detail what he terms "adequate capacities" for Cuban factories, and a summary of the more important units is given below. These capacities should be understood to be indicative only because of the many variables which Salinas himself recognized as affecting the results. Figures for clarifying and boiling-house equipment for Natal (South Africa) as given by Watson[4] average considerably higher than those given by Salinas, probably because of richer cane and the use of sulfitation processes.

Capacities per Ton of Cane per Hour.

Revolving knives	1 set per tandem. Increases the milling capacity 10% and the sucrose extraction 0.33%.
Crushers and mills	9.6 sq. ft. of roll (for 11% fiber). Equivalent to 86.4 sq. ft. of roll surface per ton of fiber per hour.
Boilers	38.6 hp. = 336 sq. ft. heating surface. Volume of combustion chamber 1 cu. ft. for each 7 ft. of heating surface.
Juice heaters	33.6 sq. ft. Velocities: 6 ft. per second.
Clarifiers (continuous) (moderately refractory juices)	576 gal. and 24 ft. of decanting area. (16° Brix juice) 2% increase recommended for each 1° increase in Brix.
Open defecators	768 gal. and 19.2 sq. ft. of decanting area (conditions as above).
Cachaceras (mud-settling tanks)	192 gal. and 5.75 sq. ft. decanting area.
Filter presses	2.88 sq. ft. filtering surface. Pressure 30 lb.; cotton filter-cloths.
Vacuum filter	2.88 sq. ft. filtering surface. Filtering in two vacuum phases.
Evaporators (quadruple)	240 sq. ft. heating surface (sirup at 55° Brix). Imbibition 20%; vacuum 26″.
Vacuum pans	38.4 cu. ft.; of volume. 76.8 sq. ft. of heating surface. For massecuites *A*, *B*, *C*.
Crystallizers	144 cu. ft.: 13⅓% for *A*; 20% for *B*; 66⅔% for *C*.
Centrifugals	3.36 sq. ft. of screening surface. Massecuites *A*, *B*, and *C*. Velocities 1100 rpm. With higher-speed machines capacity may be reduced 33⅓%.

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[3] *12th Annual Proc. Assoc. Cane Sugar Tech. Cuba* (1938), p. 179.

[4] *Intern. Sugar J.*, June, 1933, p. 243.

Final molasses tanks	14,400 gallons. Grinding 100 days without shipment of molasses.
Sugar storage	9600 cu. ft. Grinding 100 days without shipment of sugar.

A similar study by Lopez-Ferrer[5] gives formulas for calculating capacities of various equipment under Cuban conditions.

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[5] *13th Annual Proc. Assoc. Cane Sugar Tech. Cuba* (1939), p. 235.

CHAPTER 6

PURIFICATION OF THE JUICE

88. General Considerations. The juice leaving the mills is mechanically strained as described in Sec. 57 after which it is customary in most modern factories to weigh all the juice in tank scales (Sec. 768). The purification, which is termed clarification, or defecation (originally there was a distinction in the two terms), starts after the weighing station is passed. Where any part of the hot juices or mud waters is recirculated to the mills or the clarification process (as in the Petree process and compound clarification), the cold juices cannot be weighed. The weighing is done after the clarification is completed.

The primary object of the clarification is to remove from the juice the maximum quantity of impurities at the earliest possible stage in the process commensurate with other considerations such as clarity and reaction of clarified juice. In raw sugar manufacture lime and heat are the agents almost exclusively used for this purpose, although the addition of a small amount of soluble phosphate is now general practice. The manufacture of direct consumption sugars, i.e., white sugar, "plantation granulated," "yellow clarified," or any other sugar intended for consumption without refining, involves the use of other chemicals in conjunction with lime. These processes will be considered later.

Clarification by heat and lime is known as the simple defecation process, but as will be explained in the next section the method of procedure varies widely in different localities and with differing conditions. In general sufficient lime is added to neutralize the organic acids present in the juice after which the temperature is raised to 200° F. or above. This lime and heat treatment forms a heavy precipitate of complex composition, part lighter and part heavier than the juice, which contains insoluble lime salts, coagulated albumin and varying proportions of the fats, waxes, and the gums. The precipitate is flocculent and carries with it most of the finely suspended material of the juice which has escaped mechanical screening. The separation of this precipitate from the surrounding juice is almost universally accomplished by subsidence and decantation. The different forms of defecators and clarifiers to be described later are designed to carry out this separation as completely and rapidly as possible.

The degree of clarification has great bearing on the subsequent stations of the factory, affecting the pan boiling, the centrifugaling, the quality of the products, and most important of all, the yield of raw sugar. The filtration speed and working quality of the sugar in the refinery are also affected to a marked degree by raw sugar qualities traceable to the clarification. (See Sec. 350.)

89. Systems of Clarification. The clarification process, using heat and lime only, was formerly more or less uniform throughout the world, but during the past fifteen years many modifications have been devised, not only because of the stress being placed on more efficient clarification, but also because of the need for special treatment of the refractory juices from certain variety canes, notably POJ 2878. The difficulty of clarifying this juice has been the subject of many publications, and numerous variations in practice and equipment have been brought about to overcome this. This intensive study of clarification has resulted in a better knowledge of the theoretical fundamentals as well as of the practical difficulties.

The steps in the process and the modifications or variations in each step are as follows:

1. Method of adding the lime: as milk, in batches, or continuously; as saccharate.
2. Regulating quantity of lime: periodic tests; continuous recording pH; automatic addition through pH control.
3. Time of adding lime: before heating; "delayed liming" (increased reaction time before heating); after heating; fractionally before and after heating.
4. Temperatures: boiling; superheating; heating in two stages.
5. Treating juices from different mills: single; compound and separate clarification.
6. Method of settling: open settlers; continuous closed settlers.
7. Treatment of scums: single filtration; double filtration; returning to mills; redefecation separately or with thin juices, as in compound clarification.

It is evident that combinations of the modifications of these different steps make possible a vast number of procedures. Further complications are introduced by the rather general practice of adding some form of soluble phosphate, generally to the raw juice but frequently at some later stage. Sulfitation is also employed to some extent for the manufacture of raw sugars, although it is in more general use as a white sugar process.

90. Methods of Adding the Lime. The commonest practice is to add the lime in the form of a milk to the cold juice just after it leaves the weighing tanks and before it enters the heaters. In all factories of any size lime-circulating systems are in use; the lime is made up to the desired density in a tank as a suspension in water and then circulated to the various points in the plant where lime is used, the excess returning to the supply tank. Some experiments were made in Hawaii about 1921 on adding the lime at the mills with a view to preventing inversion in and around the mills, juice strainers, etc., but results were not considered favorable. This practice is objectionable from a chemical standpoint as well as for other reasons because of the solvent action of lime on some of the constituents of bagasse fiber.

There are many who advocate adding the lime to the juice after it has been heated, generally to a temperature well above the boiling point. Geerligs[1] advises liming after heating when the gum-forming leuconostoc mesenterioides is troublesome, as this organism cannot develop in acid juice. Maurice Bird[2] published results showing that "crude gums" were much lower in juices heated to 238° F. and then limed than in those heated to 212° F. after liming. Agreement seems to be general that the gummy materials (reversible colloids, p. 93) are flocculated by heat in an acid medium, though they pass through the ordinary alkaline defecation with cold liming. Some observers claim that raw juice can safely be heated to high temperatures for a short time without fear of inversion, but the point does not appear to be well established. It will be considered further in Sec. 100 on Temperatures.

91. Intermittent Liming. The addition of the lime may be either intermittent or continuous. In the older systems using open tank defecators without heaters the lime is added directly to each defecator as it is filling with juice. Where closed heaters are used, as in all modern systems, the juice is generally limed in the cold in a series of large tanks. At least three conical-bottomed tanks should be used, so that each may be thoroughly cleaned after each filling. The lime precipitates are kept in suspension by rotating paddles or by a current of air forced through a perforated pipe. This system of adding the lime has the advantage of simplicity and permits exact regulation of each tankful to any desired reaction, since there is usually time for such regulation after the tank has been filled.

A prompt and intimate mixture of the lime and the juice is highly desirable in order to avoid local overliming. To effect this, Meeker of Central Tinguaro, Cuba, devised a simple arrangement by which the outlet valve of the lime-measuring tank is actuated by the stream of juice flowing from the juice scales into the large liming tank. In this way the lime is added in a small stream and intimately mixed with the juice as the latter enters the liming tank instead of adding it in one large dose as is frequently done in intermittent liming.

A liming apparatus devised by F. P. Henchy consists of a lime-measuring tank, two quick-opening gate valves in series (one float-operated and the other lever-operated), and a lever-operated cock; the lever system is suitably connected with the juice scale discharge lever. The milk of lime from the lime-circulating system must pass first through the lever-operated valve and then through the float-actuated valve. The connection to the float valve is adjustable to regulate the desired amount of lime in the measuring tank at the shutoff position. When the juice tank valve is opened, the outlet of the lime-measuring tank is also opened, discharging the lime into the juice stream. The float falls, opening the float-operated valve, but no lime can enter as the cutoff valve has been closed by the lever system. Closing the discharge valve to the juice tank closes the outlet of the lime-measuring tank, opens the lever-

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[1] *Cane Sugar Manufacture,* second edition, London, **1924**, p. **144.**

[2] *Facts About Sugar,* **23**, 6 (1928); *Louisiana Planter,* **72**, 13 (1924).

operated lime valve, and permits the float valve to measure the determined amount of lime for the next tankful of juice. A somewhat similar arrangement but actuated by the drop of the scale tank when full is described and illustrated by Campbell and Ayulo.[3]

92. Addition of Lime as Saccharate. The addition of lime as a saccharate has been advocated frequently particularly for hot liming. The Gilchrist process which is no longer in use first heated the juice to 238° F. and then sprayed saccharate of lime into the juice to give the desired reaction.

The addition of the lime as a saccharate has been carried out in Java by mixing 100 liters of raw juice with 3 liters of 15° Baumé milk of lime (3 per cent) and using this mixture for hot liming.[4] In Van der Jagt's method the milk of lime is mixed with about ten times its volume of cold raw juice, but this is used primarily in sulfitation houses.

Behne and Jenkins[5] found saccharate liming showed inferior results compared with ordinary liming. The difference was approximately 0.5 in purity rise in the optimum pH range, and it was apparently more marked with increasing pH.

93. Continuous Liming. Continuous liming systems add the milk of lime to the juice stream in a constant stream, generally as the juice enters the heater pump. Continuous liming devices of special type have been identified with various patented defecating systems, but obviously any method of adding the lime may be used with any system of clarification. The Fleener continuous liming device has been favorably reported upon in Hawaii.[6] It consists essentially of a weir box in the usual lime-circulating system which delivers the milk of lime to a distributing pan beneath the weir. The distributing pan is divided into two parts by a plate, one part delivering the milk of lime to the juice in the centrifugal heater pump, while the other part returns the excess lime to the circulating system. By moving the weir box longitudinally above the distributing pan more or less milk of lime falls in the part of the pan delivering to the juice. As is usual in continuous liming systems, a small pipe returns a stream of the limed juice to the liming device so that the operator may test the reaction.

The objection has been raised[7] that this type of liming device delivers a constant volume of lime and therefore does not take care of fluctuations in the flow of juice. Also the use of previously limed juice as a carrier for lime may cause local overliming with resulting development of color.

The Zitkowski continuous lime meter described by King was designed to overcome these objections. The actuating device consists of two similar weirs, with the head over the lime weir maintained in direct proportion to the head over the juice weir by means of a float-operated lever.

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[3] *Intern. Sugar J.*, June, 1933, p. 218.

[4] Bussemaher, *Archief*, 1931, p. 69; *Intern. Sugar J.*, October, 1931, p. 506 (abs.).

[5] *Intern. Sugar J.*, March, 1939, p. 107.

[6] H. G. Hall, *The Planter*, Oct. 9, 1926.

[7] King, *Ind. Eng. Chem.*, August, 1931, p. 958.

Praeger[8] raises objection to the action of the Zitkowski limer because of certain errors caused by friction, and he describes a liming device to overcome these difficulties. The original articles may be consulted for details of these devices.

The automatic continuous liming devices actuated by the pH of the juice which will be described later are the most satisfactory form of continuous limer.

94. Quantity of Lime. The quantity of lime to be added to the juice varies widely with differing conditions, but a rough average figure would be 1.25 lb. of CaO per ton of cane. Figures as low as 1 lb.[9] and as high as 1.60 lb.[10] per ton of cane are reported as average for certain seasons.

95. Test Papers and Titrations. The addition of the correct amount of lime is essential to good clarification. Too little lime will give poor settling and a resultant cloudy juice, as well as possible losses by inversion, whereas too much lime will cause darkening of the juices, increase in the gummy substances (see p. 94), increased ash due to dissolved lime salts, and high molasses output.

The earliest method of determining the correct quantity of lime was by judging the appearance of the juice and the suspended matter in a test tube after liming and heating. The particles of suspended matter should move rapidly toward the surface at the sides of the tube and descend promptly at the center, forming a compact cone. This method is still used by many defecator men, and all other methods are, in a sense, supplementary to this since the ultimate test of the correctness of liming is the rapidity and sharpness of the separation of the precipitate, together with the brilliancy of the clarified juice commensurate with avoidance of excess lime salts in solution.

Cameron[11] has suggested a test for correct liming, using calcium sucrate solution in a test tube. A juice that is underlimed will show a flocculent precipitate, and the reaction of the juice will indicate overliming.

The use of sensitive litmus papers, turmeric paper, and titrations with calcium sucrate solutions, until the adoption of hydrogen-ion concentration methods, were generally depended on, reactions being expressed as cubic centimeters of 0.1 N NaOH required to bring 10 cc. of the juice to neutrality to phenolphthalein. The use of litmus requires daylight, or artificial daylight from any of the several lamps giving light of this kind. A juice limed to neutrality to litmus usually settles slowly. In raw sugar manufacture a slightly alkaline clarification is generally practiced, although in Louisiana a faintly acid reaction is advocated. In titration with standard alkali the usual reactions would represent an acidity to phenolphthalein of from 0.0 to 0.50 cc. 0.1 N NaOH to neutralize 10 cc. of limed juice, and the corresponding pH values would be from about 7.5 to 8.5 on the cold limed juice. It is to be

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[8] *Intern. Sugar J.*, March, 1939, p. 110.

[9] (Queensland) S. B. Best, *Intern. Sugar J.*, March, 1935, p. 118.

[10] (Java) Sylvanus, *Intern. Sugar J.*, December, 1933, p. 479.

[11] *Intern. Sugar J.*, November, 1932, p. 420.

understood that there can be no definite relationship between reaction by titration and by pH. The pH gives the effective acidity and the titration the total acidity, and two juices of the same pH may differ quite widely in titrated acidity. According to Fort and McKaig, "The pH value and acidity in cane juice are the result of so many factors that apparently no simple relationship exists."[12]

96. pH Control. The introduction of hydrogen-ion concentration or pH control (see Chapter 22) into raw sugar work has marked a distinct advance in the exactness of clarification practice. Any of the colorimetric methods are so simple as to permit their use by ordinary skilled labor after the desired pH number has been decided upon and prescribed by the laboratory. It is of interest that the pH of the juice in all ripe normal canes is within a fairly close range, between 5.10 and 5.40 (see Sec. 32).

In the early days of pH control it was erroneously supposed by many practical sugar men that there was a fixed pH value at which clarification could best be secured, and that once this was established, it was not necessary to deviate from it. It is obvious that this could not be true and that the best that can be obtained is an optimum value which will vary with differing conditions. More recent investigations indicate that some earlier work tended to emphasize the benefits of high pH clarification without recognizing its objections. As was stated in the seventh edition of this *Handbook,* it seems certain that in attempting to gain certain advantages due to liming to higher alkalinities (rapidity of settling, clarity of juice, elimination of irreversible colloids, etc.) greater disadvantages may be encountered through the excess soluble lime salts and the resolution of reversible colloids, or gummy substances. A careful balancing of these and other considerations will influence the pH to which the individual factory finds it advisable to lime. Defecator and press capacity must be considered; the character of the juice is also a determining factor, and when juice has been obtained from burned or deteriorated canes, it may be advisable to neutralize excess acidity with soda ash before adding the lime.

One of the earliest extensive investigations was that of Paine and Balch[13] of the U.S. Carbohydrate Laboratory in Puerto Rico who studied the relationship between pH and defecation and found that for maximum clarification it was necessary to lime to between 8.0 and 9.0 pH on cold juice. They obtained fairly good clarification, at times, between 7.6 and 8.0 pH but never below 7.5 pH. They found that a drop of 0.2 to 0.3 pH occurs on heating juice to the boiling point, so this must be allowed for if tests are made after the limed juice has passed through the heater. These investigators showed that there is a steady fall in the pH of juice kept at the boiling point for six hours; juice that was originally limed to 8.0 pH dropping to 7.0 in three hours.

Much confusion has arisen in reporting clarification results and methods with pH numbers, because of the failure to state definitely just where the pH

[12] *U.S.D.A. Tech. Bull. 688* (1939), p. 10.

[13] *The Planter,* Feb. 12, Feb. 19, 1927.

is taken. For example, a limed juice which shows 8.3 pH when cold would be about 8.0 after passing through the heater and 7.5 or even lower as defecated juice, depending on how much time it took to pass through the defecating system. For this reason such expressions as "liming to 7.5 pH" are meaningless unless it is stated at what point the pH determination is made. Inversion takes place to an appreciable degree at high temperatures at 6.8 pH, according to the findings of Walton, Hornberger, and McCalip[14] so it is generally advisable that the juice be limed to such a point that it will reach the sirup stage at not less than 6.8 pH. Sirup reactions lower than this are quite common in Louisiana and inversion does not seem to be appreciable.

Many investigators on the relationship of pH and defecation seem to agree that liming the cold juice between 8.0 and 8.5 pH marks the optimum results for the following considerations: brilliancy of juice, volume of settlings, increase in purity between raw and defecated juice, freedom from destruction of glucose on the one hand and avoidance of subsequent inversion in the process on the other. As will be shown below, others advocate pH values even lower than these to avoid all possibility of overliming.

It should be understood that the findings of different investigators as to the optimum pH are indicative for the specific conditions and cannot be blindly followed. As King[15] says, "The thousands of milling plants throughout the world present individual problems of clarification which must be met locally. The results obtained by individual investigators cannot be applied to other conditions without investigation."

The emphasis now seems to be on the avoidance of overliming and the consequent destruction of glucose, attended by increase in color, soluble lime salts, and gummy colloids.

Some of the more recently recorded observations on the subject are those of Cameron[16] (Queensland) showing a darkening in color of clarified juice at 8.0 pH, of Salinas[17] (Cuba) who found clarity improved as pH values of clarified juices increased from 7.0 to 8.0 but noted increased color beyond this point because of the destruction of reducing sugars. (Salinas also emphasized the greater ease of clarification with juices of freshly cut canes.) Tingson[18] (Philippines) showed that increases in purity between cold mixed juice and clarified juice as great as 4.16° were secured at an optimum pH of 8.7 (in the cold juice) in one series of tests, and a rise of 3.7° purity at 10.1 pH on another, but the destruction of reducing sugars in the first case was 33.6 per cent and in the second 27.4 per cent, and soluble lime salts greatly increased. (Obviously such results would be very bad in practice.) Guanzon[19] also reporting for the Philippines, finds a marked increase in viscosity of sirups and

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[14] *Ind. Eng. Chem.*, **17**, 51 (1925).

[15] *Loc. cit.*

[16] *Intern. Sugar J.*, January, 1931, p. 10.

[17] *6th Annual Proc. Assoc. Cane Sugar Tech. Cuba* (1932).

[18] *Intern. Sugar J.*, February, 1934, p. 85 (abs.).

[19] *Intern. Sugar J.*, January, 1935, p. 32.

low-grade after-products where mixed juice is limed to 8.4 pH or above. Hartman[20] reports that in Australia the aim is generally for 7.5 pH in clarified juice. King[21] says:

> The advocates of high liming have not balanced the effect of soluble lime salts and the resolution of matter insoluble at lower H-ion concentrations and particularly the destruction of reducing sugars in alkaline solutions. High liming (above pH 8.0) should be permitted only in exceptional circumstances. Low liming (below 7.0 pH) should be advocated only when the phosphate content of the juice is low.

Supporting the objections to high liming given by King, a factory in Queensland reports[22] a pH of 6.0 to 6.4 (average for season 6.2, presumably on the clarified juice), using only 1.0 lb. of lime per ton of cane, which is said to be low for the district. Average glucose ratios for the entire season showed 5.1 for first-expressed juice, 4.7 for clarified juice, and 4.4 for evaporated sirup and therefore no inversion. Increase in purity from mixed juice to clarified juice was 1.9°, with a further increase of 0.5° for the sirup and a decrease in ash content. The work throughout the factory is favorably reported upon, and the statement is made that juices have been overlimed in the past.

Summed up, the optimum pH to which juice should be limed is dependent upon many conditions and varies with the location of the factory, the variety and maturity of the cane, the capacity of the settling equipment, and other local conditions. In general, the minimum of lime which will give clear juice with a final reaction close to 7.0 pH is desirable. High liming is to be avoided, and if clear juice cannot be obtained by simple defecation except by liming to high alkalinities, the addition of phosphate or some other modification of the process should be employed.

97. Increase in Lime Salts during Clarification. A most important point on which there is divergence of opinion is in the quantity of dissolved lime salts in the defecated juice in relation to the pH. Bond[23] says:

> The remarkable fact shown is that, though lime is actually added to the juice, its CaO content may actually decrease up to a reaction of about pH 8 in the cold. Above this point the CaO content increases rapidly. Thus it is entirely possible to clarify juices with lime and obtain a clear juice of lower CaO content than originally present.

Against this King[24] found that "at no reaction above 7.6 was there less CaO than that originally present." The work of Paine, Keane and McCalip showed that increase in the quantity of lime (and consequently the pH) increases the lime-salts content of the clarified juice, and they warn against the detrimental action (long known to practical sugar men) of these excess lime salts.

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[20] *Proc. 5th Congr. Intern. Soc. Sugar Cane Tech.* (Brisbane, 1935).

[21] *Loc. cit.*

[22] S. B. Best, *Intern. Sugar J.*, March, 1935, p. 118.

[23] *The Planter,* **74**, 21 (1925).

[24] *The Planter,* **79**, 15 (1927).

King in a later study[25] states that there is a gradual rise in the calcium content of clarified juice from pH 5.5, the increase being 200 per cent at pH 8.0 but less than 100 per cent at pH 7.0. Davies and Yearwood in some of their recent studies of clarification have found that increase in calcium salts from raw to clarified juice may not be so great with prolonged stirring, but in every case they report more calcium salts after clarification than before. Honig[26] says that "with increasing pH the lime content shows a pronounced rise, especially at values above 8.0 and in a well-conducted defecation factory should not be more than 400 mg. per liter of clarified juice."

98. Drop in pH during Clarification. It was recognized quite early in pH studies on clarification that the cold-limed juice has to be at a higher pH than was desired in the clarified juice; that is, there was always a drop in pH due to heating.

Honig and Khainovsky[27] brought out that the extent of the drop in pH which occurs on heating the juice is a function of the amount of P_2O_5 present, juices of high phosphate content showing a much more marked difference in pH before and after heating than those deficient in phosphate. They modified this statement by saying that if the pH before boiling is raised to alkalinities where tricalcium phosphate is formed directly (above 8.0 pH), the drop in pH will be smaller than at lower pH values where acid phosphates are first formed and then changed to tricalcium phosphate on boiling.

King shows that if juices of different phosphate content are limed to 8.0 pH in the cold the drop in pH from raw to clarified juice is in inverse ratio to the P_2O_5, which seems to be directly contradictory to the findings of Honig and Khainovsky given above. If juices of about the same P_2O_5 content are limed to successively higher alkalinities the drop in pH is a direct function of the amount of lime present in the juice. King also says that this drop in pH seems to be closely related to the formation of titratable acidity. However, this drop in pH due to high liming is quite a different effect from that discussed in the first part of this section. It is well known that excess lime acting on reducing sugars will form acids and that the addition of too much lime may result in an acid reaction in sugar solutions. The normal drop in pH which occurs on heating cold-limed cane juice is quite apart from this action due to excess lime. It is due to the formation of the precipitate and does not represent any change in the sugar or glucose content of the juice. A similar drop in pH occurs in refinery defecation with P_2O_5 (see Sec. 366), and the extent of the drop in pH differs with the class of raw sugars melted.

99. Automatic pH Control. From the earliest days of pH control by electrometric methods, it was recognized that a continuous record of the pH of the juice could be maintained by such methods and that the quantity of lime could be automatically controlled by suitable devices through the pH recording device, i.e., the amount of lime could be added and, therefore, the resultant pH could be controlled by the pH of the clarified juice. Several de-

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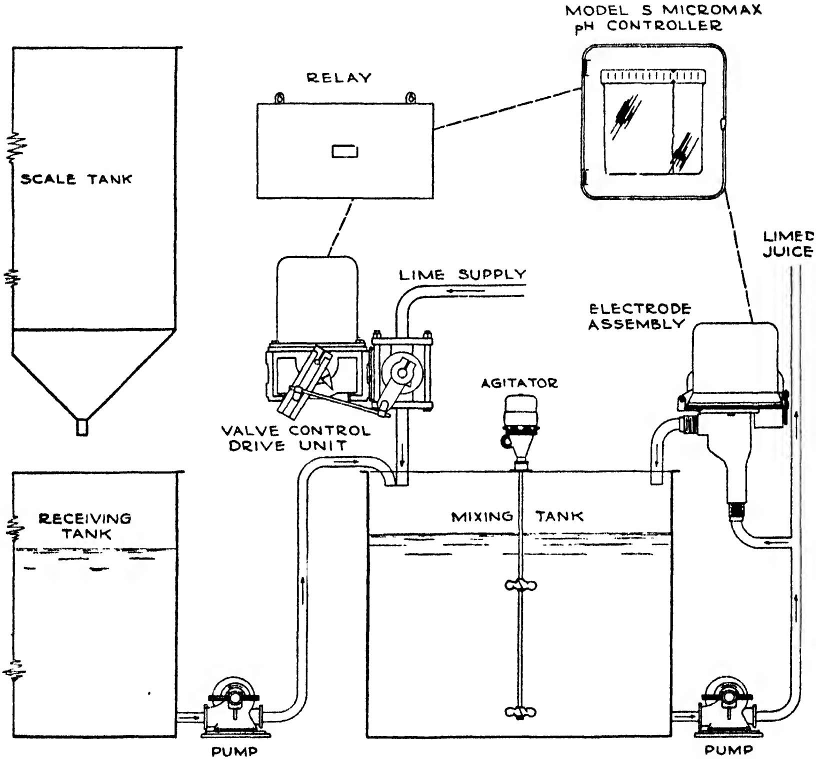
[25] *Intern. Sugar J.*, August, 1931, p. 962.
[26] *Intern. Sugar J.*, February, 1935, p. 63.
[27] *Intern. Sugar J.*, **30**, 264 (1928).

vices of this kind are now in general use of which the "Micromax Automatic pH Control" of Leeds and Northrup (Fig. 28) is representative. The description of the instrument as supplied by the manufacturer is as follows:

> This Micromax equipment performs three functions . . . measures pH continuously, indicates and records it, and controls feed of lime.
>
> The measuring circuit of the Micromax connects through electrical conduit to a pair of electrodes mounted in a flow chamber through which a continuous sample of the limed juice passes. These electrodes set up an electromotive force proportional to pH, which is measured continuously by the Micromax instrument. This instrument has a potentiometer circuit and is, in effect, an electrical balance on which the unknown electromotive force (representing pH) is weighed automatically against an adjustable standardized electromotive force.
>
> To make the reading direct in pH units, regardless of varying temperature of juice, a pair of Thermohms (electrical resistance bulbs), mounted with the electrodes, dip into the sample and connect electrically into the potentiometer circuit where their changes in electrical resistance compensate for temperature changes.



Leeds & Northrup Company.

FIG. 28. Micromax Automatic pH Control.

The operator has only to set the Micromax at the desired control point . . . thereafter the equipment operates automatically. When the pH of juice begins to depart from the control value, both the departure and the rate of departure instantly cause a change in the amount of electromotive force generated by the electrodes. The Micromax mechanism immediately actuates a motor-driven valve to increase or decrease the flow of lime to the juice. The amount of increase or decrease in lime feed is proportional to the deviation of pH from the control value. By means of a convenient dial, the operator can easily adjust the size of the correction which will follow a given deviation.

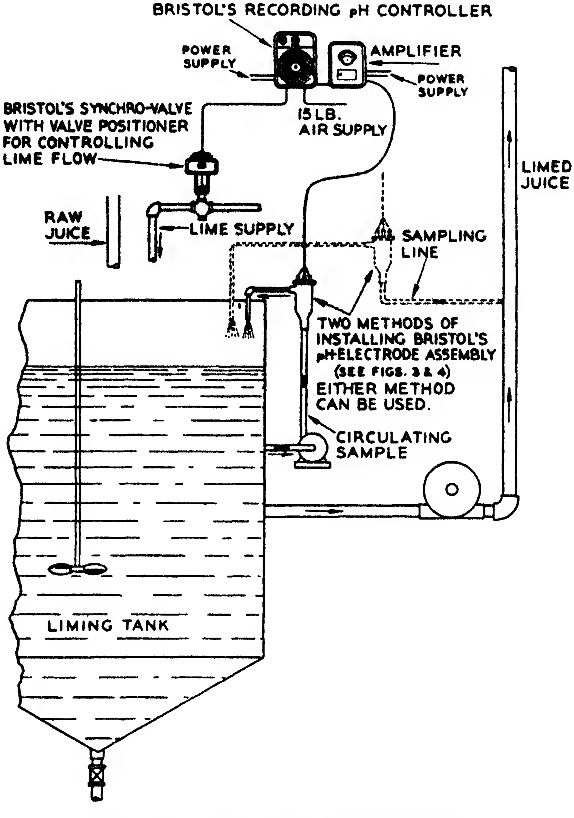


Fig. 29. Bristol pH Control System.

Lime-water of approximately 2° Baume is now widely used instead of heavy milk-of-lime. It is easier to control and minimize losses due to local overtreatment.

Each equipment consists of four essential parts . . . Electrode Assembly, Micromax Controller, Relay and Motor-Driven Valve. The electrode assembly consists of an antimony electrode, a calomel electrode and temperature compensators, all projecting through the bottom of a rubber-covered aluminum box. The lower ends project into the flow chamber through which passes a continuously flowing sample of limed juice. The Micromax is the indicating and recording controller which indicates the pH at any moment and records it continuously on a chart. The relay actuates the motor-driven valve. Included in the relay case is also an automatic droop-corrector which readjusts the normal valve position whenever there is a sustained change in the pH of untreated juice. The valve is a motor-operated unit which positions the valve quickly and accurately.

Another automatic pH control device for the addition of lime is that of the Bristol Company in which glass and calomel electrodes are used (Fig. 29).

The advantages of automatic control over manual control are obvious, provided certain precautions are taken to avoid error. The arrangement must be such that there is a minimum lag between the point where the pH is taken and the addition of the lime. Cleanliness and frequent checks of the apparatus are essential for accurate work.

Electrometric pH recording with manual control of the addition of the lime is less expensive and is more generally practiced. The pH recorder is in such a position that the operator can see it at all times, and the regulation of the lime can be altered accordingly. This gives satisfactory results, particularly when the lime is added intermittently as the juice flows from the scale tank. The time element here permits the operator to change the regulation of the lime according to the readings of the pH recorder.

A pH meter for manual control of the liming of the juice has been devised in Queensland[28] and is generally used in that country. It is a modification of the Khainovsky pH meter as used in Java and has a calomel and antimony electrode assembly. It has been found that the electrodes should be immersed in a suitable flow chamber which is self-cleaning and where a constantly flowing sample has a velocity of at least 2 in. per second.

100. Temperatures and Time of Adding the Lime. The temperature to which juices are heated in clarification varies widely, 194° F. (90° C.) and 238° F. (115° C.) being the outside limits. General practice is to heat to the boiling point or slightly above. Superheating (that is, heating well above the boiling point) gives more rapid settling and a compact mud but Balch and Paine[29] found that the clarified juice was darker and more turbid when heated to 110° C. than the same juice heated to 100°. Geerligs[30] finds no difference in the juices due to the higher degree of heating, though others claim distinct advantages.[31] It is now generally agreed that temperatures slightly above the boiling point are the maximum for good practice and that superheating is not advantageous except in unusual circumstances.

Within the past ten years there has been much study as to when is the best time to add the lime to the juice, whether before heating or after heating. This has given rise to a number of modifications of the simple defecation process in which the lime is added to the cold juice (cold liming), then is heated and settled.

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[28] *Intern. Sugar J.*, January, 1941, p. 21.

[29] *Ind. Eng. Chem.*, **20**, 266 (1928).

[30] *Cane Sugar and Its Manufacture*, second edition, London, 1924.

[31] Bird, *The Planter*, **61**, (1922); *Facts About Sugar*, **23**, 6 (1928).

Hot liming is generally understood to mean adding the lime after the juice has been heated to the final temperature. The reason advanced for the practice is that certain colloids (albumin and hydrous silica) are precipitated with heat at the pH of raw juice. Less lime is needed (15–20 per cent),[82] more rapid settling is reported, but the agreement is not general as to the removal of non-sugars or the effect on the sucrose and reducing sugar content of the juice. Muller[83] said that superheating before liming would remove organic silica compounds not removed by the ordinary process, but Guanzon[84] found no evidence of this, nor did he find any advantage in heating before or after liming so far as removal of non-sugars is concerned.

Honig states that the lower the alkalinity the better the removal of silica and sulfates.[85] Several observers have reported little or no inversion even with superheating before liming, but Guanzon[86] speaks of "heavy inversion losses with hot liming." King[87] found an average increase of 25 per cent in the glucose content of several factory runs, and individual tests showed 80 per cent increase. There was an increase in color after liming these juices. Decomposition of invert sugar through local overliming is overcome, according to Bussemaher,[88] by the use of lime saccharate so as to give instantaneous mixture, and this practice (Java) is reported to give clearer and lighter-colored juices.

Evidently there are advantages to be gained by hot liming but these are offset by the dangers of inversion and decomposition of reducing sugars. Modifications of the hot liming process have therefore been developed with a view to retaining the advantages and eliminating inversion and decomposition dangers.

In Natal, South Africa, heating to 150° F. (66° C.) prior to screening and chemical treatment was found to keep strainers in better condition by preventing gummy fermentation, and greater quantities of wax were removed.[89]

Adding the lime in doses, or fractionally, has been reported from various countries. Such a method is the Ventura system in which the juice is first limed in the cold to 6.1–6.4 pH then heated to 212°–220° F., and lime again is added to a pH of 7.4–7.8. Clarification by this method (in the Philippines) is described[90] as a fractional precipitation of colloids at definite pH's and is reported as giving better clarification than either cold or hot liming, but scale formation in juice heaters is increased. Better refining quality sugars are re-

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[82] Jenkins, *Intern. Sugar J.*, November, 1933, p. 420.

[83] *Intern. Sugar J.*, 1921, pp. 23–579.

[84] *Philippine Agr.*, 1934, No. 5, p. 353; *Intern. Sugar J.*, January, 1935, p. 32 (abs.).

[85] *Intern. Sugar J.*, February, 1935, p. 63 (abs.).

[86] *Loc. cit.*

[87] *Loc. cit.*

[88] *Loc. cit.*

[89] *Intern. Sugar J.*, May, 1934, p. 186.

[90] Pidlavan, *Philippine Agr.*, 1932, No. 2, p. 140; *Intern. Sugar J.*, November, 1932, p. 435.

ported.[41] King[42] reported favorably on a similar procedure of double liming. According to Honig[43] the most common practice in Java is to lime 40 per cent of the raw mixed juice to 9.5 pH and the remaining 60 per cent to 6.0–6.6 pH, then to heat this to 65° C. and add the cold 9.5 pH juice to give a mixed juice of 7.6–7.8 pH.

101. Fractional Liming and Double Heating. In 1936 Davies, Duncan, and Yearwood reported results with a process called "Fractional Liming and Double Heating" (abbreviated to "FL and DH"),[44] which has gained favor rapidly particularly for clarifying refractory juices such as those from POJ 2878. This consists of liming the juice to 6.4 pH, boiling, liming to 7.6 pH, again boiling and settling; the essential and unique feature is the second heating after the second liming (see Fig. 30). The advantages found are: much greater rapidity of settling, 35 per cent less lime used than for cold liming, greater elimination of non-sugars (particularly with juice from POJ 2878), reduction in mud volume, and greater clarity of juice. Additional equipment is small since the secondary heater has only to raise the temperature from about 180° to 212° F., and need be only about 10 per cent of the working capacity of the primary heaters. Primary heaters tend to keep cleaner because of the acid reaction of the juice.

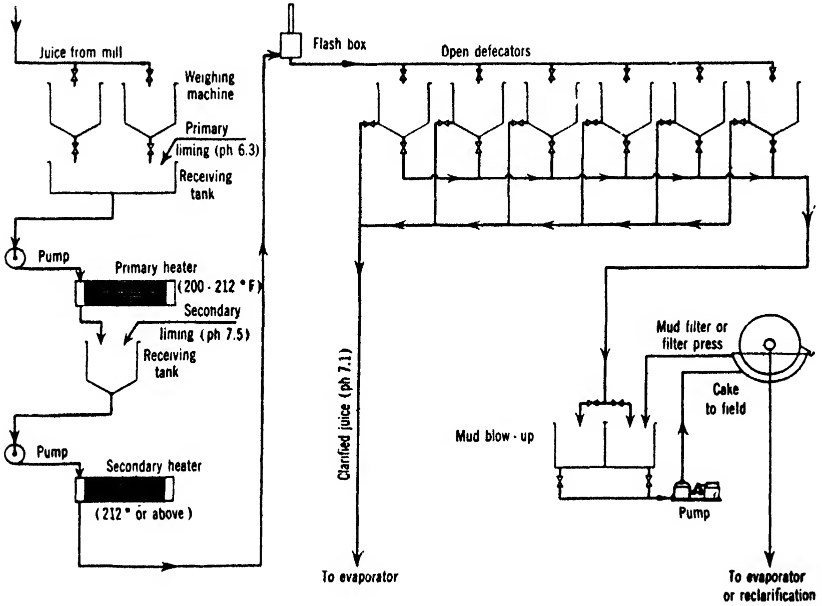

FIG. 30. Diagram of Fractional Liming and Double Heating.

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[41] Guanzon, *loc. cit.*

[42] *Loc. cit.*

[43] *Intern. Sugar J.*, January, 1940, p. 34.

[44] *Intern. Sugar J.*, August, 1936, p. 298.

The process is reported in use in Puerto Rico, Jamaica, Antigua, Trinidad, and Queensland,[45] and also in Cuba. At times better results have been obtained with primary heating to 200° F. and secondary to 215° F. The report from Guanica Central, Puerto Rico,[46] where the process is used with Dorr clarifiers states that the rise in purity from mixed juice to sirup was 1.46 in 1938, against 1.26 average for the past five years. Moore [47] described the automatic control of the pH for the secondary liming in Antigua by means of the Micromax controller-recorder (see Sec. 99), and states that temperatures and pH must be controlled between narrow limits for optimum results. Results for three crops "have made possible satisfactory clarification when ordinary methods have failed." Lime reduction of 15 to 20 per cent is also reported. The addition of phosphate and even sulfitation failed to give satisfactory results when POJ 2878 was ground, but the FL and DH process gave good clarification with all varieties.

In Queensland FL and DH has been reported to be greatly superior to the ordinary methods in practice; in fact, satisfactory operation was quite impossible with cold liming when grinding 80 per cent of POJ canes.[48] It was found that for these conditions, a primary pH of 5.7 and secondary pH of 7.8 gave optimum conditions as to purity rise and mud elimination. The difference between these pH values and those recommended by Davies may be due to the low pH of the raw juice (about 5.0). Close control of pH for both primary and secondary liming is recommended.

It is of particular interest that the FL and DH procedure has gained such impetus, since there is no commercial force behind it as there is with patented processes and special apparatus. The evidence seems to be that it is particularly well fitted to overcome the refractory nature of POJ 2878 and similar juices upon which there has been so much study and experimentation.

W. D. Nelson reported favorable results for this process in one factory in Louisiana but did not find that it worked so well in another,[49] and Aurioles [50] reports "very satisfactory results have been obtained with this clarification system" at Central Zaza in Cuba. There have been a few adverse reports, but in general the results from all over the world are in accord with those reported by Chacon and Mendez from Puerto Rico that "the FL and DH process is the most efficient and practical method of treating refractory juices (as those from POJ 2878); by heating them in an acid medium, a coagulation of viscous matter (reversible colloids) takes place whereas in cold liming such matter is held in suspension, preventing a good clarification of the juices."

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[45] Davies, *Proc. 6th Congr. Intern. Soc. Sugar Cane Tech.* (Baton Rouge, 1938), p. 900.

[46] *Proc. 6th Congr. Intern. Soc. Sugar Cane Tech.* (Baton Rouge, 1938), p. 899.

[47] *Proc. 6th Congr. Intern. Soc. Sugar Cane Tech.* (Baton Rouge, 1938), p. 910.

[48] Behne and Jenkins, *Intern. Sugar J.*, March, 1939, p. 107.

[49] *Intern. Sugar J.*, 1940, No. 2.

[50] *14th Annual Proc. Assoc. Cane Sugar Tech. Cuba*, pp. 245–247.

102. Effect of Mechanical Treatment on the Precipitate. In one of their most recent studies Davies and Yearwood[51] show that the method of stirring the precipitated floc has great bearing on the efficiency of clarification, particularly the FL and DH process. They found that vigorous mechanical stirring after the floc is formed tends to tear off particles and prevent the settling. Vigorous stirring before the formation of the floc, that is, during the addition of the lime, is beneficial, but after the floc has been formed the mechanical treatment should be extremely gentle to avoid damage to the precipitate.

They point out that these findings are in accord with accepted practice of the past ten years in pumping muds to the filter presses, where it has been found that reciprocating pumps are much preferable to centrifugal pumps. It may very well be that some of the adverse reports on FL and DH have been due to the violent stirring and consequent damage to the floc. Davies and Yearwood show that by far the poorest results were obtained with compressed air agitation. The use of compressed air for agitation has been recognized as uneconomical, and it is now proved to be actually detrimental. The results of this investigation would indicate that careful consideration should be given to methods of stirring in all clarification processes.

103. Delayed Liming. An improvement reported from Hawaii during the past few years is so-called delayed liming, by which the reaction time between the addition of the lime and before heating is from ten to fifteen minutes. This is accomplished by having an extra tank which holds a ten- to fifteen-minute supply of juice. This tank is equipped with a mixing device and its supply of juice is maintained at a constant level by an overflow or float valve.[52]

This cold digestion for ten to fifteen minutes has proved of distinct benefit, and Davies and Yearwood have corroborated the value of prolonged stirring after liming.[53] They found the benefit of prolonged stirring to be an increase in gravity purity rise, a greater decrease in ash, a smaller increase in calcium, lower turbidity, a higher rate of settling, and lower final mud volumes.

THEORETICAL CONSIDERATIONS AND NATURE OF THE PRECIPITATE

104. The Role of Phosphates. A large number of investigations have been made throughout the world as to what happens to cane juice when it is subjected to the action of lime and heat. The accumulation of evidence indicates that the phosphate content is the most important factor in efficient clarification, although it has been shown lately that this is not invariably so. Walker in one of the earliest investigations showed that the addition of phosphates to soils increased the phosphoric acid content of the juices and improved clarification. He suggested that the need for phosphoric fertilizers on soils could be measured by the brilliance of the clarified juice; that is, a juice

[51] *Intern. Sugar J.*, January, 1941, p. 8.

[52] Fleshman, *Intern. Sugar J.*, July, 1940, p. 258.

[53] *Intern. Sugar J.*, October, 1940.

which does not settle properly is deficient in P_2O_5 and comes from a soil deficient in P_2O_5.

It has been generally agreed by most investigators that the proportion of P_2O_5 in the juice should not be less than 30 parts per 100,000. McAllep and Bomonti[54] found that Hawaiian juices would not clarify properly if less than 30 to 35 parts of P_2O_5 were present. Bond[55] also showed that phosphates are the determining factor, and Paine, Keane, and McCalip,[56] working with Puerto Rican juices, proved that it was immaterial whether the phosphate was naturally present or added in the process. They found the elimination of colloids to be a direct function of the percentage of P_2O_5 present when the lime is added to a given pH.

Honig[57] says that, as a factor in clarification, the P_2O_5 content is second in importance only to the pH, but R. H. King[58] calls attention to the fact that ferric, alumina, and silica ions are also precipitated when lime is added, forming a very gelatinous precipitate which occludes considerable organic matter. King in a later article[59] says that the amount of phosphate in the juice seems to be the general limiting factor in clarification, but he again calls attention to the role of the soluble silicates. The P_2O_5 content of the juice is the vital factor in Queensland, according to Norman Smith.[60]

It may be mentioned here that the addition of some form of soluble phosphate to juices deficient in P_2O_5 has become fairly general practice throughout the world since the above findings have been reported. The methods used and results will be described in a later section (Sec. 108).

There have been exceptions reported to this rule of the more phosphate the better the clarification. With Cuban juices from different parts of the island Lanier[61] stated that juices from Pinar del Rio require less phosphate for optimum flocculation than those from Santa Clara province, and says that the P_2O_5 content is not entirely responsible for good or bad clarification. Salinas, also working in Cuba, emphasized the need of at least 0.1 per cent albumin as well as 0.030 per cent P_2O_5 in juice.[62]

Several investigators have found POJ 2878 juices to contain sufficient phosphate for good clarification, but the results in practice have been poor. McCleery,[63] in Hawaii, and Gundar Rao and Shakar[64] in India both state that POJ juices which clarify very poorly have a higher phosphate content than juices from other canes that clarify well. The latter observers suggested that

[54] *Hawaiian Sugar Planters' Record,* **26,** 139 (1922).

[55] *The Planter,* **74,** No. 21 (1925).

[56] *Ind. Eng. Chem.,* **20,** 262 (1928).

[57] *Archief,* 1934, No. 14; *Intern. Sugar J.,* February, 1935, p. 63.

[58] *The Planter,* **79,** 287 (1927).

[59] *Ind. Eng. Chem.,* August, 1931, p. 961.

[60] *Intern. Sugar J.,* January, 1931, p. 10.

[61] *4th Annual Proc. Assoc. Cane Sugar Tech. Cuba* (1930), p. 93.

[62] *6th Annual Proc. Assoc. Cane Sugar Tech. Cuba* (1932).

[63] *Intern. Sugar J.,* April, 1934, p. 166 (abs.).

[64] *Intern. Sugar J.,* November, 1939, p. 433.

the higher colloid content may account to some extent for the refractory behavior of the POJ juices. They further suggested that the explanation might lie in "effective phosphates," i.e., those in true solution and not in the colloidal state, but determinations of colloidal phosphate do not bear this out. Their conclusion was that even considering effective phosphates and total colloids, the differences in workability of juices cannot be explained, but the possibility is suggested that it may be due to the stability of the colloids.

Davies [65] showed that the juice of POJ 2878 averaged nearly three times as much P_2O_5 (0.035 per cent) as the juice of BH 10–12 (0.013 per cent), which is very easily clarified. He suggested the possibility of organo-phosphate compounds in the juice, i.e., part of the phosphate may not be available. He noted that the P_2O_5 content of the clarified juice is over twice as great in POJ 2878 as in BH 10–12 and suggests "mg. P_2O_5 to be eliminated" as the optimum for clarification; also that the variety must also be considered. In continuing the investigation, he devised a method of determining organic colloidal silica and showed that organic silica per cent of total solids is in inverse ratio to the ease of clarification, i.e., organic silica is a detrimental factor in clarification. During clarification at pH 7.2 the organic silica changes to inorganic silica compounds. These findings may account for the apparently conflicting data regarding silica removal given by other investigators.

105. Nature of Precipitate. Elimination of Ash. The precipitate formed by the action of heat and lime depends on many factors such as the variety and maturity of the cane, the technic of clarification, the pH and other variables; and Paine and Balch [66] state that "within the range of moderate alkalinity used for cane juice defecation it seems certain that few if any of the reactions are quantitative."

In a study of Louisiana juices Walton and Fort [67] found very little difference in the total ash content before and after clarification, although the composition of the ash varies with the method of clarification. Fort [68] in the most recent study of elimination during clarification found that, whereas there is usually a decrease in the carbonated ash, there is generally an increase in the total salts present. This is due to the increase in the proportion of organic salts, which were about 40 per cent of the total salt content before clarification and over 60 per cent after clarification. These investigators agreed with King that the precipitation of phosphate is a function of the pH and calcium. Honig [69] says that P_2O_5 removal is very much dependent on the pH up to 7.0, but above that only a slight effect is observed. He also says that in the precipitation of silica, the lower the alkalinity, the better the removal, but also that the greater the phosphate content of the juice, the lower will be the silica in the clarified juice. King gives conflicting data regarding

[65] *Proc. 6th Congr. Intern. Soc. Sugar Cane Tech.* (Baton Rouge, 1938), p. 900.
[66] *Loc. cit.*
[67] *Facts About Sugar,* October, 1932.
[68] *Facts About Sugar,* September, 1939.
[69] *Loc. cit.*

silica removal, showing 70 per cent removal at pH 10 and only 35 per cent at pH 8.0. He agrees with Honig that silica removal is desirable as giving less scale in heating equipment and better filterability in the commercial sugar.

The iron and alumina are almost completely removed at pH 8.0 according to King and Walton and Fort, though Honig states that good results for well-defecated juice are 30 mg. of Fe_2O_3 and 10 mg. of Al_2O_3 per liter. The sulfates, if low in the raw juice, are not removed to any extent; but if they are present in excessive amount (600 mg. per liter) some elimination results (King, Fort and Walton, and Honig). According to these authorities, magnesium removal is a function of the alkalinity and rises sharply between 8.5 and 9.0 pH.

The changes in the lime content of the juice during clarification have already been commented upon (see Sec. 97), and it has been shown that high liming or a strongly alkaline clarified juice is to be avoided. King and Tingson[70] have emphasized that when acids are formed during clarification from the destruction of glucose or through the souring or deterioration of juice the calcium content of the clarified juice increases tremendously. Honig places the upper limit of CaO in a well-clarified juice at 400 mg. per liter. There is no doubt that the CaO content of the clarified juice should be held to the lowest possible limit commensurate with clarity. Fort[71] found almost without exception an increase in lime content due to clarification (7.0 pH clarified juice). The amount of lime increase was dependent on the cane variety to a great extent. Fertilizers and soil conditions also influenced the increase. There seemed to be a relationship between lime increases, residual nitrogen compounds, and total titrated acidity. These two factors may indicate nitrogenous acids.

106. Elimination of Organic Non-Sugars. The removal of fine particles of cane fiber may be considered under this head although bagacillo is not entirely organic in composition. The benefits of fine screening have been considered previously (see Sec. 59), and the solvent action of lime and heat on cane fiber has been studied by many observers, furnishing further arguments against high liming. The removal of all bagacillo before clarification, although desirable, has an adverse effect on settling. The removal of colloidal non-sugars has been widely studied, and the results present many apparent problems, possibly because of the difficulty of estimation of colloids and also because of variations in the character of juices, methods of clarification, and degree of liming. The degree of milling is a great factor in the colloid content of the juice as is also the amount of saturation water, and it has already been shown that lime and heat acting on cane fiber particles form inorganic colloids in the juice. The colloids eliminated in the process have been shown by Paine and his colleagues to be largely the irreversible type, i.e., those colloids generally high in ash which after drying will not redissolve or redisperse when taken up with water again. The reversible colloids (which

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[70] *Loc. cit.*

[71] *Loc. cit.*

consist of the gummy substances low in ash) pass through the defecation process almost untouched. They also showed that with an increase of lime salts in the defecated juice the amount of these reversible colloids or gummy substances increases in an almost direct linear relation, the assumption being that the excess lime over that needed for defecation either converts some of the irreversible colloids into the reversible type; or that it has a peptizing effect on the pectinous materials of the juice, or that it enters into direct chemical combination with the reversible colloids.

Excessive lime in defecation has long been known to give poor-working, low-grade materials in the boiling house and dark-colored, gummy sugars. The work cited above offers a scientific argument in favor of avoiding the use of more lime than the minimum needed for good clarification.

Albumin has been recognized as one of the precipitating agents in clarification, and Salinas [72] has suggested a minimum of 0.10 per cent in conjunction with 0.030 per cent of P_2O_5 as necessary for good clarification. The greatest elimination of nitrogen compounds has been found to be at pH 6.5, and this has been recognized and made use of in the fractional liming and double heating method of clarification (see Sec. 101). Davies proved that protein elimination is quite different with different varieties and different methods of clarification. With the regular defecation process (cold liming) he found BH 10–12 showed 83 per cent protein elimination and POJ 2878 54 per cent, but with FL and DH the BH variety showed 94 per cent and POJ 2878 84 per cent. Similarly, colloidal elimination (as measured by dye value, Sec. 323) was 32 and 18 per cent for the same varieties with cold liming, and 36 and 22 per cent respectively with FL and DH.

The removal of so-called gums, i.e., the material precipitable by alcohol, more correctly referred to as alcogel, as studied by Walton and Fort [73] in Louisiana, was found to range from 18.7 to 53.2, the lower figure being for simple defecation and the higher for sulfitation. They found wide variations in the gum content of juices of the same apparent purity and even the same organic non-sugar content. Dymond [74] in South Africa found the alcogel removal to range from 36 per cent for simple defecation, 42 per cent with added phosphate, to 65 per cent with sulfitation and phosphate. Fort found 22 per cent gum removal with sulfur-lime defecation but less with lime alone.

The removal of wax during clarification is evident, since a large part of the wax of the raw juice is present in the press cake. It is probably absorbed by the flocculent precipitate, or it may lose its waxy nature by taking on water at boiling temperature as suggested by Dymond.[75] That the cane wax persists through all the processes of the raw sugar mill and through the refining

[72] *Loc. cit.*

[73] *Facts About Sugar,* October, 1932, p. 440.

[74] *Proc. South African Sugar Tech. Assoc.,* 1936, p. 81; *Intern. Sugar J.,* December, 1936, p. 480.

[75] *South African Sugar J.,* 1925, p. 242.

process has been proved by two studies of C. F. Bardorf [76] who found this material in raw sugars and in the foam from sugar liquors in the refinery.

107. Increase in Purity. Fort [77] states that the two most important factors in purity increase during clarification are the phosphate and protein content of the juice. He believes the purity rise to be an inherent quality of the juice due to these components, and he questions whether the relative merits of different clarification procedures can be judged by purity increase unless the comparison is made on portions of the same juice. Davies and associates [78] make the following statement: "A rise in apparent purity during clarification or evaporation is generally taken as indicative of the elimination of solids, but it may also indicate the destruction of solids, which is seldom considered." Their work shows that this actually occurs during evaporation and pan boiling giving a rise in purity although sucrose and reducing sugars have actually been lost.

108. Addition of Phosphate. Soon after the findings regarding the value of phosphate in juice was reported, addition of P_2O_5 in some form to refractory juices became prevalent and is now common practice throughout the sugar world. In a large factory in Cuba [79] powdered triple superphosphate of the type used in fertilizer is added to each scale tank of juice in the amount of 0.44 lb. per ton of cane. The material, containing 48 per cent P_2O_5 and costing $40 a ton at the factory (in 1938), is dissolved in small cast-iron tanks, and the proper amount of solution is added to each scale tank of juice. The proportion added equals 10 parts P_2O_5 per 100,000 parts of juice, and since the untreated juice averages 18 to 20 parts the juice after treatment approaches 30 parts. The cost of the treatment is close to one cent per bag of 330 lb. of sugar or 0.3 cent per 100 lb.

Better clarification, improved refining quality of sugar, and less darkening of sugars in storage have resulted from this addition of phosphate.

Another Cuban factory adds ¼ lb. of the triple superphosphate to the cold juice per ton of cane (6 parts P_2O_5 per 100,000) as regular practice but adds a second dose to the hot juice in open settling tanks with such refractory juices as POJ 2878. The total added phosphate sometimes reaches 2 lb. per ton of cane. The juice is much cleaner, and the resulting sugars of better refining quality.

Reports from all over the world might be cited to show the improvement due to added phosphate. The amount generally added is sufficient to bring the P_2O_5 content of the raw juice to 0.30 or 0.40 per cent. The advantages are clearer juice, greater colloid elimination, less lime salts in clarified juice, more rapid settling, faster mud filtration, better sugars, and better working quality of low-grade materials in the factory. The disadvantages are greater

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[76] *Ind. Eng. Chem.*, March, 1928, p. 258; April, 1929, p. 366.

[77] *Loc. cit.*

[78] *Sugar*, December, 1942, p. 29.

[79] *Proc. 6th Congr. Intern. Soc. Sugar Cane Tech.* (Baton Rouge, 1938), p. 1032.

mud volume, higher lime consumption and greater expense. Most users are convinced that increased yields more than offset the extra cost.

It is obvious that the addition of phosphate may be practiced not only with simple defecation but also with modifications such as hot liming, fractional liming, or delayed liming, or with sulfitation, and all these methods have been reported as in use. McCleery[80] found that POJ juices in Hawaii could be satisfactorily clarified by adding lime to pH 11.0 and then reducing the reaction with phosphoric acid, but three times as much lime was needed, and the volume of mud was excessive. Nothing is reported about destruction of glucose. In Australia the addition of phosphate to the FL and DH process was found to give added improvement (in comparison with phosphate and cold liming) but the volume of muds was greatly increased.[81]

Phosphosulfitation is reported in use in Mauritius, South Africa, and Java, details of which will be given later in discussing sulfitation.

TREATING JUICES FROM DIFFERENT MILLS

109. Basic Idea. The juices expressed by the different mills in the train have been shown to have different composition, which is exaggerated with heavy maceration and the high pressures used in modern milling (see Sec. 53). The common practice is to combine all the juice which is not returned to the mills for maceration and send the combined juice through the clarification system. Obviously this is the simplest method of clarification, but it means that the portions of the juice of high purity, low ash, and low colloid content are treated together with those portions of lower purity, higher ash, and high colloids. A separation of these different juices naturally suggests itself, and methods have been devised for such treatment.

110. Compound Clarification. This system is advocated by the Petree-Dorr Company for use with Dorr clarifiers and has found wide acceptance in all sugar-producing countries. It is the only generally used system for separate treatment of juices from different mills. The juices from the crusher and first mill (called primary juices) are sent to a primary Dorr and heated from 212° to 220° F., and the primary clear juice goes to the evaporators (see Fig. 31). The primary mud is mixed with the juice from the second mill, and this mixture goes to the secondary Dorr (about three-fifths the size of the primary clarifier); the secondary clarified juice is returned to the primary Dorr; the secondary mud is filter pressed; press juice is also mixed with the secondary clarified juice, the cake being discarded. Juice from subsequent mills is used for compound maceration. General practice is to lime the secondary juice to pH 6.5, thereby gaining the advantages (for this portion of the juice, at least) described in Sec. 101 for fractional liming or preliminary treatment in an acid reaction. A further advantage claimed is that the compound system removes

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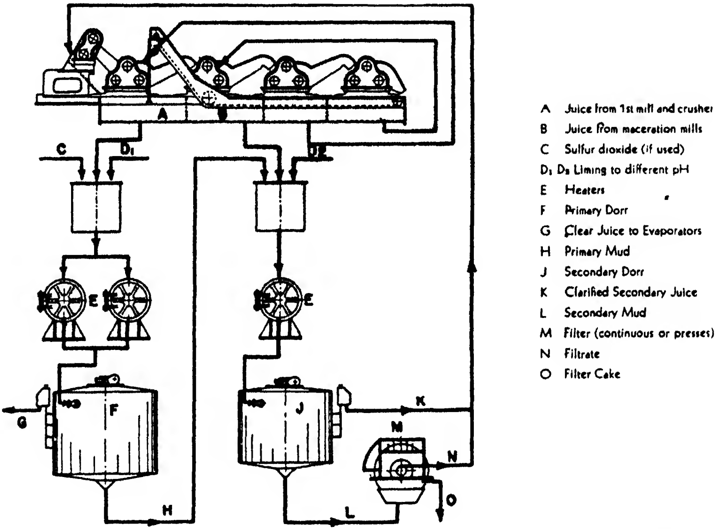
[80] *Rept. Hawaiian Sugar Planters' Assoc.*, September, 1933, p. 36; *Intern. Sugar J.*, April, 1934, p. 166.

[81] Smith, *Intern. Sugar J.*, July, 1937, p. 267.

the first flocculated material by decantation, whereas in the FL and DH system the floc remains in the juice for the second liming and heating, and some of this precipitate may be dissolved, or the colloids may redisperse. This point has not been subjected to proof, but it appears possible.

Compound clarification, in addition to the advantages claimed for the single Dorr clarifier (see Sec. 120), shows a greater elimination of colloids and an increased rise in purity over single defecation, according to Paine, Keane, and McCalip,[82] which, as stated above, may be due in part to the clarification of the secondary juice at pH 6.4.



The Dorr Co.

FIG. 31. Compound Clarification.

Because of the return of secondary clear juice to primary raw juice and primary mud to secondary raw juice the ordinary means of chemical control (weighing the cold raw juice) cannot readily be used. The boiling-house control begins with the clarified juice as it enters the evaporators, and this entails the weighing (or measuring) of a very hot liquid. The entire clarification system is therefore omitted from the control. E. M. Copp [83] of the Petree-Dorr Company reports comparative figures in a Peruvian factory in which both the hot and cold juice were weighed. He advances the idea that weighing the hot clarified juice is more accurate than weighing cold juice because the insoluble matter has been eliminated. The insoluble matter is included in the weight of the cold juice, but the polarization data and analyses consider only the soluble solids, and this may introduce a source of error. Copp's

[82] *Ind. Eng. Chem.*, **20**, 262 (1928).

[83] *Facts About Sugar,* **1932**, No. 8, p. 349.

figures show that there "is practically no clarification loss of sucrose even when running an acid secondary juice." These results are certainly contrary to what would be expected and contrary to general sugar house experience. Over 10 per cent of the levulose was destroyed during clarification, and there was a sharp change in the relationship of true sucrose to polarization before and after clarification which would appear to indicate losses.

The compound system has been the subject of many favorable reports from various countries, and it has been commended as a means of clarifying the refractory juices of POJ canes. However, a recent report[84] states that in comparative runs at Fajardo, Puerto Rico, "simple defecation is demonstrated to have appreciable advantages (clearer juice, sucrose recovery, evaporator efficiency) over compound defecation."

111. Separate Clarification of Juices. This process treats the lower test juices from the last mills separately and then keeps them separate through clarification, evaporation, and even through boiling to grain. The process as practiced at Central Moron, Cuba, is described by Gonzales Maiz[85] as follows:

> The process consists of separately treating juice from crusher and first two mills, called juice "A" approximately 65–75% according to imbibition; and juice "B" from the three or four last mills (approximately 25–35% according to imbibition). These juices are separately limed, heated and evaporated to form two sirups "A" and "B." Sirup "A," which is richer, is used to crystallize. Instead of macerating with residual juices, water alone is employed on the bagasse, and this has prevented an appreciable diminution of the purity of the juice. The advantage of this process lies in the separate treatment of two types of juice of different purities. Juice "B" contains a high percentage of colloids derived from wax, gums, pectin, starch and proteins, of difficult flocculation, extracted through imbibition and high pressures at the last mills.
>
> Both types of juice require a different alkaline reaction. The great dilution of the residual juices affords a greater difference between the density of the floc and that of the clarified juice, permitting a rapid settling. This results in an appreciable increase of the purity of the residual juices after clarification in relation to the raw juice from which it is derived. Juice "A" containing a high Brix, but a low colloidal content, has an excellent defecation.

This system is further discussed by Salinas.[86] He lists a series of advantages among which are raw sugars of high refining quality and lower molasses factor. He says that the system does not involve extraordinary expense and does not require greater clarifying capacity than other systems.

The same general process has also been reported as in use in various parts of the world, particular advantages being cited for it in white sugar manufacture by Fort and Walton[87] in Louisiana. There can be no doubt that there is much to commend the idea, if practical considerations are such that it can be carried out. It is an axiom in sugar manufacture and refining that ma-

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[84] Ramos, *Rev. agr. Puerto Rico,* **33,** 393 (1942) (abs.); *Sugar,* December, 1942.

[85] *10th Annual Proc. Assoc. Cane Sugar Tech. Cuba* (1936), p. 46.

[86] *13th Annual Proc. Assoc. Cane Sugar Tech. Cuba* (1939), p. 165.

[87] *Sugar Bull.,* **11,** No. 6, 5 (1932).

terials of different purities should be kept separate wherever possible, particularly where the character of the impurities differs as sharply as it does in the juices from the first and last mills. Many advantages suggest themselves for the separate treatment and clarification of the two classes of juice. For example, if phosphate is to be added it would probably be more effective if added largely or entirely to the last mill juices where the colloid content is higher, rather than to mixed juice as is customary. Additional weighing tanks would be required to maintain the normal methods of control of the factory. If desired such modifications as fractional liming and double heating could be practiced on the secondary juice or on both classes of juice.

R. H. King[88] suggests combining the last mill juice with mud from settling tanks, liming to 10 pH and resettling, then reusing the clear juice for tempering the mixed juice. He found this procedure to give very much better clarification than straight treatment of the mixed juices. He recommends working the secondary clarification at 90° C. to avoid the destruction of glucose, and he also suggests separate weighing of the juices to avoid difficulties in the control.

112. The Petree Process: Return of Muds to the Mills. The Petree process was the forerunner of compound clarification already described. The distinctive idea is the return of the mud waters, properly diluted with thin juice from the last mill, to the blanket of bagasse behind one of the earlier mills, the bagasse acting as a filter to remove the precipitate from the mud waters. Thus the mud is removed from the process and sent to the furnaces with the bagasse, and the filter press station is entirely eliminated. The flow diagram (Fig. 32) shows the method of handling the juices and the difference between the Petree process with the return of muds to the mills and compound clarification as now practiced.

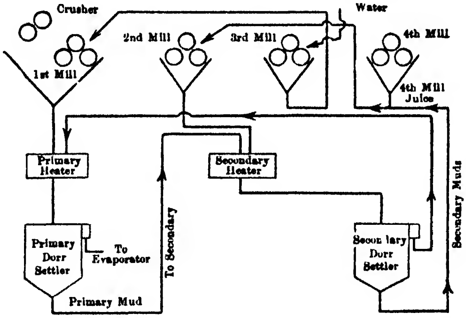

FIG. 32. Flow Diagram of Petree Process.

The advantages of the Petree process (as distinct from compound clarification) rest almost entirely in the elimination of filter presses. The large

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[88] *Sugar News,* 1933, No. 11, p. 533.

number of men needed to operate the presses are removed from the payroll; first cost and repairs to press cloths are saved; repairs and renewals to presses are eliminated, and the whole process is simplified. These outward savings are so evident and impressive that the process gained rapid favor when it was first introduced (about 1923).

The money savings brought about by doing away with the filter presses were soon found by many to be more than offset by less evident disadvantages. Re-expression of part of the mud from the bagasse and its return to the process led to a circulation of impurities with losses of sugar that were evident in spite of the fact that the control could not include losses in clarification (as already discussed under compound clarification). Mechanical difficulties in milling reduced extractions to a noticeable extent, so that the process began to be abandoned almost as rapidly as it had been adopted at first. Return of muds to the mills is still employed in a considerable number of factories, but the original commercial proponents of the plan no longer advocate it in connection with compound clarification, which is at least indicative that the elimination of the filter station, with its obvious attendant money savings, is not sufficient to overcome the operating and technical difficulties occasioned by returning the mud to the mills.

113. Sulfitation in Raw Sugar Manufacture. The use of sulfur dioxide in addition to lime for white sugar manufacture is common practice in many parts of the world (see Sec. 125), but its use to aid defecation for raw sugar manufacture where juices are refractory has been adopted occasionally in some sections. Hawaii[89] reports that with POJ 2878 and the difficulties occasioned by grab harvesting (see Sec. 17) sulfitation was found to be the best remedy and was in use in nine factories in 1935, six of which had rotary Glens Falls type sulfur burners with fan blowers and tower absorption. (See Sec. 131.)

APPARATUS AND PROCESSES

114. General. Since the beginning of modern manufacture of raw sugar there have been an almost endless number of devices patented for separating the precipitate from the clear juice. The principle universally used is subsidence and decantation, filtration being rarely resorted to in raw sugar manufacture. The settling devices or subsiders fall into two main classes, those which are intermittent in their operation and those which operate continuously. The latter are the more modern type and new designs are constantly being patented.

J. G. Davies in a monograph on simple clarification[90] shows diagrams of twenty-one different continuous settlers which were in more or less general use at the time of publication and several new designs have been brought out

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[89] McCleery, *Repts. Assoc. Hawaiian Sugar Tech.*, 1935; *Intern. Sugar J.*, September, 1936, p. 358.

[90] Published by Imperial College of Tropical Agriculture, May, 1936 (full bibliography, 176 references).

since that date. It is obviously outside the scope of this chapter to give descriptions of all such apparatus and only those in common use will be described.

115. Defecation and Clarification with Open Tanks. This is the process that was practiced since the early days of the cane sugar industry, with little improvement until comparatively récent years. It is now little used except in the smallest factories and is described here for its historic interest.

The raw juice is pumped from the mill tanks to double-bottomed vessels termed "defecators." The inner bottom of the defecator is of copper and the outer shell of iron. As soon as the bottom of the defecator is covered with juice, milk of lime is added to it to neutralize the acids and steam is turned into the space between the bottoms. The workman regulates the steam pressure so that by the time the defecator is filled with juice the latter will have nearly reached its boiling point. The moment the thick scum that covers the surface "cracks," the steam is shut off, and the juice is left undisturbed for the subsidence of impurities.

If the quantity of lime required has been properly gaged, a part of the impurities rise to the surface with the scum, forming the "blanket," and a part settle to the bottom of the defecator.

Several defecators are usually arranged in a series and are filled with juice in regular order. Sufficient time is allowed after the cracking for settling, and then the mud is drawn off from the bottom of the defecator and sent to the filter presses, the clear juice is run into the evaporator charge tanks or into clarifiers, and finally the scum is sent to the presses.

In addition to the defecation process as already described, clarifiers or eliminators are frequently used. The ordinary clarifier is a tank fitted with steam coils and a mud gutter. The clear juice is drawn from the defecators into the clarifier, where it is boiled and skimmed and is then run into settling tanks.

116. Closed Juice Heaters. The heating of the juice, either before or after liming, preliminary to the settling period is done almost universally in closed heaters generally of the type originally patented by Deming. These are horizontal banks of tubes through which the juice flows in a cast-iron casing into which exhaust steam or vapors from effects are admitted. The essentials of an efficient heater are that the juice should move at sufficient velocity to produce a scouring effect and that the condensate should be removed from the tubes quickly. A speed of flow of 5 or 6 ft. per second is recommended to keep the tubes from fouling too rapidly, and multifold circulation in the heater gives the proper flow speed and insures effective heat interchange. (See Chapter 10 for more detailed description.)

117. Open Settlers. A fairly large proportion of the raw sugar in the world today is still made with intermittent settling in open tanks, but the trend is strongly toward the continuous types of clarifiers to be described later. The process is a modification of the old defecation process described in Sec. 115. The juice is first limed in the cold in a series of large liming tanks as previously described and thence through closed heaters of the type described above. The temperature of the juice is generally raised to the boiling point or a little above. The heaters deliver the juice into large settling tanks generally provided with heating coils. Sometimes the juice is simply settled and drawn off from the mud, but much better results are obtained by the use of coils to reheat the juice to the cracking point before settling.

Spencer preferred a rectangular defecating tank, with rounded corners and the draw-down arrangement described below. These tanks may be of large capacity in excess of 6000 gal. Though a deep tank requires a rather long time for settling, it has the advantage of yielding a smaller proportion of mud. In so far as possible, it is preferable that the juice be conveyed from the defecators through pipelines rather than canals.

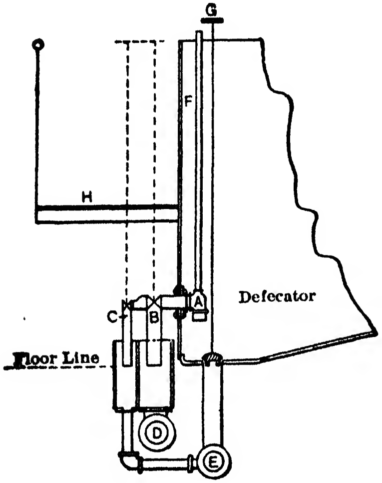

FIG. 33. Draw-Down Pipes for Defecators.

The actual average working depth of the tanks should not exceed 6 ft. The bottom of the tank should slope sharply from the rear end toward the front and from the sides to the center. This incline is necessary to facilitate the removal of the precipitates (mud, scum, or cachaza). Copper coils about 4 in. in diameter should provide approximately 25 sq. ft. of heating surface per 1000 U.S. gal. of actual juice capacity in the tank (30–35 sq. ft. per ton cane per hour). The coils should be well above the bottom of the tank to facilitate the removal of the mud.

The draw-down arrangement is shown in Fig. 33. At *A* is a side outlet elbow, with a ventilating pipe, *F*, leading above the top of the tank. In drawing down, the juice flows through the nipple, the elbow, *A*, and the cross valve, *B*, into the small collecting box, and thence to the trunk line, *D*. The angle valve, *C*, is opened for a moment before drawing down the juice for the removal of mud that may have settled in the pipeline or that may be near the outlet nipple. The valve, *G*, is used in washing the mud into the main line to the scum tanks, *E*. It will be noted that the pipe is ventilated to prevent syphoning and that the juice therefore stops flowing when its level reaches that of the bottom of the draw-down pipe and no mud can be drawn down. The elbow and nipple insure that juice shall be drawn from below the scum. It is advisable to locate about three or four draw-down pipes in each defecator, with difference of level of about 4 in., measured between centers. The lowest pipe should be about 12 in. above the bottom of the tank. The draw-down pipe should be about 3.5 in. in internal diameter in a defecator of 4000 gal. working capacity. If the juice has been properly limed and heated, there is no probability of drawing down mud, provided a sufficient settling time is allowed. Ample tank capacity and proper supervision of the liming are essential. Eight 5000-gal. tanks are sufficient for a grinding capacity of about 1400–1500 tons of cane per day (80 cu. ft. per ton cane per hour). These figures are in accord with other standards which prescribe that the number of units shall be of such size and capacity that filling will take place in fifteen to twenty minutes, and that there shall be a minimum of one and a half hours for settling, but they were devised when juices were easier to clarify than at present. Salinas[91] gives figures about 25 per cent higher than those for modern conditions in Cuba.

Round tanks with conical bottoms have been advocated by many authorities as giving more rapid settling but Tromp[92] says that in his experience no difference has been found in the time of settling in round or rectangular tanks.

The conditions governing the design of intermittent settling tanks are given by Warren[93] in an extensive study of intermittent clarification. Briefly they are: (1) smaller units increase the effective settling time; (2) bottom filling and breaking the force of the incoming juice with a baffle permits settling to start before filling is complete; (3) round tanks give more rapid settling; (4) the larger the number of draw-off valves the more rapid the settling and discharge.

More recently Davies[94] has extended the study of the effect of the flow of the incoming juice and has found that very high performance can be obtained in intermittent settlers by the use of a center well consisting of a cylindrical casing of about 10-in. diameter into which the juice inlet discharges at the top and which reaches to about 1 in. above the normal mud level (about 7 in.). The benefits of this easily installed and inexpensive device are due primarily to low inlet velocity of the juice, which cuts down swirls and turbulence. Much faster settling and decreased mud volumes result.

Davies[95] has also shown that with these center wells (or feed wells since they are not located in the center of the tank) it is possible to arrange the intermittent type of subsiders or settlers for continuous operation, and the plan has been put into actual practice.

118. Cleaning the Heating Surfaces of Defecators and Juice Heaters. The heating surfaces of coil defecators used in combination with juice heaters foul but little. Those in which the whole defecation process is conducted require frequent cleaning. Dilute muriatic acid is used.

Juice heaters usually foul very quickly. The usual method of cleaning is to circulate hot caustic soda solution through the tubes and follow this first

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[91] *6th Conf. Assoc. Cane Sugar Tech. Cuba* (1932).

[92] *Machinery and Equipment of the Cane Sugar Factory,* London, 1936, p. 367.

[93] *Intern. Sugar J.,* 1928, p. 666.

[94] *Intern. Sugar J.,* July, 1940, p. 245; October, 1941, p. 306.

[95] *Intern. Sugar J.,* November, 1941, p. 335.

with water and then with hot dilute muriatic acid. The soda solution may be returned to a storage tank for repeated use, decanting it from the mud and adding caustic soda from time to time to maintain a strength of about 1 lb. of soda to 7 gal. of solution. Occasionally the scale in the tubes is of a very resistant nature and must then be removed by scraping the surfaces. If the tube area is properly adjusted to the volume of the juice, so as to force a very rapid current, the scaling is much reduced. (See also Sec. 217 *et seq.*)

SETTLERS FOR CONTINUOUS CLARIFICATION

From the beginning of modern sugar house practice there have been a great number of attempts to perfect some form of closed continuous clarifier. These did not gain favor until about 1920, but since then several patented systems have been brought out, many of which are now in successful use. The earlier patents were mostly based on Deming's system, whereas the later styles have been largely the flat shallow-tray form of which the Dorr clarifier is typical, so both these types will be described in detail, and other forms will be compared with these.

The advantages of continuous clarification are savings in labor, fuel, and space; reduced filter-press capacity requirements because of denser muds; reduced losses in press cake; and reduced sugar losses at the clarification station. If the capacity of the continuous clarifier is ample for the requirements, it will give improved clarification which will in turn mean better work in the boiling house and better sugars.

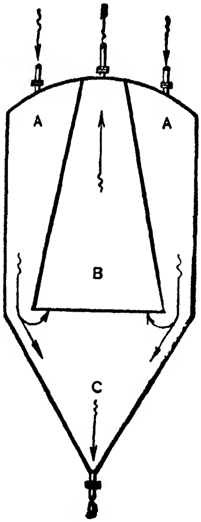

FIG. 34. Deming Settling Tank.

119. Deming's Process. This method came into prominence about forty years ago, but it was largely superseded by intermittent settling (described in the previous paragraphs) until lately when other forms of continuous systems have come into use. Probably no factories are now operating with the original Deming process, but some Deming separator tanks are still in use. As the forerunner of modern continuous systems it is of interest from the historical viewpoint. The cold-limed juice is heated to approximately 235° F. and is then passed into an eliminator where it parts with the gases, is slightly concentrated, and warms the incoming cold juice on its way to the heater. The eliminator is a cylindrical closed iron vessel with a conical bottom and is provided with a large heating surface in copper tubes. Cold juice circulates through the tubes and condenses the steam set free when the hot juice enters the lower section of the eliminator. A partial vacuum is produced by this condensation, and the air and other gases are withdrawn from the hot juice. The upper part of the eliminator is connected with a pump or the vacuum system.

The temperature of the juice should be reduced to about 210° F. in the eliminator. From here it is pumped into the outer compartment, *A*, of the closed settler or pressure separator, shown diagrammatically in Fig. 34. The mud is drawn off continuously from the conical bottom of the tank at *C*, and the clear juice is discharged from the central cone, *B*. In many factories using Deming's heaters and settlers, the juice is heated only to 218° F.

In his first experience with these tanks, the earliest of their type in Cuba, Spencer noted that the mud drawn off after several hours' operating had an offensive odor, indicating decomposition. He immediately installed very slowly moving scrapers in the tanks to prevent the mud from settling on the conical bottom of the separator. Certain spores resist the high temperature of the heater and cause the decomposition. In Cuban practice it was found necessary to liquidate and clean the tanks as often as may be practicable.

120. The Dorr Clarifier. This is a continuous system of defecation first introduced in Cuba about 1918 which has gained rapid favor throughout the various sugar-producing countries. The Dorr clarifier is used either for simple clarification or for double defecation in the operation of compound clarification (Sec. 110).

The multifeed Dorr [96] (see Fig. 35) is essentially a cylindrical steel tank having an inverted conical bottom of comparatively slight slope and divided by parallel steel trays of similar slope into a flocculating cell, *A*, shallow settling compartments, *B*, and a somewhat deeper mud-thickening chamber, *C*. The cover is in the form of a truncated cone which serves also to support the revolving mechanism. The choice of diameter of the tank and the number of settling compartments depend upon the capacity required and the floor space available. The drive may be either a steam engine with countershafts and belting or an electric motor with speed reducer, chain, and sprockets, connected to the worm gear which rotates the vertical center tube.

The central tube, *D*, extends downward into the upper part of the mud-thickening chamber, and suitable openings provide for the passage of the flocculated solids and juice from the flocculating cell, *A*, into each compartment below it. In all compartments, radial arms are rigidly attached to the center tube and revolve clockwise with it. These arms are provided with adjustable squeegee blades, *K*, so arranged as to scrape the entire surface of the trays and bottom, gently pushing the sediment towards the center. The small amount of solids settling in the flocculating cell itself enters the central tube with the juice through the ports provided. From each settling compartment, the mud is discharged through an annular opening around the central tube, and it continues downwards, between the tube and a concentric boot, to the mud-thickening chamber where it reaches its maximum density. The scrapers in this bottom chamber push the mud into a small central inverted cone, *E*, of relatively steep pitch, whence it is continuously withdrawn to the mud box, *H*. This box is provided with means of adjusting the volume of the

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[96] Description supplied by manufacturers.

mud discharge so as to maintain the mud level in the clarifier at the most desirable operating height.

In the flocculating cell, spiral skimmers, *L*, constantly move any foam or scum towards the periphery until it comes into the path of the hinged hanging blades, *M*, which, revolving with the central tube, sweep it into the foam canal, *O* leading to the mud box, *H*.

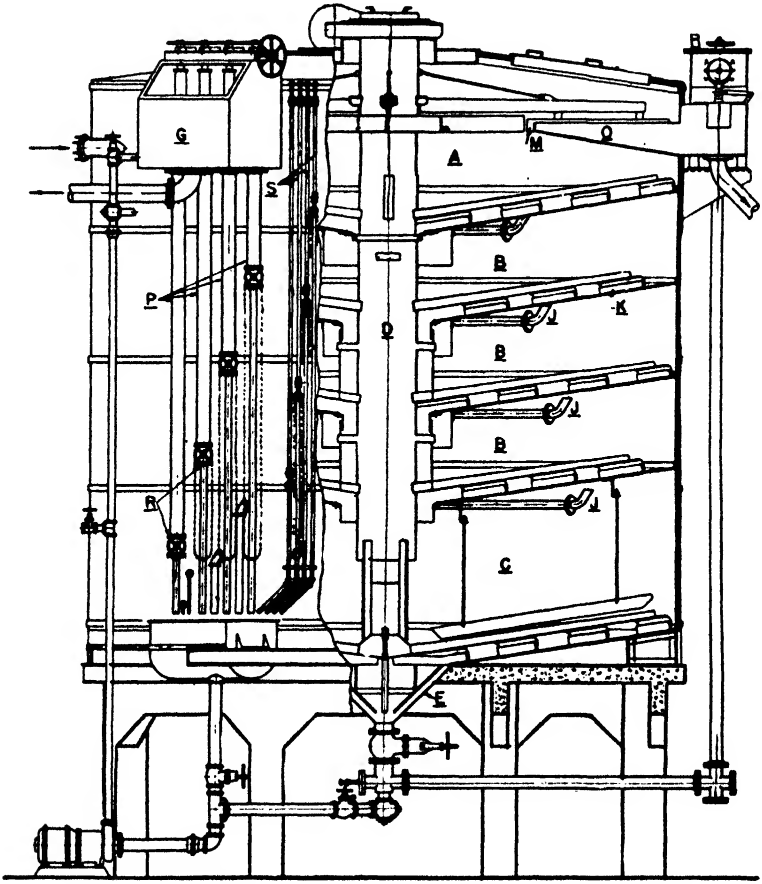

Fig. 35. Dorr Clarifier.

The juice, after liming and heating, passes through a flash tank (not shown), which is vented to the atmosphere, and then enters the flocculating cell through the side of the tank and just below the operating juice level, allowing time for the completion of the chemical reactions of defecation and formation of floc. The juice flows into the center tube, thence through openings into the several settling compartments where the sediment is continuously removed as described above. The clear juice is continuously drawn off from the top of each compartment through circumferentially located pipes, *J*, with multiple inlets formed by upturned nipples. These pipes are connected by risers, *P*, outside the clarifier tank with the overflow box, *G*, where the discharge from each compartment is controlled by vertically adjustable sleeves. From this overflow box the clean hot juice may be gravitated direct to the evaporator supply tank. For emptying the clarifier, valved drain pipes, *R*, are provided on each riser, also several decanting valves, all of which discharge into a small sump tank to which a liquidating pump is connected. Gas and air vents, *S*, are provided for each compartment.

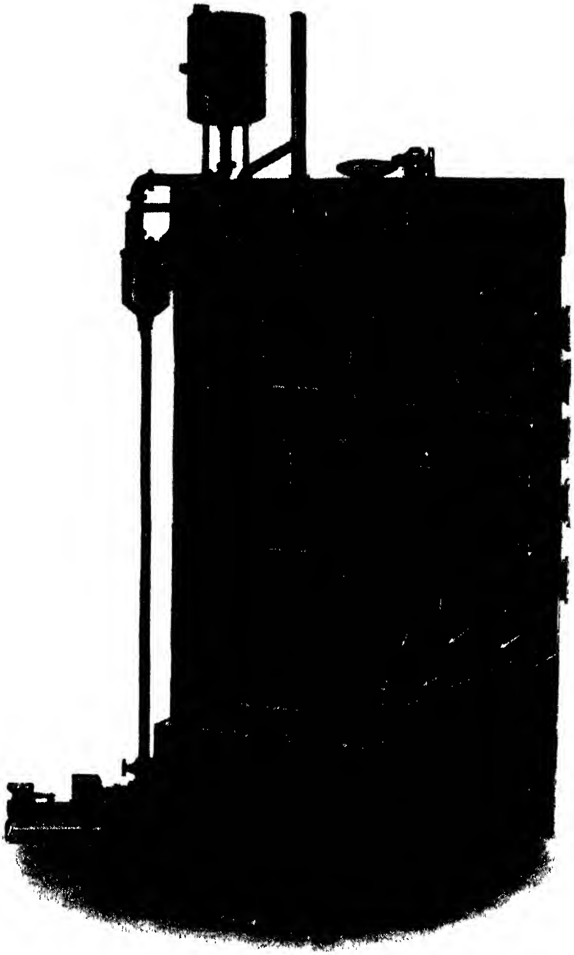

Fig. 36. Graver (Seip) Clarifier.

The clarifier is thoroughly insulated on the top and sides, the drop in temperature from hot feed intake to clear overflow being normally only about 2° C.

The percentage of muds withdrawn from the Dorr will vary with the kind and condition of cane, but will generally give less than half of the volume of muds from the old-style open settlers. One large Louisiana factory which weighed the muds reported 6.2 per cent on the weight of juice for the 1939 crop.

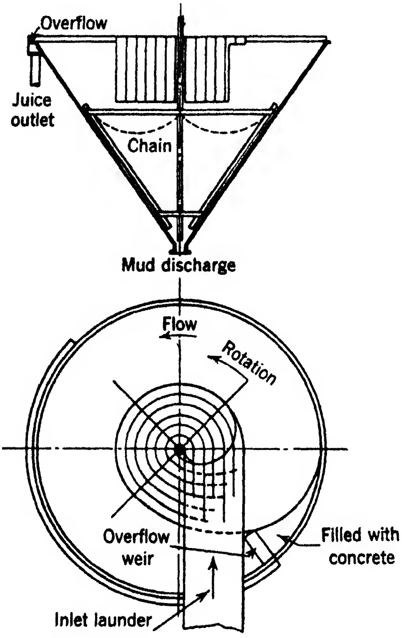

Fig. 37. Australian Subsider.

121. The Graver (Seip) Clarifier. This equipment is also of the circular tray type but there are several distinctive features for which advantages are claimed. The flow of juice and muds is around the periphery of the trays instead of through a central well, and the clear juice is collected at the center of the "umbrella" after having undergone what the manufacturers term "upward sludge filtration." The construction details will be seen from Fig. 36.

122. Fortier Clarifier. The clarifier operates without mechanical scrapers and differs in form from those already described.[97] The flow of juice through the settling compartments is horizontal and slow. The sludge settles onto inverted V-shaped inclined plates from which it falls by gravity into a thickening chamber provided with a large slowly revolving scroll which moves the sludge toward the outlet.

123. Queensland Conical Subsider.[98]

The standard settling equipment in Queensland is the continuous conical subsider. Its body takes the form of an inverted cone (Fig. 37), usually 18 ft. in diameter and 11 ft. deep, giving a working capacity of approximately 900 cu. ft. and a surface area of 250 sq. ft. The limed and heated mixed juice enters near the center and is guided by vertical baffle plates in a spiral path, the clarified juice leaving by an overflow weir at the circumference. The mud, assisted by a set of slowly rotating scrapers, collects in the lower portion of the subsider and is drawn off at the bottom, the rate of discharge being regulated by an ordinary cock or by a mechanically operated valve.

It will be seen that the action of the subsider as regards clarity of outgoing juice is a function of rate of flow and surface area, while the consistency of

[97] *Intern. Sugar J.*, December, 1940, p. 420.

[98] *Intern. Sugar J.*, June, 1940, p. 212.

mud discharged depends on the depth of the subsider and the thickening action of the scrapers.

DEFECATION IN WHITE SUGAR PROCESSES

124. Factory-Made White Sugars. A direct consumption sugar is one which is boiled directly from the concentrated cane juice without the intervening step of making a raw sugar to be redissolved or "remelted" for further purification or refining. Such factory-produced sugars range from the "turbinado" grade, which is a high-test washed raw sugar (generally dried with steam in the centrifugals) to "plantation granulated" which is an off-white or near-white product just below the quality of standard granulated as made by the boneblack refineries.

In order to produce such sugars it is necessary to resort to more elaborate processes than simple defecation. The use of SO_2 or CO_2 in conjunction with lime (the sulfitation and carbonation[99] processes respectively) are the oldest and best-known methods (in fact the only methods) which are in general use.

It is difficult to produce a sugar of uniform quality in the factory owing to the variable purity of the raw material. Obviously, any selection of the juices to give higher purities for white sugar manufacture will aid in maintaining uniformity of product. Sugars made by the carbonation process are of more uniform quality than those made by sulfitation, but the expense is much greater and for this reason carbonation is not employed to any great extent outside of Java and Formosa.

The light-colored canes seem to produce the best white sugars, but there is some difference of opinion on this point. Great attention to detail is essential to successful white sugar manufacture because the product is to be used for direct consumption. The same care and cleanliness must be exercised throughout the process that is observed in sugar refining by the more elaborate methods. Double purging of the sugars in the centrifugals (see Sec. 290) is practiced in Java to reduce the staining of the sugar through the imperfect removal of the molasses in the first purging.

SULFITATION PROCESSES

125. Sulfitation in Louisiana. This is a very old process and possibly originated in the English or French colonies. It is one of the simplest methods of using SO_2 and when carefully conducted produces a fairly good, irregular, near-white or off-white sugar.

The cold raw juice is pumped through a sulfur tower or box through a countercurrent of sulfur dioxide. The juice should absorb as much gas as possible (acidity 3.0 to 4.0 ml. of 0.1 N alkali for 10 ml. of juice). This sulfitation is followed by liming to slight acidity (1.5 to 2.0 ml.), and the juice

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[99] "Carbonation" is the term generally used in beet sugar practice in the United States and is therefore employed in this book, although it is recognized that "carbonatation" is scientifically the correct term.

is then heated, settled, and decanted, as is usual in the defecation process. The juice is sometimes reheated to boiling and brushed. Evaporation to a thin sirup follows, and the sirup is allowed to settle for six to twenty-four hours before being boiled to a massecuite.

A. G. Keller has pointed out that this process is on the decline in Louisiana, only about ten factories practicing it in 1939.[100] It is almost invariably used in conjunction with the production of direct consumption molasses. Only one boiling to grain is general, yielding a sugar of variable quality which must be heavily washed in the centrifugal. The molasses is allowed to settle for several weeks if it is to be sold, or it is diluted to 60° Brix and reboiled to yield an inferior raw or second sugar and "boil-back" molasses for the market. Yields naturally vary widely, but 100 lb. of white sugar per ton of cane and 10 to 12 gal. of first molasses are considered good.

Fort and Walton[101] recommend separate treatment of juices (see Sec. 111), using the higher test crusher and first mill juices for white sugar and the lower test last mill juices for raw. Heavy sulfuring (to a titrated acidity of 3.5 to 4.0 ml. 0.1 N NaOH) before liming gives a distinctly better quality white sugar than lighter sulfuring, according to these writers, and the final pH of the clarified juice should be 6.5 to 6.8. Another suggestion by them is the removal before liming of the non-sugars that have been flocculated by sulfuring; also they recommend reheating the multiple-effect sirup and removing the flocculated impurities by filtering or settling.

126. Sulfitation after Liming. In this process the lime is added before sulfitation in large excess over that required to neutralize the juice. Approximately 8 gal. of milk of lime of 26.5° Brix is used per 100 gal. of juice, thus producing with the sulfurous acid a very heavy precipitate which may be readily removed by settling and decantation. If a larger quantity of lime is used, e.g., 10 to 12 gal., the precipitate may be removed by filter-pressing. The sulfitation is continued to neutrality to phenolphthalein. The decanted or filter-pressed juice, after concentration to sirup, is usually cooled and sulfited to slight acidity. An acidity equivalent to that required in 10 ml. of sirup to neutralize 2.5 to 3.0 ml. of alkali 0.1 N is a suitable amount.

127. Harloff's Process. In the Harloff process, the juice is first heated to 75° C. and the lime and SO_2 are added simultaneously in such a way as to maintain the reaction acid to phenolphthalein and alkaline to litmus, except toward the end when a quantity of milk of lime is added to attain a strongly alkaline reaction, after which sulfitation is completed to neutrality to litmus. The limed and sulfited juice is finally heated to boiling in juice heaters and then is settled.

A process of the Harloff type, involving hot liming, fractional liming, and phosphoric acid is used in South Africa.[102] Double screening of juice is prac-

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[100] *Sugar Bull.*, March, 1940.

[101] *Sugar Bull.*, 1932, No. 6, p. 5.

[102] Latham, *Proc. 5th Congr. South African Sugar Tech. Assoc.*, 1931, p. 109.

ticed (see Sec. 59), Mitchell or Universal vibrating screens being general for the second screening. The screened juice is heated to 140° F. (60° C.), 90 per cent of the lime is added, after which it is sulfited to at least 3 mg. SO_2 per liter and run into connecting tanks with stirrers. The remainder of the lime, sufficient to bring the reaction to about 8.7 pH is added and then phosphoric acid to 7.4 to 7.6 pH, after which the juice is sent through juice heaters to bring the temperature to 212° F., and thence goes to settling tanks. Muds are heavily diluted and again settled, and then are filtered in plate-and-frame presses, the filtrate going to the hot raw juice. The sirup is not treated. The amount of chemicals per ton of sugar are: lime 73.5 lb., P_2O_5 4.5 lb., sulfur 28.5 lb. A similar process of phosphosulfitation is in use in Mauritius.[103]

Comparative experiments in South Africa as described by Dymond[104] showed that Harloff's process with phosphate added (the phosphosulfitation described above) gave the best clarification of any of several modifications of simple defecation and sulfitation. The efficiency was compared by measuring the removal of gums or alcogel, i.e., material precipitated by alcohol. In the phosphosulfitation process the removal was 65.8 per cent, whereas the simple defecation showed only 36.1 per cent removal. In these experiments liming *before* sulfiting always gave better results than sulfiting before liming.

128. Continuous Sulfitation. Continuous sulfitation as a substitute for the Harloff process was introduced in Egypt and is described by Neuville[105] as follows:

> Milk of lime at 15° Be is introduced right into the hot mixed juice pipe-line (juice at 75° C.) a few feet before it reaches the sulfitation tank, and in this line there are two L's to facilitate mixing. Liming is carried to an average of 8.6 pH, and the time of contact between juice and lime is about 8 secs. Sulfitation is carried out in a circular sheet-iron tank, about 9 ft. in diameter, in the bottom of which is a perforated plate, above which the limed juice enters, and below which the gas is admitted.
>
> It is essential to have two sets of furnaces delivering separately to the sulfitation tank, in order that the operation shall be continuous when re-charging.
>
> On leaving the sulfitation tank the juice is always between 7.0 and 7.4 pH, after which it is heated to boiling and settled. This continuous sulfitation process has worked to complete satisfaction. Lime has been reduced, and the sulfur consumption has fallen to 45 percent of that used previously with the Harloff process. Juices have been very clear and uniform; and the settlings have filtered well, giving cakes of good consistency and reduced volume.

129. Bach's Sulfitation Process. In this process lime is added and is precipitated by sulfurous acid both in the juice and in the sirup, each followed by subsidence and decantation or by filter-pressing.

From 5 to 7 gal. of milk of lime of 26.5° Brix are added to each 1000 gal. of cold raw juice. This is then sulfited to neutrality to phenolphthalein and

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[103] Haddon, *Intern. Sugar J.*, June, 1933, p. 244.

[104] *Intern. Sugar J.*, December, 1936, p. 480.

[105] *4th Congr. Intern. Soc. Sugar Cane Tech.* (Puerto Rico, 1932); *Intern. Sugar J.*, July, 1932, p. 281.

finally heated to full boiling, settled, and the clear juice decanted as in the ordinary defecation. The clear juice is evaporated to the customary density, approximately 55° Brix.

From 16 to 17 gal. of the milk of lime per 1000 gal. of sirup are now added and is then sulfited to neutrality to phenolphthalein. The sulfited sirup is heated to about 194° F. (90° C.) and is then filter-pressed. The filtered sirup contains a bisulfite of lime and is therefore heated to about 195° F. and run into settling tanks, and after the decomposition of the precipitates the clear liquor is decanted and cooled as previously, after which it is sulfited to distinct acid reaction to phenolphthalein and is then ready for the vacuum pan (pH 6.1).[106]

130. Control of Temperatures and Reactions. Bogtstra[107] compared the simultaneous method of adding the lime and SO_2 (as described above in the Harloff process) with pre-sulfuring, i.e., adding the SO_2 to a reaction 4.3 to 3.8 pH and then the lime to 8.0 pH, finally sulfiting to 7.0 pH, and found little difference in the character of the white sugars made. The deterring factor in the color of the sugar is the avoidance of glucose decomposition by regulating the temperature and reaction. It was observed that "from the same raw juice it is possible to make sulfitation sugars having 100 per cent differences in color, depending on whether temperature and alkalinity had been controlled." He found that a juice temperature above 70° C. was invariably detrimental to color, and also that the maximum reaction in the simultaneous method was 8.5 to 9.0 pH, this point being reached soon after the commencement of sulfuring (not at the end as originally recommended by Harloff) and maintained throughout. A high alkalinity peak, especially when combined with a high temperature, was always fatal to good colored sugars. He concludes that the key to the production of good sulfitation sugars is thorough mixing of lime and juice to avoid local overliming, and the maintenance of temperatures below 70° C.

131. Sulfur Stoves or Ovens and Sulfitors. The type of stove shown in Fig. 38 is used quite generally in Louisiana. The body of the stove is water-jacketed for cooling. A large pipe, usually 6 to 8 in. in diameter, also water-jacketed, leads to the washing and cooling box and thence to the gas main. This serves as a subliming chamber and catches a large part of the sublimed sulfur. Suitable valves are provided for disconnecting the stove from the main pipe line so that a stove may be cleaned without interfering with the others in the battery. Air for the combustion of the sulfur should be drawn through a drying box and then forced into the stove by a compressor or drawn through by an ejector.

The drier is preferably a cast-iron box provided with a tight-fitting door and trays for holding layers of quicklime. The air enters the box at the bottom and passes over the lime on its way to the compressor.

[106] Farnell, *Intern. Sugar J.*, **27**, 141 (1924).

[107] *Intern. Sugar J.*, October, 1934, p. 401.

A rotary sulfur burner, made by the Glens Falls Machine Works (Fig. 39) is in use in beet factories and the more modern cane installations which use sulfur. This burner uses induced draft. The sulfur is fed to the rotary burner by a mechanical feed (not shown in the cut) and melts by its own heat of combustion in the rotating cylinder presenting a large surface for combustion

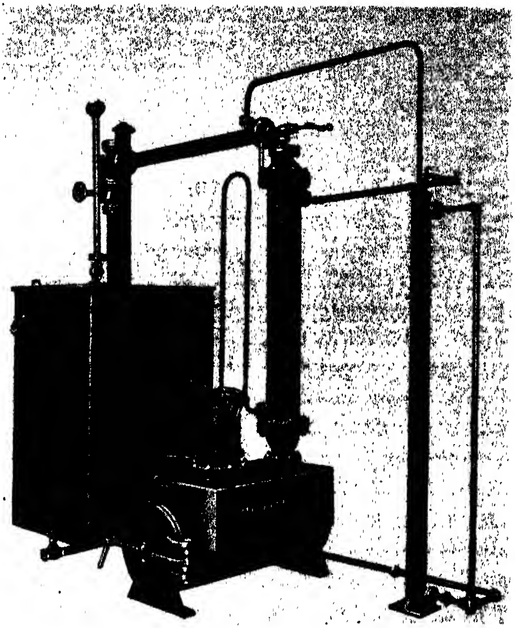
Squier Mfg. Co.

FIG. 38. Sulfur Burner.

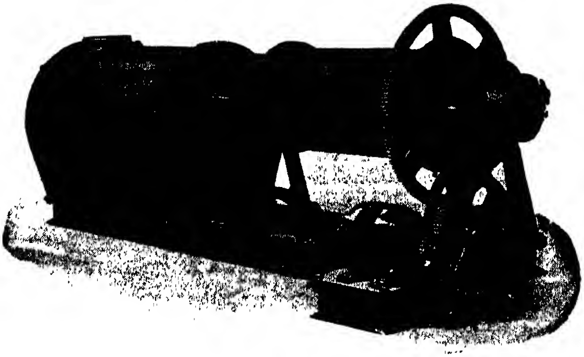

FIG. 39. Rotary Sulfur Burner.

as the sulfur drips through the air. Air is drawn in at an adjustable neck ring and anti-sublimation sleeve at the connection between the rotating drum and the combustion chamber shown at the left. This is a cast-iron or brick-lined compartment with baffles in which the oxidation of the sulfur and mixing with the diluting air are completed. A uniform gas (5 per cent to 16 per cent SO_2 as desired) free of sulfuric acid is delivered to the sulfitors.

132. Sulfitation Tanks. Sulfitation tanks are of many forms, but the simplest one is a moderately deep iron or steel tank provided with a cover and chimney to the outer air, suitable test cocks, valves, and a perforated pipe for distributing the sulfur dioxide. The pipes and tanks should be arranged to facilitate cleaning at frequent intervals. The bottom is preferably conical. Perforated pipes for steam jets to break down foam should be provided in sirup sulfitors. A layout for sulfuring sugar juices recommended by the manufacturers of the rotary sulfur burner described above includes the burner connected to a bank of cooling pipes made of 6 in. cast-iron, lead-lined pipe over which a water spray falls. The cooled gases pass into the bottom of a sulfuring tower consisting of a vertical cylindrical sulfiting tank about 4 ft. in diameter by 15 ft. high, the upper two-thirds of which is filled with grids made of hardwood 2 in. by 4 in. on edge, alternate grids being placed at right angles.

The juice enters the top of the tower above a perforated steel plate, falls in a spray through the wooden grill work where it encounters the rising current of SO_2. The flow of gas through the system is induced by an air ejector which is located in the gas outlet pipe of the tower and which is supplied with air by a small blower. The sulfured juices are drawn from the conical bottom of the sulfitor.

To saturate large quantities of lime, as in Bach's process, intermittent work is customary, and at least three tanks are generally installed, although two tanks may be used in this method. The juice enters the first tank at the bottom and is sulfited to approximately the desired test. It overflows and enters the second tank at the bottom and is sulfited to the required acidity. From the second tank the juice flows through juice heaters and filters to the charge tanks of the evaporator.

133. Plantation White Sugar with Vegetable Carbons. The literature on this subject is voluminous and is largely made up of claims and descriptions of processes by carbon manufacturers and inventors. However, the manufacture of direct consumption sugars with vegetable carbons has not gained especial favor, and no appreciable amount of white sugar is made in this way. The reason for this is that the decolorization of cane juice with vegetable carbons is not economical, no matter how well the juice is defecated or clarified, as the cost of the process is high and the results do not equal sugars made from melted raws. The refining of melted raw sugars by vegetable carbons (see p. 327) is an established process and is described in detail in the chapter on Refining.

THE CARBONATION PROCESS

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134. History. The purification of cane juices by an excess of lime and by removing the surplus by carbonic acid was introduced in the cane sugar industry in Java around 1880. The original method was the same as adopted in the beet sugar industry: to the cold or heated juice, quicklime was added and slaked. The limed juices were brought to a temperature between 80° and 90° C., were carbonated until a slight alkalinity remained, and then were filtered.

The amount of lime used was between 4 and 5 per cent on juice, and the result of this carbonation was a clear juice with a high amount of soluble lime salts and a dark color. This made it possible after the second carbonation to obtain a white sugar that could be used as direct consumption sugar. The equipment used was the same as applied in the continental beet sugar industry. Alterations in the process were proposed and soon introduced. The most important were: (1) mixing the raw juice to be treated with milk of lime instead of slaking the lime in the juice itself; (2) a carbonation process at lower temperature, which made it possible to obtain a light-colored juice.

A great step forward was made in 1910 by the introduction of the carbonation according to de Haan. He formulated the conditions for the carbonation of raw cane juice as follows.

First, the carbonation of cane juice, consisting of the addition of milk of lime and carbonating the excess of lime, should be held to a temperature of 55° C. At higher temperatures reducing sugars are decomposed and dark-colored reaction products are formed with the result that a sugar of poor color is obtained.

Second, for a good filtration it is necessary that the milk of lime be added at just such a speed that the alkalinity never exceeds 800 mg. CaO per liter. To accomplish this the speed of addition of milk of lime has to be regulated by a continuous control on the alkalinity according to the lime precipitated by the carbonic acid.

In a very short time most of the Java factories adopted de Haan's method, and for many years it was the standard practice. The amount of lime used per ton of cane or per ton of sugar produced was reduced considerably, the purity of the thin juice was increased, and the quality of the manufactured white sugar improved.

The maximum amount of sugar produced annually in Java by the carbonation process was 1,200,000 tons. Outside Java, carbonation was practiced only on a limited scale in the cane sugar industry. It existed in Formosa, where about 20 per cent of the total sugar production was made with the carbonation process; in South Africa (Mount Edgecombe); Puerto Rico

(Guanica); on an experimental scale in one of the factories in Hawaii; and on a limited scale in Argentina.

The maximum total amount of sugar produced during one year in the past was 1,700,000 tons. During the last five years only 7 to 8 per cent of the total world production of cane sugar was made with the carbonation process.

135. Latest Developments in the Carbonation Process. After the introduction of control in the industry a better insight in the factors determining the results of the carbonation process could be collected in combination with an exact measurement of the temperature, rate of flow, effect of mixing, etc. For a good filtration, as the first step, it has been found necessary to add lime to a rather high alkalinity, pH 10.5 to 11. This reaction must be kept for a certain time, for instance five minutes, during which period a precipitate of phosphate of lime is formed and well coagulated. In the second stage of the carbonation this precipitate acts as a nucleus, by which a sedimenting, good filtering, coarse precipitate is formed.

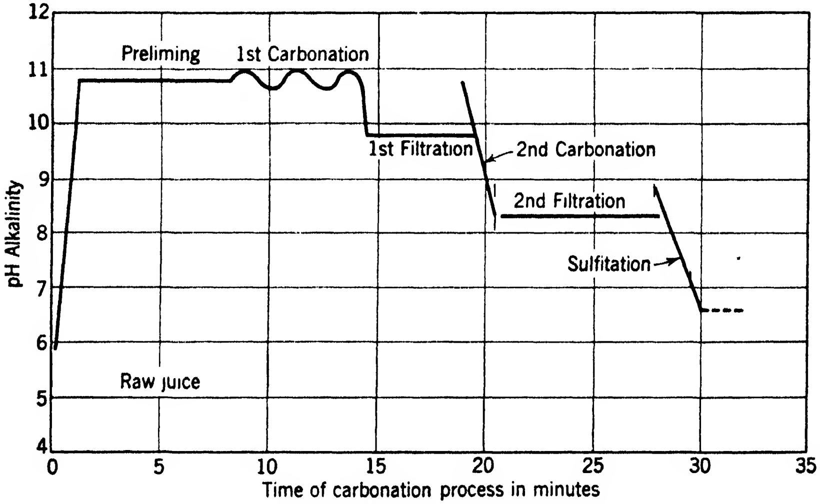

Fig. 40. Simple Carbonation Process.

The scheme of the carbonation process is given in the chart, Fig. 40.

The second precaution is to prevent too high a pH during the first carbonation since otherwise complex compounds between CaO, $CaCO_3$, and sucrose may be formed, which decompose slowly and increase the losses of sucrose in the discarded filter mud.

136. The Practice of the Carbonation Process. The standard system for the carbonation process is the discontinuous process, although the continuous process has been introduced with good success in a number of Java factories. The technological details of the process have been worked out and established.

a. Size of the carbonation tanks for the first carbonation is usually 60 to 70 hectoliters per hundred tons of cane per twenty-four hours with the average time for the carbonation per batch eight to twelve minutes. Minimum height for the juice to be carbonated is 2 to 2½ m. to obtain a sufficient gas absorption. Density of the milk of lime is 15° to 20° Baumé.

b. The second carbonation reduces as much as possible the excess of lime remaining in the filtered first carbonation juice. This means that 100 to 250 mg. CaO per liter of juice has to be precipitated and this is mostly a continuous process. The size of the second carbonation tank is 5 to 7 hectoliters per 100 ton of cane per twenty-four hours.

It is a customary practice to heat the filtered first carbonation juice to a temperature of about 70° C. The slightly alkaline juices are susceptible to fermentation at a temperature of 55° C., especially the development of lactic acid-forming microorganisms. Secondly, at the higher temperature the precipitation of the lime is more complete and in a coarser form and has better filtering properties.

c. The filtration of the carbonation juices is usually done in plate-and-frame presses. The maximum pressure is 3 kg. per sq. cm. Reciprocating plunger pumps as well as rotary pumps are used.

A number of factories have introduced Sweetland presses with success. In exceptional cases Vallez presses have been used. On an experimental scale, for the first carbonation, rotating vacuum filters have been applied.

The filtering surface required for the first carbonation juice in pressure filters is 20 to 25 sq. m. per 100 tons of cane per twenty-four hours. For the second carbonation, a surface of 5 to 8 sq. m. is sufficient.

137. Other Forms of Carbonation. The number of variations in the carbonation process is very great. The process is used only for white sugar manufacture as it gives a very pure juice, making it possible to produce a white cane sugar comparable in quality with white beet sugar and superior to such other forms of cane juice purification as sulfitation and phosphatation. The economy of the process is based upon the higher value of the white sugar compared with the price of raw sugar. Besides that, by the greater removal of non-sugars a slightly higher yield may be obtained. Based on the experience of many years, it is estimated that the increased yield of recoverable sugar by the carbonation process is 1½ to 2½ per cent.

The variations in the carbonation process are:

a. Using standard defecation, settling and removing of the muds, and carbonating the clear juices. By this process it is not possible to attain the same purity of thin juice obtained with the direct carbonation of the raw juice. With the present knowledge of the carbonation process, the following purities of the purified juice may be obtained:

MAXIMUM AMOUNT OF IMPURITIES PER LITER OF THIN JUICE

Lime (CaO)	Less than 70 mg.
Phosphate (P_2O_5)	Less than 10 mg.
Magnesia (MgO)	Less than 20 mg.
Silicic Acid (SiO_2)	Less than 20 mg.
Sulfate (SO_4)	Less than 300 mg.

It is a remarkable fact that by carbonating defecated clear juice the purity is always less than that of a direct straightforward carbonated raw juice.

b. The formation of the precipitate in two successive steps with double filtration and the return of the precipitate of the last carbonation to the first stage of the carbonation process. By this complication no decided advantages in the purity of the juice are obtained. This process becomes only practically workable with automatic and rotary filters as the amount of labor in the filter station is increased.

c. An improvement introduced in one of the largest mills in Java carbonates the partly concentrated juice (instead of the clear juice) at a Brix between 40° and 45°. This is the so-called middle juice carbonation. By the concentration process, the removal of the non-sugars per unit weight of Brix is increased and with a smaller amount of lime a purified juice may be obtained that compares favorably with the juice obtained by the standard practice.

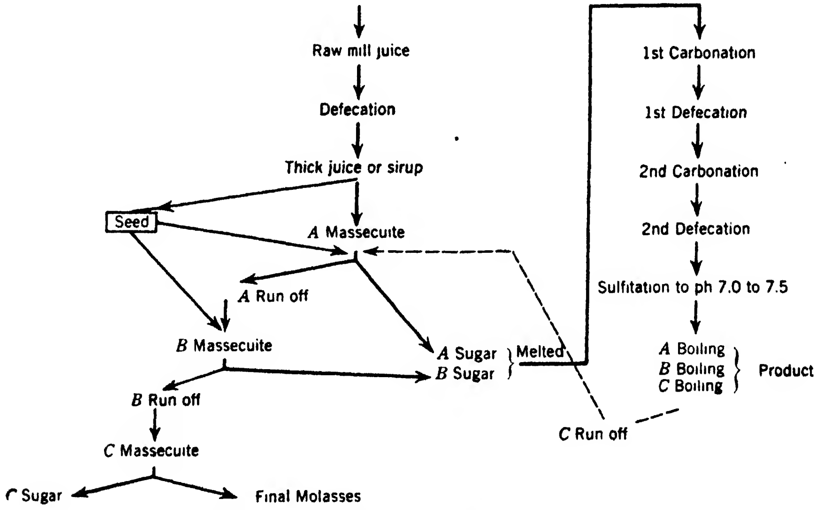

FIG. 41. Carbonation with Melted Sugars.

d. The carbonation of remelted sugars. This practice has been adopted by a great number of European cane sugar refineries and a number of Java factories as the method of pre-purification before the bonechar treatment. In a cane sugar factory, it is possible by making a superior grade of washed raw sugar to get a solution of high purity of remelted sugar. By treatment of this solution with lime and carbonic acid and an adjustment of the final pH to 7.0, light liquors result without any further treatment with bonechar or vegetable carbons from which a superior grade of granulated sugar equal in quality to standard refined sugar may be made. The advantage of the last-mentioned method is that a cane sugar factory is able to deliver its products, either raw and refined, in a ratio adjusted to the requirements of the market. The scheme for this process is given in Fig. 41.

138. Amounts of Lime Required. It is customary for carbonation factories to burn the limestone for the required lime themselves. The burning is done by mixing limestone with coke in vertical limekilns. The ratio of limestone to coke is from 10 : 1 to 15 : 1, according to the size and shape of the kiln and the quality of the limestone.

The carbonic acid formed by the burning of the coke and the dissociation of the limestone is removed with pumps and washed with water to remove dust and traces of SO_2 which are present from the sulfur in the coke. The purified CO_2 is used for carbonating the limed juices. The CO_2 content of the gases of the limekiln varies from 32 to 38 per cent. The rest is nitrogen with 1 to 3 per cent oxygen free of CO. The burned lime is slaked with water, screened to remove the coarse unslaked particles, and used as a cold milk of lime in a density of 15° to 20° Baumé. The amount of limestone for carbonating raw juice is 25 to 30 kg. per ton of cane.

For the carbonation of middle juice with the same purification effect as with the de Haan carbonation of raw juice, 15 kg. of limestone per ton of cane is sufficient. For the manufacture of white sugar by carbonating remelted sugars, 7 to 8 kg. of limestone per ton of cane may be considered to be the maximum for superior white sugar.

The trend of the carbonation process in the cane sugar industry is in the direction of the carbonation of remelted sugars. It is one of the cheapest, cleanest, and most reliable processes in the cane sugar industry, insuring standard quality of the sugar.

CHAPTER 7

TREATMENT OF SCUMS AND CLARIFIED JUICE

139. General Practice. The clarification, by the ordinary means, has divided the whole juice into two portions: (1) the clarified juice, and (2) the precipitated settlings known as scums or muds. The clarified juice which constitutes the greater portion (80 to 90 per cent) generally goes directly to the evaporators without further treatment, although filtration is sometimes practiced, as will be shown later. The mud waters, after various methods of treatment, are generally filtered. In the Petree process (Sec. 112) the mud waters are sprayed on the bagasse on the mills, the mud remaining on the bagasse and the thin juice going into the main juice stream.

TREATMENT OF SCUMS AND MUDS

140. Diluting, Settling, and Filtering. The scums and juice tank bottoms as they come from the clarification system are generally filtered through filter presses or in modern practice through vacuum filters. Preparatory to filtration the more general practice is to heat these scums and mud waters with steam in "blow-ups" ("cachaceras" in Cuba), settle the mud, and decant the clear juice. The residual settled mud is again diluted, heated, and settled, and the thin juice again decanted. A further dilution before settling reduces the amount of sugar in the mud and facilitates filtration, since a mud water high in sugar content does not filter as readily as a thinner solution.

141. Adjusting the Reaction. The mud waters filter-press more readily when alkaline; therefore, lime is usually added to them. Great care should be taken to avoid an excess of lime for the same reasons that are given as objections to overliming the clarified juice, and a pH of 8.0–8.5 would seem to be the limit for limed mud waters. Some consider it better practice wherever possible to omit all additional lime at this station, filtering the press juices at the same reaction as the clarified juice.

Many recommendations have been made for high liming of muds, however. DeGoede[1] cites a factory in Java which limed the muds to pH 10, returning the press juice to the cold unlimed juice. Extra lime was used, and the danger of glucose decomposition was recognized, but the advantages of better filtration and clearer juice were thought to outweigh these. Honig and Thompson examined the addition of lime in various Java factories[2] and

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[1] *Intern. Sugar J.*, April, 1933, p. 154 (abs.).

[2] *Intern. Sugar J.*, August, 1933, p. 313 (abs.).

found the remarkable result that liming scums to a pH lower than 10.5 actually had a detrimental effect, and that pH 11.0 is needed to secure marked improvement, but this was not recommended because of the decomposition induced and the large increase in dissolved lime salts. Sulfitation after liming the scums was found to facilitate filtration greatly, and carbonation with flue gases was also efficacious. In this same study the use of bagasse ash (sifted, washed, and passed through a fine screen) to the amount of 1 per cent on scums, increased filtration rates 78 per cent and 2 per cent addition gave 107 per cent improvement. Disadvantages noted are that it appears to cause cloth splitting and wear on pumps, and to induce the resolution of undesirable non-sugars in the juice. These authors studied the addition of 0.2–0.25 per cent of fine bagasse and found an increase in filtration rate from 17 to 25 per cent, but they objected to the increased Tyndall effect in the filtrate.

The addition of fine bagasse (bagacillo) as is done with the Oliver-Campbell filter described later has been fairly general practice in those factories with double screening systems (see Sec. 59), and reasons have already been given for avoiding strong alkaline reactions in the presence of bagasse fiber. The use of kieselguhr or diatomaceous earth (see Sec. 162) is efficacious in the formation of a press cake, but the added expense has precluded general adoption.

J. V. Hayden[3] described a process for the treatment of muds (in use in Australia where it is called Killer process) which involves the continuous dilution of the muds as they come from the clarifiers and the discarding of concentrated settlings without filtering. The mud in the juice subsider is drawn continuously into a mixer while a constant stream of hot water is added together with the necessary lime. The mixture is passed through a juice heater and into another subsider the overflow of which goes to the bagasse as maceration, while the mud which gravitates in a heavy thick stream is discarded. The sugar content of the mud is from 0.6 to 1.2 according to the amount of maceration water used. (The sugar loss per cent cane would appear to be much higher in such a process than if the muds were filter-pressed before discarding.)

King and Villareal[4] recommended mixing last mill juice with mud waters and then liming to 10 pH and resettling this mud and juice. The resettled juice would be used for tempering the mixed juice. To avoid (or reduce) glucose decomposition heating to 90° C. was recommended, but it would seem from King's own previous publications that such highly alkaline material even at this temperature would cause heavy decomposition as well as resolution of much of the precipitates.

142. Filter Presses. The filter press is probably the oldest specialized piece of apparatus in use in the sugar industry. Its invention is credited to Howard and dates over a century and a quarter ago. It still remains the

[3] *Intern. Sugar J.*, December, 1931, p. 540.

[4] *Sugar News*, 1933, No. 11, p. 533; *Intern. Sugar J.*, March, 1934, p. 126 (abs.).

means of filtration for muds in the majority of the sugar factories of the world. Briefly, a filter press consists of a number of iron plates and frames or recessed plates over which filtering cloths are placed. The frames and plates are supported on a heavy framework and are clamped together by a powerful jack screw. The cloth itself makes the joints between the frames and plates. There are two general types of presses, the center-feed with recessed plates and side-feed plate-and-frame presses.

The center-feed presses are made up of heavy recessed plates with a round opening in each and a corresponding hole in the cloth to form the inlet channel. The cloth is clamped to the plate at each of these openings.

There is no hole in the cloth in the plate-and-frame press. A lug projects from each frame and plate, and in each is an opening to form the mud channel. Rubber rings or cloth "stockings" form the joints between the lugs. The plate-and-frame type of press is generally used in raw sugar factories because of the greater space for the cake. Center-feed presses are in use in many refineries.

When a press is in use the mud collects in the frames or recessed plates and the filtered juice runs off through suitable openings.

A pressure of 40 lb. to the square inch is sufficient for very good press work. With heavier pressure there is risk of breaking the presses and liability of forming a coating of almost impervious material upon the cloths.

143. Washing the Cake. Filter presses are usually arranged so that the press cake may be washed or the juice it contains be displaced with water. In displacing the juice the cocks are closed on the water plates, and water is forced through the cake, driving the juice ahead of it. This is feasible only in defecation factories having very large filter press capacity, as the displacement is exceedingly slow. This method is applicable in the carbonation and certain of the sulfitation processes. In order to economize wash water and consequently fuel for evaporating, the last runnings of a previous press are often used in beginning the washing of the cake. In raw sugar manufacture the more general practice is to filter diluted muds after resettling and to discard the cake without washing.

Galbis and Bach devised a scheme for washing presses in which the supply of scum juice is shut off from the press before the frames have completely filled with press cake so that an open space is left between the two inner surfaces. The current of wash water is run through the same channels as the juice, and this open space permits free access of the water to all parts of the cloths. This scheme is only applicable where a firm, hard cake is found. Hot water is used in sweetening off at the same pressure as the juice when the filtration was stopped.

Geerligs [5] says that in practical working of this scheme the sweetening off is stopped when the effluent reaches a density of 2° to 3° Brix, the remaining liquid in the cake being expelled by compressed air or steam. He reports an

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[5] *Cane Sugar Manufacture,* second edition, 1924, p. 180.

average filtration time of one and one-half to two hours and one-half hour for sweetening off and steaming, the sucrose content of the cake being reduced to less than 1 per cent. Experience in Cuba has shown that washed cake is extremely variable in composition when sampled in different parts of the frames and general practice is to dilute muds and not to wash.

144. Double Filter-Pressing. Double filter-pressing is a method of exhausting the press cake of sugar in the defecation and other processes but with certain marked disadvantages as mentioned below. The first pressing is conducted as usual. The first press cake is conveyed to a machine for breaking it, and then it is beaten to a cream with water. This cream is next heated to boiling and is then pumped into a second set of filter presses. The filtrate from these presses may be used in diluting mud (cachaza) preparatory to the first pressing. This method requires a filter-press plant of sufficient capacity to devote about one-third of the presses to the second filtration. The sucrose content of the press cake may readily be reduced below ½ per cent without excessive use of water.

Many factories which adopted double pressing have discontinued it because of the conviction that the gain in sucrose was not sufficient to offset the redissolved impurities put back into the process. Allen[6] published results to show that no such adverse conditions could be ascribed to double pressing, but Keane, McCalip, and Paine[7] in a similar study concluded "that it seems debatable whether the sugar recovered is of sufficient value to offset the effect of the harmful impurities which enter the process in this operation." The factories which discontinued double pressing have done so largely on their own experience of washing out excess impurities for return to the process, rather than on the findings of others.

The practice of double filter-pressing was again advocated in Java by Stok[8] for press cake from POJ 2878 because the high wax content made sweetening off difficult by the ordinary method. Sugar losses were reduced to one-third.

145. Oliver-Campbell Cachaza Filter. A development of the past ten years is the Oliver-Campbell rotary vacuum filter for mud waters. This is a vacuum filter of the rotary type especially adapted for use in the cane sugar industry. The filter consists of a rotating drum, covered with perforated copper plate, which dips into a bath containing the material to be filtered. As the drum rotates, suction is applied to the different segments and a thin cake is formed on the filtering surface. The cake so formed passes under washing sprays and is then dried by suction, and the cake is removed by scrapers. (See Fig. 42.)

Settlings are drawn from the clarifiers and pumped to the feed mixer, *A*, where bagacillo is added. From the mixer the settlings are fed to the filter. A constant level is maintained in the filter tank by an overflow to an over-

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[6] *The Planter,* 71, 214 (1923).

[7] *Facts About Sugar,* Oct. 8, 1927.

[8] *Intern. Sugar J.,* March, 1930, p. 139; *Archief,* 1929, No. 53, 1102.

flow tank, *B*, on the floor below from which settlings are pumped back to the mixer.

The vacuum pump, *C*, maintains constant vacuum in the receivers, *D*, which are connected to the filter valve. One of these receivers is for cloudy filtrate; the other for clear filtrate. Because both of these filtrates are hot and when under vacuum likely to flash into steam and carry off vapors, provision should be made for a condenser, *E*, and moisture trap, *F*. Here the vapors are condensed and run back to a seal pit. The filter valve referred to above has a double port. As each section of the filter drum starts rotating through the tank containing the settlings, the vacuum is automatically applied to the first port drawing juice through the filter medium. This first filtrate is cloudy, but the cake which quickly forms prevents passage of additional solid material so that the filtrate becomes clear. At this point the flow automatically shifts to the second port. Small pumps, *G*, remove the cloudy and clear filtrates, the former returning to the mixer if thick settlings are being handled, or to the juice heater supply tank for redefecation if the settlings are thin. Clear filtrate goes to the evaporator supply tank or to the juice heater supply tank, as desired. In actual practice the filtrate is not generally clear enough to send to the evaporators, and it is returned to the unclarified juice for redefecation. This is better practice in any case, as will be explained later.

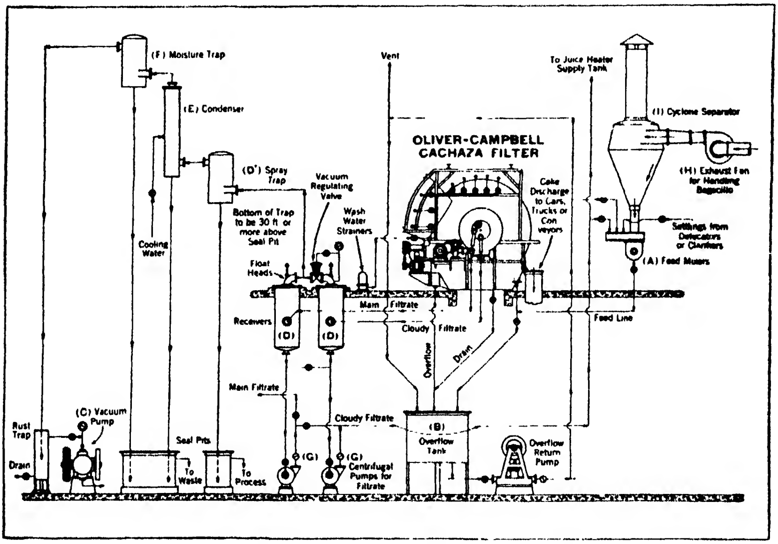

Fig. 42. Oliver-Campbell Filter and Accessories.

Under average conditions the polarization of the filter cake can be reduced to between 0.2 and 1.0 per cent with but little dilution of filtrate.

Generally a small amount of bagacillo is added to the settlings before they are fed to the filter. The bagacillo recovery unit consists of a shaking screen or air separator or other means for recovering the fines from the bagasse. Also needed are an exhaust fan, *H,* and suitable ducts for conveying the bagacillo to a cyclone separator, *I,* from which it drops into the mixer.

The quantity of insoluble matter in settlings, expressed in per cent of weight of cane, may vary from 0.4 to 0.9 per cent. It usually averages slightly over 0.6 per cent.

146. Leaf Filters. Kelly, Sweetland, and Vallez filters, similar in general form to those used in refining (Sec. 373), have found some use in the raw sugar industry though not to any considerable extent.

147. Quantity of Press Cake. The proportion of filter-press cake varies greatly with the locality, efficiency of milling, and juice straining, and press efficiency. The quantity of lime used in the clarification has also an effect on the proportion—the more lime, the more press cake. The addition of earth from flooded canes also modifies the proportion.

The amount of cake per ton of cane gradually increases as the season advances and is at its maximum at the end of the grinding. Washing the cake reduces the weight not only through the removal of sugar but also by the washing out of soluble impurities. Double filter-pressing produces less cake for the same reason. Too short a press cycle, resulting in cakes with high moisture percentage, is a frequent cause of excessive quantity and attendant loss of sugar. An example of this is a factory that usually produced a crop average of 35 to 40 lb. of cake per ton of cane which reduced this number to about 31 lb. by lengthening the time under pressure. The cake per ton of cane ranges from 15 to 40 lb. in Cuba, with efficient factory work. Allen[9] gives the following figures for single and double pressing covering six years of each class of work in a Puerto Rican factory.

	Per Cent Sugar	Per Cent Moisture	Pounds Cake per Ton of Cane	Square Feet Filtering Area per 1000 Tons Cane
Single pressing	3.925	55.15	25.25	2206
Double pressing	1.233	59.03	27.40	3156

Note: The greater weight of cake with double pressing is accounted for in this instance by the higher moisture content. Similar figures for a large Cuban factory showed:

	Per Cent Sugar	Pounds Cake per Ton of Cane	Purity of Press Juice
Single pressing	5.08	33.60	81.2
Double pressing	1.90	28.00	72.5

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[9] *The Planter,* **71, 250 (1923)**

The figures for Java as given by Stok[10] were:

	Pol Cake	Sugar Lost Per Cent Dry Matter	Pounds Press Cake per Ton Cane
Single pressing	4.51	10	45
Double pressing	1.35	3	32

148. Disposal of Filter Press Juice. The filtered scum juice from the presses may be disposed of in one of several ways: (1) by clarifying and resettling separately and then mixing with the clarified juice; (2) by returning to the defecators for redefecation and resettling in conjunction with the main stream of raw juice; and (3) by pumping it direct to the charge tanks of the evaporators. The use of decanted mud waters for saturation has also been practiced.

The first method (separate clarification) is rarely resorted to, and the third method of mixing the material with the defecated juice is objectionable if excess lime has been added to the scums before filter pressing. Under these circumstances the more alkaline press juice may set up a slight precipitate in the defecated juice and this precipitate, in combination with the excess soluble lime, increases the scaling of evaporator tubes. The second method of sending the press water back through the clarification system is general practice and was considered to offer no objections whatever since it was believed that any excess lime would react with the undefecated juice and be an aid to clarification.

In the work already cited, Keane, McCalip, and Paine[11] have shown that this action does not take place and that 80 per cent of the excess lime in the filtered press juice finds its way into the evaporator even though it is mixed with the raw juice and redefecated. They also showed that all the reversible colloids formed by the action of lime (see Sec. 106) pass through the redefecation untouched and go to the evaporators exactly the same as if the press juice had been sent direct to the evaporator instead of passing the second time through the defecation system.

This work indicates the necessity of a careful control of the liming of the scum waters and the avoidance of the addition of any lime at this station if a good cake can be secured without it. In Hawaii, with more alkaline clarification, it has been found that no lime is needed at the press station, and the press water is sent directly to the evaporators according to the third method given above. It is probable that returning the press juice to the undefecated juice for retreatment (method 2) is to be preferred, even with correct lime control, in order to avoid the possibility of muddy press juice (caused by some failure at the presses) being added to the clarified juice going to the multiple effects.

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[10] *Loc. cit.*

[11] *Ind. Eng. Chem.*, **20**, 373 (1928).

Furthermore, it is impossible to have such correct control of pH and lime content of the two juices, clarified and press juice, that no after-precipitation will occur at any time.

With the Oliver-Campbell filter described above it is general practice to return the filtered mud waters to the clarification system as they are not clear enough to add to the clarified juice.

TREATMENT OF CLARIFIED JUICE

149. Juice Direct to Evaporators. In practically all raw sugar manufacture, the clarified juice decanted from the defecators is sent directly to the multiple-effect evaporators without further treatment. If the clarification has been defective, no amount of screening will greatly improve the juice nor will any further form of filtration give satisfactory results. Cloth filtration, unless filter aids are added, is practically impossible as the fine flocculent material which has passed through the defecation process soon clogs the pores of the cloth.

It is now recognized that the sooner the juice is brought to the sirup stage the better, and that clear juice is to be obtained by correct clarification rather than by an after-treatment. This should be understood in reading the descriptions of the screens and filters which follow.

Filtration of the entire juice after sulfitation prior to liming is general practice in the Argentine, but according to Barkow[12] compound clarification is displacing this procedure.

150. Mechanical Screening. A mechanical screening through fine wire cloth is effective in removing floating particles of bagacillo and other suspended matter which the defecating precipitate has failed to enmesh, but even this is not generally resorted to. A 100 by 100 metal cloth is employed frequently, but there are difficulties in keeping the screen from stopping up. Geerligs[13] describes a slanting bronze gauze screen containing 2000 meshes per square centimeter (about 13,000 per square inch) as well as a rotating screen similar to the Peck strainer (Sec. 59). The Mitchell electric vibrating screen has come into use for this purpose at some Cuban factories.

A device patented by Hadfield in Hawaii[14] consists of a screened compartment inside a square shallow tank, the screening surface being 150 by 150 Monel metal cloth. The clarified juice flows into the tank outside the screened compartment and then finds its way through the fine screen. This would very quickly block up were it not for a spray of strained juice which is gently played on the outflow side of the screen. The idea is not to dislodge the particles of bagacillo, sediment, dirt, etc., which are caught on the screen, as this is taken advantage of as a filtering medium. The spray is the principal part of the system.

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[12] *Facts About Sugar,* 1938, No. 6, p. 43.

[13] *Cane Sugar Manufacture,* second edition, London, 1924.

[14] W. G. Hall, *The Planter,* **72, 290** (1926).

A factory running 50 tons of raw juice per hour is said to require three tanks containing 24 sq. ft. of screen surface each. Cleaning and washing takes but a few minutes, the frequency depending on the cleanliness of the juice.

151. Mechanical Cloth Filters. The use of bag filters such as are used in refining (Sec. 372) is rarely resorted to in the defecation processes. Occasionally in white sugar work use is made of this form of filter (Sec. 125) but only when some other reagent is used in addition to lime.

Mechanical cloth filters of the Danek type are also rarely used. They consist of a large number of rectangular bags suspended in a closed iron box. Each bag or pocket has an independent discharge pipe communicating with the inside of it. A metal distender prevents the bags from collapsing. The clarified juice flows into the filter by gravity, under a low head, fills the box, and filters into the bags. The mud collects on the outside of the bags and from time to time falls off. The pressure is so low that the mud is not impacted upon the cloth. When the cloths become foul, the flow of juice is shut off and the mud is sent to the filter presses.

152. Sand Filters. Several types of sand filters for juice and sirup have been tried. As the name of the filter implies, the filtering medium is fine, sharp sand. Pulverized coke may also be used. The filtering material must be of grains of uniform size, otherwise the filter will soon clog.

When the filtering medium is clogged with mud the juice it contains is displaced with water, and the sand is then thoroughly washed with hot water under pump pressure or otherwise, according to the type of the filter. After washing, the filter is again ready for service.

These filters have been used with only moderate success in the cane industry, and their use is not general. Geerligs[15] reports the removal of considerable cane fiber, albuminoids, wax, and mineral matter by sand filters.

153. Excelsior Filters, Bagasse Filters, etc. Filters using fibrous materials such as excelsior and bagasse consist of small tanks with false bottoms filled with the filtering medium through which the juice flows upward and out by overflow. These filters have been largely discontinued because of the great heat losses inherent in their use and also because of the possibilities of fermentation and inversion. This latter difficulty is particularly evident in the case of a fermentable material such as bagasse.

154. Alcohol Precipitation. In a very old French process, alcohol was used as a precipitant in sorghum juice. Filtration followed without difficulty. The U.S. Department of Agriculture modified this process in adding an equal volume of strong alcohol to sorghum sirup of about 55° Brix: A very heavy precipitate consisting partly of gums was thrown down and was very easily removed by filter-pressing. The alcohol was recovered by distillation and the sugar was crystallized as is customary. Such processes have been patented for the cane sugar industry but have never come into use.

[15] *Loc. cit.*

155. Centrifugal Separators. Several forms of centrifugal separators have been brought out by inventors from time to time. In these the machine differs from the ordinary centrifugal in having no perforations in the basket. The defecated juice or even juice without previous removal of the mud (cachaza) is run into the machine near the bottom of the basket. The mud is thrown to the wall of the basket by the centrifugal force, and the clean juice flows over the rim.

The cost of the plant and the expense for power, loss of heat, oil, etc., are possibly the reasons for the failure to adopt this process. If the defecation is good and the separation of the precipitates therefore sharp, the centrifugals will deliver very clean clarified juice.

In experiments in Queensland with a De la Val high-speed centrifugal Behne[16] showed that a considerable proportion of colloids, waxes, and ash could be removed from clarified juice, but he considered the results inconclusive from a practical standpoint.

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[16] *Intern. Sugar J.*, November, 1933, p. 421.

CHAPTER 8

CHEMICAL REAGENTS USED IN SUGAR PROCESSES

156. List of Substances Proposed. The great number of individual substances and combinations of substances which have been proposed for use in the clarification and decolorization of sugar products may be judged by a study of the Tables 51 and 51*a* on pp. 798 and 812. This list was started by Von Lippmann in 1886 when it contained only 112 items. The translation of the 1909 list, published in previous editions of this *Handbook*, is given in Table 51, p. 798 and contains 622 substances. Recently another translation [1] has been published showing 742 items, comprising 490 inorganic chemicals, 198 organic materials, and 54 electrolytic substances. Table 51*a* on p. 812 includes all the additions made since the 1909 translation, and this is published by courtesy of the *International Sugar Journal.* Quoting from the introduction to this translation,[2] "Such a list as this is really of very great interest to the chemist, if only to show him how frequently in the last half century the same wonderful 'discovery' has been made."

157. Lime. The use of lime for the treatment of cane juice dates back to antiquity, and it is still universally used for this purpose because of its low cost and effectiveness. Up to twenty-five years ago the only form in which it was available was in large lumps of quicklime (CaO), but the introduction of powdered hydrated lime ($Ca(OH)_2$) and, more recently, of pulverized quicklime of extreme fineness, has made the use of lump lime or rock lime more or less obsolete in the cane factories of Louisiana and completely so in the refineries of the United States.

Where the freight rates will not permit the use of lime in these modern forms, the older lump lime is still used and great care must be taken in slaking it.[3] The essential conditions are that too much water must not be added, thus reducing the temperature, and that full time must be allowed for the reaction. Slaking tanks should be large so as to retain the heat of the reaction and promote slaking.

There are several methods of preparing the lime for use in defecation, the commonest and most convenient method being to prepare a milk of lime (also called lime cream in some countries) of 12° to 15° Baumé (approximately 10

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[1] *Intern. Sugar J.*, May, 1937, to February, 1938.

[2] *Intern. Sugar J.*, May, 1937, p. 187.

[3] A detailed description of the manufacture of lime on the plantation from Cuban limestone, with instructions on design and operation of kilns, is given by Martinez, *9th Annual Proc. Assoc. Cane Sugar Tech. Cuba* (1935), p. 247.

to 13 per cent CaO). The milk of lime is kept agitated by air or mechanical stirrers (preferably the latter) in a supply tank, and is pumped to the liming station through pipelines which return to the supply tank so that the reagent is kept continuously circulating. Some automatic liming systems use a lime of 2° Baumé (see Sec. 99) as permitting more accurate adjustment of the reaction. Occasionally authorities advocate adding the slaked lime to a portion of the juice to form a saccharate (see Sec. 92).

158. Quality of Lime. The lime used in the purification of the juice should be very pure. The magnesia and soluble silicates of the lime used in the defecation processes form scale in the evaporators.

Geerligs[4] gives the following tests for a good lime for clarification purposes. It should become very hot in a few minutes when treated with half its weight of water. It should form a soft cream after slaking when mixed with ten times its weight of water; this cream should not contain more than one-tenth of the original weight of lumps which fail to pass through a fine sieve, and most of these particles should soften in an hour's time. He places the maximum allowable percentage of silica, of oxides of iron and alumina, and of magnesia at 2 per cent each. As will be noted in the analysis given below, these allowable percentages of impurities are much higher than obtain in the hydrated and quicklime now sold in the United States.

Hydrated Lime. Hydrated lime is sold in powdered form packed in heavy paper sacks such as are used for cement. The convenience, cleanliness, and purity of lime in this form have led to its adoption in most refineries and in cane factories in Louisiana and other sections where the freight rates will permit. Its use in the British West Indies is on the increase. A hydrated lime from the southern part of the United States showed:

Available calcium hydroxide	97.00%
Equivalent calcium oxide	73.43
Calcium carbonate	2.04
Total calcium oxide	74.57
Silica	0.40
Iron oxide	0.08
Alumina	0.17
Magnesia	0.22
Sulfur trioxide	0.09
Loss on ignition	24.47

Fineness 100% through 300-mesh sieve

Pulverized Quicklime. Pulverized quicklime is a development of the past few years. It is separated by air flotation, and the better grades show 99 per cent and more through a 300-mesh screen. Packed in waterproof paper bags it will keep in dry storage for several months, but in a moist atmosphere the bags tend to burst in a few weeks. The analysis is about the same as that for hydrated lime on a non-volatile basis. The price and analysis of the two

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[4] *Cane Sugar Manufacture,* second edition, London, 1924, p. 165.

products are such that the pulverized quicklime results in a saving of 20 per cent over the powdered hydrate, and where the storage problem is not acute the quicklime is displacing the hydrate.

Lime from Oyster Shells and Coral Sand. The manufacture of lime, both quick and hydrated, from oyster shells is now a commercial project in Louisiana. The analyses of the finished products are similar to those given above.

Lime from coral beach sand is used to some extent in the Hawaiian factories. The following analysis of lime from this source shows high magnesia content: silica 0.18 per cent, lime 91.7 per cent, magnesia 4.15 per cent. Such a high percentage of magnesia would be considered objectionable by many sugar technologists.

159. Sulfurous Acid. The production is described in the section on white sugar processes (Sec. 131). This reagent is little used in raw sugar manufacture at present.

Aside from its bleaching effects the sulfurous acid produces a heavy precipitate with the lime which assists mechanically in the clarification. It also breaks up some of the lime salts and thus reduces the viscosity of the sirup and massecuites. Sulfured sirups and massacuites boil much more freely in the vacuum pan than those made without this reagent.

160. Carbonate of Soda ("Soda Ash"). Juices which have begun to ferment are better neutralized with soda, rather than with lime, as the latter produces soluble salts that are objectionable. The use of soda ash to neutralize excess acidity in juice from burned cane is common practice in many factories. The soda salts are also useful in neutralizing molasses. The carbonate was formerly thought to have a beneficial effect when taken into the pan in boiling low-grade massecuites, but Grogan[5] has cast doubt on its value. He found that lime is better than soda ash in boiling stringproof massecuites. Better yield, better colored sugar, quicker maturing massecuites, and the elimination of foaming in the pan were obtained by the use of lime instead of soda.

It is therefore doubtful whether soda ash has the advantages which have generally been ascribed to it. It is much more expensive than lime and has a detrimental effect on the color of the juice. Its use should be restricted to those cases where it has been definitely shown by experiment that lime will not serve as well.

161. Phosphoric Acid. Phosphoric acid and soluble phosphates have been employed in cane sugar manufacture and refining since about 1850. They have become increasingly important in both branches of the industry in the past decade.[6] In the defecation of the juice, the fertilizer triple superphosphate made by treating phosphate rock with *ortho*-phosphoric acid, is used because of its cheapness. The arsenic and lead content of this material

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[5] *Intern. Sugar J.*, July, 1932, p. 266.

[6] See Meade, *Proc. 6th Congr. Intern. Soc. Sugar Cane Tech.* (Baton Rouge, 1938), p. 1032.

is relatively high, but this is not of importance in raw sugar manufacture. For refining, pure food regulations require the use of phosphoric acid of high purity, and this is now made practically free of lead, arsenic, and sulfates by smelting phosphate rock with coke and silica in a blast furnace or an electric furnace. The acid is shipped in bulk in rubber-lined tank cars, greatly reducing the delivered price. A typical analysis follows.

Phosphoric acid (H_3PO_4)	75.68%
Phosphoric anhydride (P_2O_5)	54.73%
Iron as Fe	Less than 0.03%
Lead	0.3 ppm.
Arsenic as As_2O_3	0.2 ppm.
Fluorine	1.0 ppm.
Color	Water white

Monocalcium phosphate pastes also free of objectionable impurities are made by mixing this acid with lime in proper proportions. In the refineries, "blackpaste" is now made by treating discard boneblack dust with this clear phosphoric acid (see Sec. 366), still further reducing the cost of available P_2O_5 when discard boneblack is at a low price.

162. Various Other Chemicals Used in the Defecation of the Juice. A large number of substances other than those named above have been proposed for use in purifying the juice, but only a few have been practically applied in manufacture, and these to a limited extent.

Among substances advocated but not generally adopted are pepsin,[7] sodium aluminate,[8] and silica gel. Hyposulfite of soda, sold under various trade names, has been used in bleaching molasses and has also been added to the vacuum pans during white sugar boiling to improve color. These chemicals release SO_2 into the massecuite, acting on the coloring matter, lime salts, and iron salts.

Kieselguhr (diatomaceous earth, sold by two large companies under the trade names of Celite and Dicalite) is used as a filtering medium. There are many different grades, varying according to rate of flow and clarity of filtrates. The addition of this material to juice or sirup greatly facilitates filtration, but because of high costs its use has been limited to the refining branch of the industry (see Sec. 367).

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[7] Lanier, *3rd Conf. Assoc., Sugar Tech. Cuba* (1929).

[8] Wayne, *Facts About Sugar,* April, 1931.

CHAPTER 9

EVAPORATION AND HEATING

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163. The Evaporator Station. The juice as it comes from the clarification system contains the natural water which was expressed from the cane together with the water added for maceration purposes. The greater portion of this water must now be removed, and this is done in all modern factories by multiple effect evaporators, so called because the steam is made to do duty several times, that is, in multiple effect.

164. Definitions. Evaporation is the process of removing water from a solution by the application of heat. Heating is the process of increasing the temperature of a solution by the same procedure. The unit of heat, the Btu., is the amount of heat required to raise the temperature of one pound of water one degree Fahrenheit. Latent heat is the number of Btu. liberated by one pound of steam when it condenses into water, or the number of Btu. required to change a pound of water into a pound of steam.

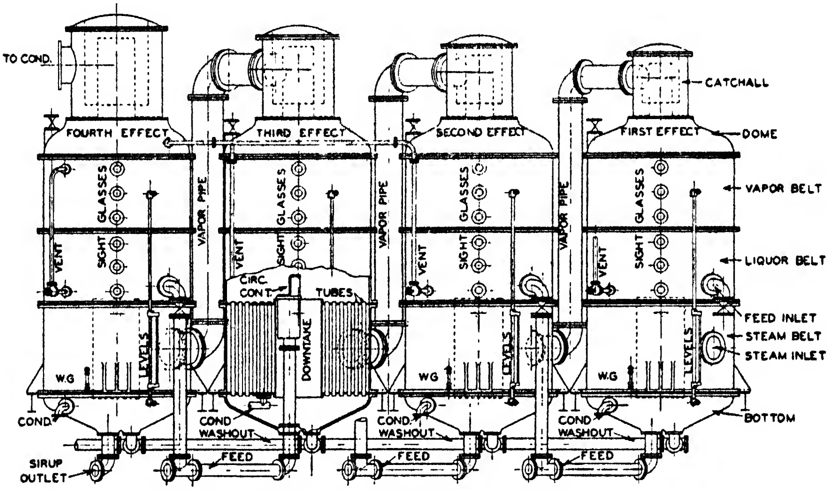
165. Elementary Evaporator. An evaporator is made up of two closed spaces, separated from one another by thin metal walls in the form of tubes, coils, or plates, called the heating surface. Steam is supplied at a fixed temperature and pressure into one of these spaces, where it condenses, thus giving up its latent heat. In the other space a solution at a lower pressure and temperature absorbs this released heat by the evaporation of water. The rate of heat flow through the heating surface is proportional to the difference of temperature between steam on one side, and the boiling liquid on the other.

To evaporate one pound of water, it is necessary to supply the equivalent of its latent heat at the pressure at which evaporation takes place, assuming that the solution is at the boiling point. If not, sufficient additional heat must be provided to raise it to that point. If, on the other hand, the solution enters the evaporator above this temperature, there will be a flash or self-evaporation, and the quantity of heat required to evaporate one pound of water will be less than the latent heat by the amount of Btu. thus released.

166. Multiple-Effect Evaporation. From the above simple statement, it is easy to understand how evaporators can be hooked in tandem, the vapor from one becoming steam for the next. It is necessary only to lower the pressure as successive units are added, thus establishing an adequate difference of temperature to enable the apparatus to function properly. This is multiple-effect evaporation.

DESCRIPTION OF APPARATUS

167. The Standard Evaporator. In the cane sugar industry, the multiple effect evaporator (Fig. 43), is made up of three, four, or five evaporators connected in series, the individual evaporators being called effects, bodies, cells, units, or pans, according to local custom. In the usual form, each of the bodies consists of a closed, cylindrical, vertical vessel, the lower part of which is a dished bottom, rather shallow, but of the full diameter of the shell. It has a manhead and openings for feed, drain, condensate, and a few other details.



STANDARD QUADRUPLE EFFECT EVAPORATOR
WITH CIRCULATION CONTROLS

FIG. 43. Standard Quadruple Effect Evaporator.

Above is the calandria, steam belt, or heating element, cylindrical in shape, about 4 to 6 ft. high, with tube sheets above and below it, having a large central tube, downtake, or downcomer. The tube sheets are filled with tubes, generally of copper, 1¼ in. to 2¼ in. in diameter, and about 1/16 in. thick, constituting the heating surface. There is a large steam or vapor inlet at the side, and in the tube sheets are drain pipes at the bottom, and vent pipes at the top, leading to the outside and eventually connecting to the condenser.

Above the calandria are the vapor belts, totaling perhaps 10 ft. in height, with sight glasses, thermometer, manhead, and other accessories. Next comes the dome or top cover to which is attached a catchall, saveall, or separator, designed to catch the drops of juice which might be entrained by the rapid vapor currents and lost. This catchall has a drain and a vapor outlet at the side.

Large vapor pipes connect each catchall with the steam belt of the succeeding effect, and finally the catchall of the last body with the condenser, where the air is removed by a vacuum pump and the vacuum is maintained by the use of cold injection water.

Steam enters the calandria of the first effect and causes the juice to boil on the inside of the tubes. The vapors leaving this juice and proceeding through the dome, catchall, and vapor pipe become steam for the second effect, and the vapors from the second effect become steam for the third, etc., the vapors from the last going to the condenser.

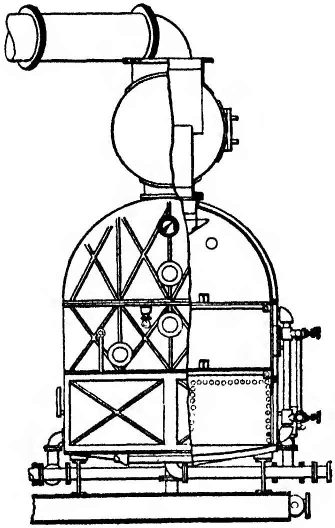


Fig. 44. Horizontal Tube Type Evaporator.

Juice is fed to the first effect and passed on to the second, the third, the fourth, etc., by appropriate feed pipes with control valves, and the concentrated sirup is removed from the last effect by a pump. Condensed steam or vapor in the steam chests must be removed continually by appropriate means, otherwise the accumulation will soon flood the heating surface and immobilize the evaporator.

All these functions are continuous, and are controlled by the evaporator operator, who adjusts the supply of steam to the first effect so as to regulate the density of the sirup leaving the last body at the desired concentration. He must also control the levels of the juice to obtain the best performance by carrying them at just the right point. (See Fig. 43.)

168. Other Types of Evaporators. The evaporator described above is the standard short tube type usually found in sugar factories. Several other designs should be mentioned briefly.

The horizontal tube evaporator was the original Rillieux apparatus. It has been considerably modified by various later designers. Steam entered the tubes at one end, and condensate and non-condensable gas left by the other. Tubes were 2 in. in diameter and 10 ft. long. Presently, the tubes are 1¼ in. diameter and 12 to 14 ft. long. A modification known as the Wellner-Jellinek has the bank of tubes arranged for three passes instead of one, each being proportioned to the volume of uncondensed steam entering, in order to maintain a higher velocity of flow. Figure 44 shows the present practice, in which it will be noted that the shells are not cylindrical, but rectangular with rounded tops and bottoms. There have been many such installations in the beet sugar industry.

The Climbing Film evaporator (Fig. 45) is a long tube vertical apparatus originated by Paul Kestner, a Frenchman, about thirty years ago. There have been many satisfactory installations since it was introduced, and the design has seen a number of improvements. There are three difficulties: (1) danger of entrainment losses, (2) necessity of providing for expansion and contraction, and (3) great sensitivity to change. Originally, the claim was made that it would not scale up, but this has not been substantiated. It has several advantages, among which is the short time of contact between the liquid and the heating surfaces, the small amount of floor space required, and the fact that it operates very well with solutions of a foamy nature. The principal difficulty is its lack of adaptability to rapidly varying conditions of operation.

The Lillie is the original film evaporator, whose object was to eliminate the effect of hydrostatic head loss by providing a method of operation whereby the heating surfaces were always covered with thin films of the solution to be evaporated. There is only one tube sheet, and the tubes are about 4½ in. diameter in a pitched horizontal position, so that the condensed steam on the inside drains towards the tube sheet against the direction of steam flow. The free ends of the tubes are blanked off and provided each with a very small orifice through which the non-condensable gases are discharged into the vapor space. There is a minimum volume of liquid in the evaporator at any time, and a pump is provided for circulating it from the bottom of the shell into a distributing tray above the bank of tubes, from which it drips, uniformly covering the heating surface. This apparatus gives very good heat transmission, and a minimum time of contact. Feed from one effect to the other is controlled by float valves. The Lillies installed in raw sugar factories have been replaced generally by the standard effect but there are still many in use in sugar refineries.

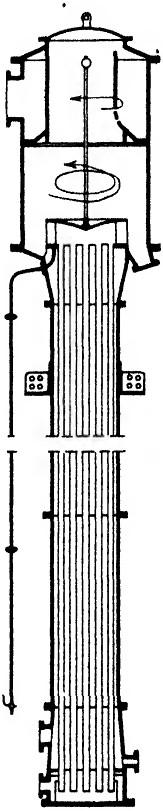


FIG. 45. Kestner Vertical Single Effect Pressure Evaporator.

169. Catchalls. In all evaporators, there is the possibility of loss of sucrose by entrainment, due to the carrying over of small drops of juice or sirup by the rapidly moving vapor currents. The apparatus to overcome this is called a separator, catchall, saveall, or catchpot, in the different localities of the sugar industry.

The operating principle of all separators is a change of direction, permitting the droplets of entrained liquid to veer away from the path of the vapors, where, upon impingement on a wetted surface, they can be salvaged and returned to the evaporator. Little entrainment will occur in any but the last effect, so that is the point to check carefully. Figure 46 shows various types of catchalls which have been in extensive use. (See also Fig. 59.)

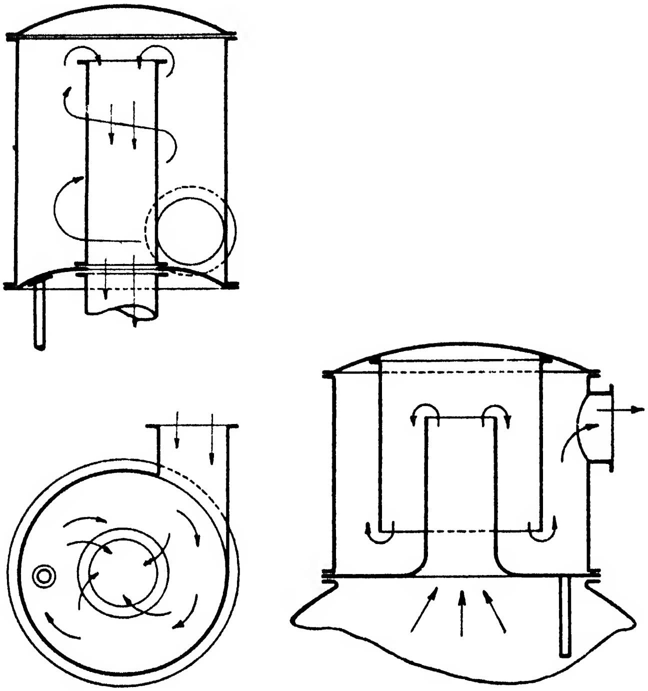


FIG. 46. Catchalls.

170. Condensers. In order to obtain a vacuum, it is necessary to condense the vapors liberated by the apparatus at the corresponding temperature.

A condenser is a closed cylindrical vessel into the upper part of which cold water is admitted. The water, after being broken up into sheets or thin streams, comes in contact with the hot vapor, condenses it, and thus increases its own temperature. This hot water now leaves through a pipe about 35 ft. long, and discharges below the level of water in a recipient called the hot well. It is much easier to understand this work if the condenser is considered as a heater whose function it is to raise the temperature of the water to as near that of the steam being condensed as possible. Subtracting the temperature of the hot water from the temperature of the steam gives the terminal difference, which is the accepted measure of condenser performance. Zero terminal difference is 100 per cent efficiency.

In order to raise a vacuum on a system of this sort, it is necessary to remove all the air and non-condensables, and this is the function of the vacuum pump.

Three designs of condensers are in use in the sugar industry: (1) the countercurrent condenser in which the vapors enter near the bottom (Fig. 59); (2) the parallel current condenser in which the vapors enter the top; and

(3) the ejector condenser which is a modification of 2 in which the water enters as fire streams converging on the water outlet, and requiring no vaccum pump (Fig. 47). When well designed and properly proportioned, these all give good results, i.e., a terminal difference of 10° or less.

It is the decided preference to have independent condensers for all pans in order not to disturb the delicate temperature balance required during their operation. In this regard, the ejector condenser answers the purpose very well, because it is of little use to have independent condensers unless each is provided with its own vacuum pump.

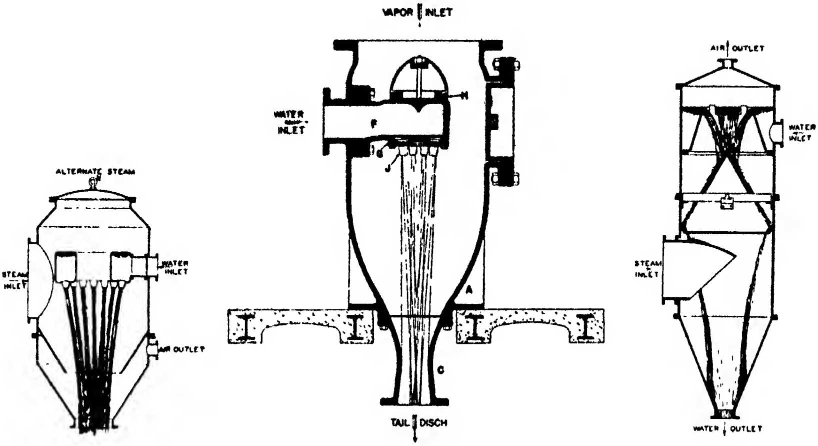


Fig. 47. Evaporator Condensers.

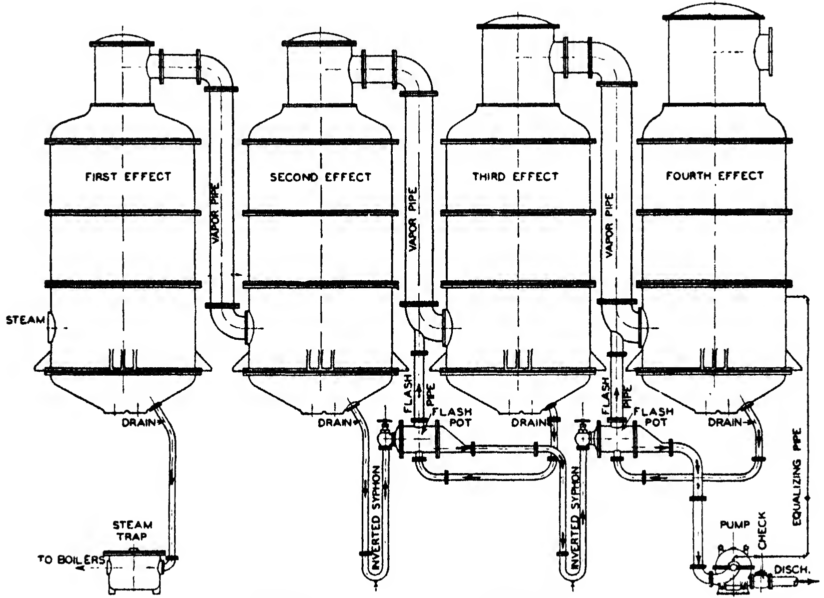
171. Vacuum Pump. A vacuum pump is simply a specially designed low-pressure compressor used to remove air from a vacuum system and discharge it to the atmosphere. The capacity required for a given condenser load is subject to wide fluctuations, depending on local conditions. A good rule of thumb is to provide piston displacement equal to the volume of injection water to be used. One curious fact is that the peak load comes at about 18 in.; it will take considerably less power either above or below this point.

172. Condensate Systems. Ample and satisfactory provisions must be made for the removal of condensed steam or vapor from the steam compartments of evaporators as fast as it is made. Any accumulation blanks off heating surface and slows down the apparatus.

If evaporation is under pressure, a simple steam trap is all that is required. If there is a vacuum and the evaporator is high enough, barometric drains can be used, which consist simply in allowing the drain pipes to extend to the ground floor into a tank where the ends of these pipes can be sealed. Water is then removed from the tank by a pump, whose suction inlet should always be higher than the ends of the condensate drains, to avoid breaking the hydraulic seals.

Formerly, it was customary to have a "sweet water pump" attached to the bottom of each steam compartment, whose function was to remove not only the water but also the non-condensable gas. Many are still in operation.

It has been the practice to send the condensate from the first effect to the boilers, because of its purity and high temperature, and to make up the shortage by adding condensate from the other bodies, preferably of highest temperature.



CONDENSATE FLASH SYSTEM FOR
QUADRUPLE EFFECT

Fig. 48. Condensate System.

A substantial steam saving can be made by "flashing the sweet water," i.e., by having the condensate from No. 2 body go to the steam chest of No. 3, where it flashes down to vacuum temperature, and so on for successive bodies.

The main objection to this scheme is that it tends to flood the steam chests, particularly if the drainage pipes are of restricted area. To overcome this, there is a modification in which external flash tanks are used, as shown in Fig. 48. Here only the flash or self-evaporation enters the steam chest, since the condensate proceeds from flash tank to flash tank, and all of it is removed from the last.

The condensate from each steam chest should be tested at regular intervals for sugar by the alpha-naphthol method to guard against contamination of boiler feed water.

173. Feeding Systems. The feeding procedure is to admit defecated juice to the liquor side of the first effect, and then transfer it successively to the second, third, etc., and finally to the last, whence it is removed, concentrated into sirup.

The juice entering the first body is usually below the boiling point, but in transferring to succeeding bodies the reverse is true and there will always be a flash in the feed pipe due to the decreased pressure and temperature. This means that the volume passing through these pipes is many times the volume of the juice because of the swelling effect of the steam bubbles for which an allowance must be made, and calculated juice velocities (omitting flash) must not exceed 3 to 4 ft. per second.

Juice or sirup must be removed from each body at a point where the maximum concentration is known to obtain. Each body should have a satisfactory liquor level gage, indicating the height of the boiling juice. After the proper points have been determined, marks can be placed so that the correct levels are at once apparent. Usually this means that half of the bottom sight glass should be clear of froth. Any point higher or lower than this shows faulty operation. It will be found that the liquor levels, when this point is reached, will be surprisingly low.

Nor will the same liquor level give the same froth level always. It depends on the cleanliness of the tubes and the rate at which the evaporator is run. Since this is so important it has resulted in the development of a number of automatic level control devices. Most of these operate with floats and are objectionable for that reason. Still others are adjustable overflow controls in which the float is eliminated. There is still the necessity of adjusting for changed rate, or fouled heating surface, although a fair compromise can be found. Another design, the so-called circulation control (Fig. 43), regulates the level of the froth above the heating surface, irrespective of the hydrostatic level of the juice, and this design has given excellent results. Like the overflow control, it has no moving parts.

174. Instruments. In order to be able to control the work of evaporators it is indispensable that they should be equipped with adequate instruments and accessories as given in the following:

For each body: liquor level gage; vacuum or pressure gage; thermometer; water gage on steam chest; sight glasses, one as near tube sheet as possible; one 16 in. manhole in liquor space, one in bottom; acid vapor valves; safety valve.

For the set: a sirup test cup; a steam flow meter (optional); steam valve.

JUICE HEATERS

175. Design of Heaters. The designs of juice heaters parallel those of surface condensers. Since the range of heating is much greater, many more passes will be required. The tubes should be so nested that the juice velocity is at least 5 to 7 ft. per second. If the velocities are too low, not only will the heat transmission be less, but also the heating surfaces will foul rapidly.

Of the many types of juice heaters, the simplest is made of pipe and fittings

(Fig. 49). Each pipe constitutes a unit having a certain number of tubes expanded into tube sheets. The flow of steam and juice is simple, and the friction losses are low. Each unit is one pass, in other words juice runs in parallel in all the tubes in one pipe. Combinations can be made to suit any heater problem.

The conventional cylindrical heater, either vertical or horizontal (Fig. 50) has cells in each head, in each of which is enclosed a definite number of tubes, so that the juice passes through each cell successively and finally out of the heater. These heads have hinged covers bolted on. In the horizontal units it is customary to provide baffles on the steam side to guide the path of steam, as in steam chests of evaporators. Copper tubes are used almost universally owing to better heat transmission.

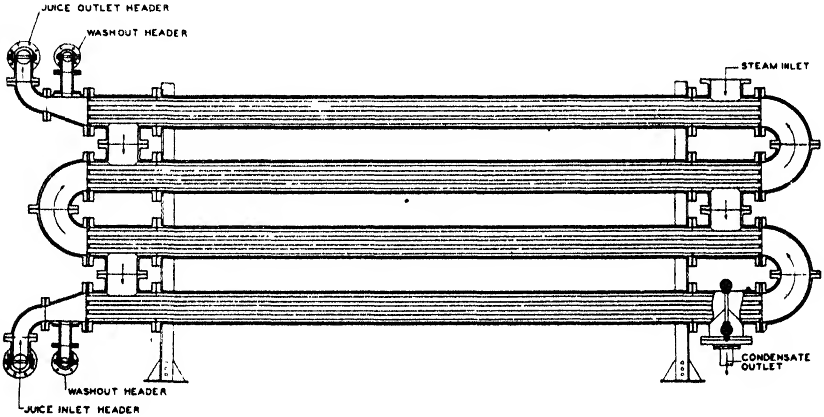


Fig. 49. Four-Pass Unit Heater.

When heaters lose efficiency, they have to be cleaned by circulating a light caustic soda solution through the juice paths, while at the same time applying heat. (See Sec. 218.) In all sugar factories of any size, there is always a spare heater so that each unit can be cleaned without retarding the operation of the mill.

The heat transmissions obtainable are in the neighborhood of 250–300 Btu. per square foot per hour per degree difference, depending on the speed of circulation and cleanliness.

HEAT TRANSMISSION

In order to obtain the best performance out of an evaporating apparatus, it is essential to know the fundamental facts governing the transmission of heat through surfaces. This is an extremely complicated matter, and only the high lights will be covered in this text.[1]

In an evaporator, pan, or heater, the resistance to the flow of heat is made up of three distinct parts: the steam film resistance, the metal resistance, and

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[1] See *Heat Transmission*, by McAdams.

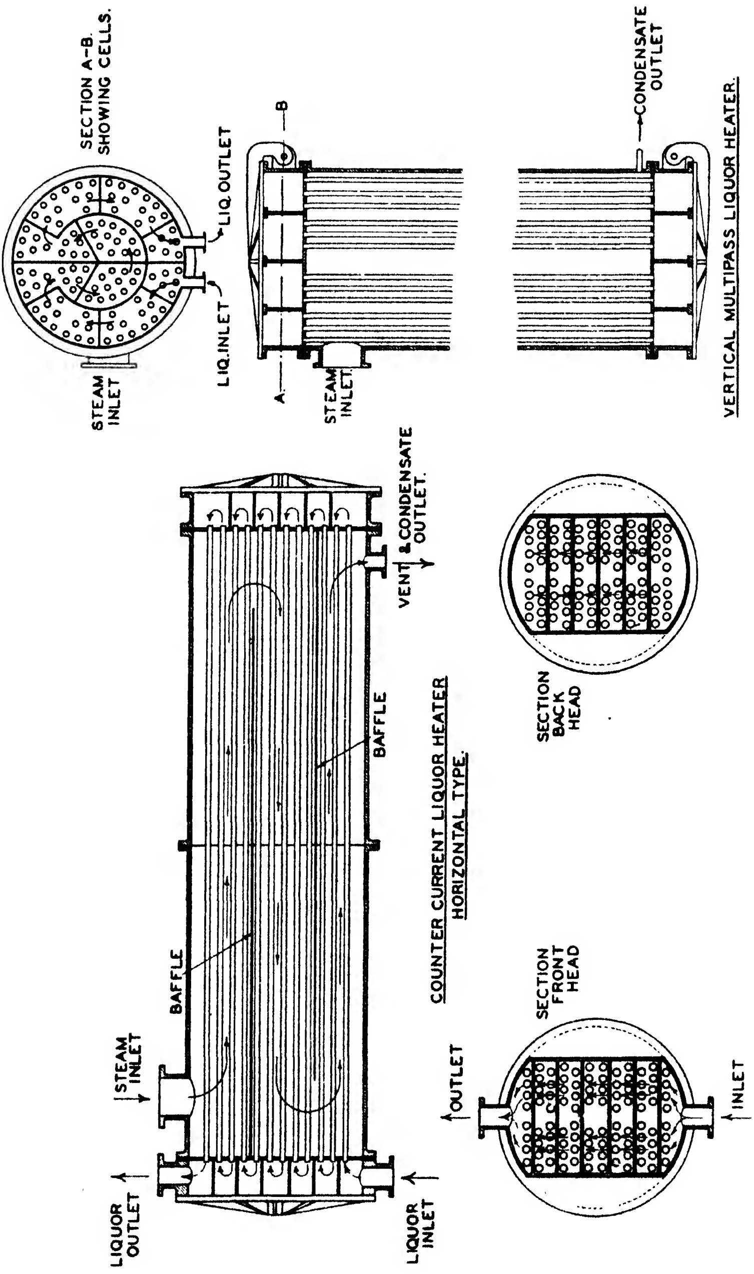

Fig. 50. Vertical and Horizontal Heaters.

the liquid film resistance. Naturally, the lower these influences, the higher the heat transmission will be.

176. Steam Film Resistance. By the steam film resistance is meant the resistance to the entrance of heat into the surface of the metal tubes. This is understood as representing the impediment offered by the film of water present because of the condensation of steam. Even with perfectly new, clean tubes, it will amount to a considerable figure. This condition is aggravated if the tubes become corroded on the steam side, or if deposits of any kind are formed, as these increase the thickness of the film of water. In like manner, it often happens that the exhaust steam used has not been cleaned of lubricating oil, which forms a coating and retards the work. (For practice in cleaning, see Sec. 218.)

177. Gas Venting. The occurrence of non-condensable gas in the steam compartments of evaporators is inevitable. Some of this gas is air that has entered the system under vacuum, through any leaky joint, valve, or stuffing box. Ammonia is liberated from the juice by the breaking up of organic compounds, incident to alkalization, heating, and concentration. Gas, from whatever source, accumulates and must be removed continuously by means of vent pipes properly located and ultimately discharging into the condensers. Non-condensables will attack the tubes and hinder efficient transmission of heat, thus causing decreased capacity.

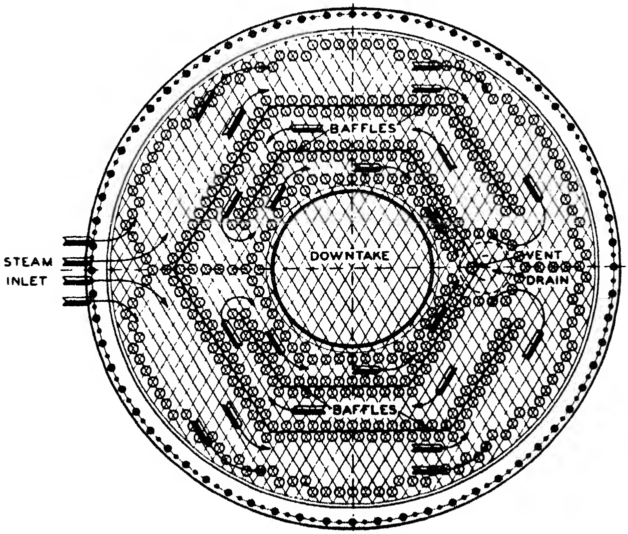

FIG. 51. Steam Baffles for Evaporators.

178. Baffling of Evaporators. In vertical evaporators, the accepted method is to insert baffle plates between the tubes extending from the upper to the lower tube sheets. These baffles are arranged so that they cause a definite flow of steam to one point whence the non-condensables can be removed (Fig. 51). Ammonia gas is lighter than steam and will tend to rise, whereas air and carbon dioxide are heavier and will tend to accumulate in the lower part. The baffling should be such as to prevent stratification by keeping a high vapor velocity in the steam chests, but even so, provisions should be made for gas removal from both the upper and lower parts.

179. Steam Temperature. It has been established in practice that the heat transmission will be greater as the temperature of steam or vapor being condensed is higher. Figure 52 shows the value of the overall heat transmission U, as the steam temperature is increased.

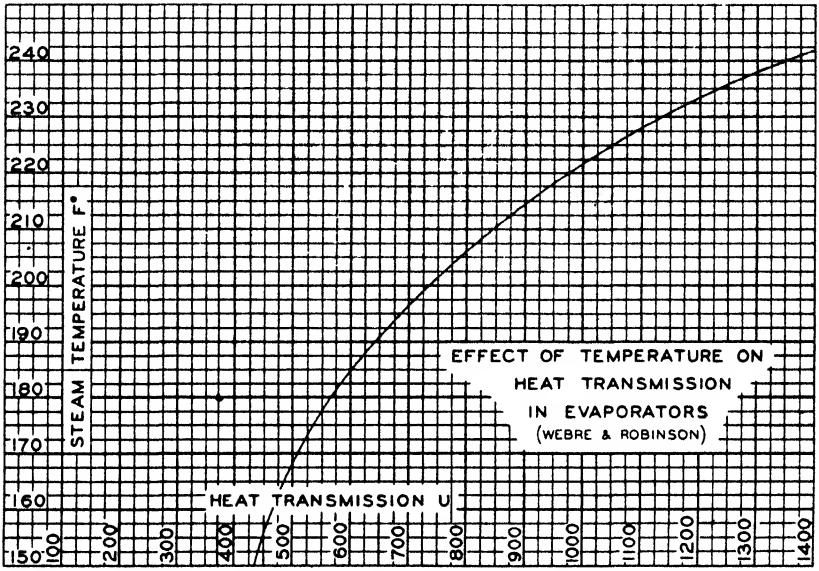

FIG. 52. Heat Transmission and Steam Temperature.

180. Metal Resistance. The resistance to the flow of heat through the metal of the tubes is small. The conductivity of various metals usually expressed as K is given in Btu. per square foot per hour per degree difference on metal 1 ft. thick. The conductivity of the principal metals [2] together with the heat transmission if the metals are only 1/16 in. thick, as in ordinary evaporators, is given in the table below.

METAL	K(12 IN. THICK)	U($\frac{1}{16}$ IN. THICK)
Aluminum	85	16,320
Yellow brass	55	10,560
Copper	238	45,700
Wrought iron	40	7,680
Nickel	32	6,120

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[2] Walker, Lewis, and McAdams, *Principles of Chemical Engineering.*

Since it is seldom that the heat transmission exceeds 1000, the influence of the metal is minor.

181. Liquid Film Resistance. The greatest impediment to the flow of heat is on the liquor side of the surface, and on this score much can be said and done. It is not merely a matter of liquid film, but the whole series of physical reactions.

182. Hydrostatic Head Losses. The correct boiling temperature of any solution at any pressure or vacuum is that at which ebullition takes place at the surface of the liquid. If boiling occurs below the surface, such boiling is at a higher pressure owing to the superposed hydrostatic head, and hence it is also at a correspondingly higher temperature. The heating surfaces of evaporators and pans are always covered with juice or massecuite to a greater or lesser depth, and hence the influence of the hydrostatic head will be felt. The average pressure on the surface, taken at one-half the indicated juice level in the tubes, is the determining criterion. The effect of the pressure will be greater as the vacuum increases, since the increment of temperature for a given increment of pressure is greater as the pressure is lower.

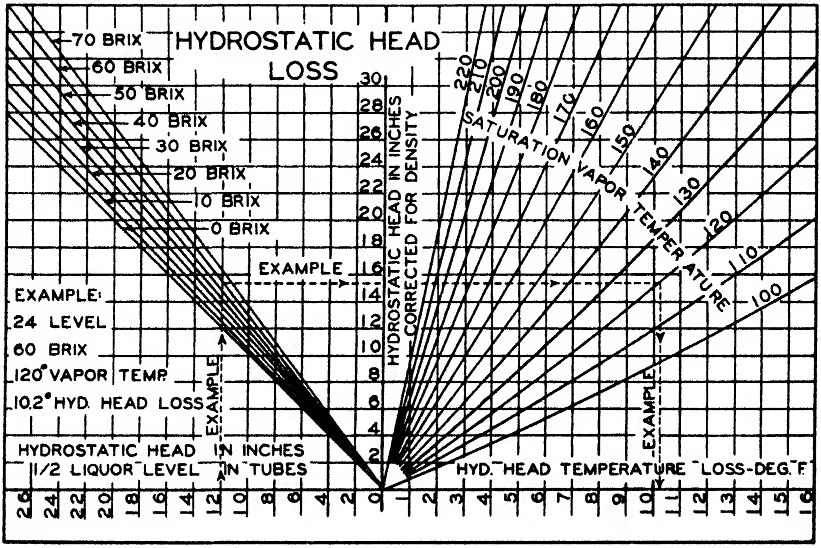

Fig. 53. Hydrostatic Head Loss.

This is illustrated in Fig. 53. A correction is given for the variation of densities. The difference between the temperature at the surface of the liquid and that at the average submergence is called the hydrostatic head loss, shown in the diagram, and means that so much of the available temperature difference for the evaporator is dissipated, with resulting decreased capacity. In all but apparatus with mechanical circulation, this loss is inevitable.

The objective is to limit it to a minimum by maintaining the lowest liquor levels consistent with efficient operation. This is effected by regulating the height of the froth above the upper tube sheets to about 8 to 10 in. With more than this, a reduction of capacity will result. With less, there is danger of running dry on some of the tubes.

The actual level of the liquid in the indicating water gage glass to obtain this optimum point is a function of many variables such as: (1) the diameter of the tubes, (2) the length of the tubes, (3) the operating speed of the evaporator, (4) the cleanliness of the tubes, and (5) the pressure or vacuum in each body.

With a given length of tube, the smaller the diameter the lower the liquor level required, because the cross section of the path of the vapors is reduced and hence, the velocity is increased.

With a given tube diameter, if the length is increased, little additional juice level is required for a reason similar to the above, namely, a speed acceleration towards the top.

The influence of hydrostatic head on an evaporator such as that studied in case I (p. 154), is easily estimated.

The overall temperature difference available is determined thus:

A Temperature corresponding to 9 lb. gage pressure		237.0°
Temperature corresponding to 26 in. of vacuum		125.0°
Overall temperature difference		112.0°
B B.p.r. losses also calculated for case I are (Fig. 54):		
First effect (Bx. = 19.3°)	1.0°	
Second effect (Bx. = 25.0°)	1.5°	
Third effect (Bx. = 36.3°)	2.5°	
Fourth effect (Bx. = 70.0°)	10.0°	
Total		15.0°
Temperature difference with B.p.r.'s deducted		97.0°
C Hydrostatic head losses for 24 in. level (Fig. 53):		
First effect	1.3°	
Second effect	1.8°	
Third effect	3.5°	
Fourth effect	9.6°	
Total		16.2°
Temperature difference with hydrostatic head loss deducted		80.8°
Loss of capacity due to hydrostatic head		16.7%

Thus, surprising as it may seem, the working level in a well-proportioned long-tube evaporator is not much more than that in a short-tube, which partly accounts for the fact that the heat transmission in the former is certainly as good as it is in the latter.

As the speed of operation increases the height of the liquid can be lowered a great deal and still retain the proper froth level. This results in a reduction of the hydrostatic head and, therefore, in a substantial increase in overall heat transmission.

If the tubes foul, the scale will be thicker at the lower than at the upper ends, and for that reason the juice levels have to be raised to obtain adequate froth heights. Then also, as the tubes foul, the evaporator slows down, and that imposes higher levels as was pointed out above.

As the vacuum increases, the specific volume of the vapors increases, which means that more cubic feet of vapor are released per unit of time inside of the tubes. This in turn means lower levels.

The flow of the mixture of vapor and liquid in an evaporator tube is analogous to an air lift pumping water from a well. The difference is that the bubbles grow progressively from the lower part of the tube, by far the greater proportion being formed at the upper ends because of the decreased hydrostatic head. At the bottom of the tube only heating occurs, and the rate of flow is sluggish. The heaviest scaling and incrustation usually occur at the point at which vapor bubbles begin to form.

183. Viscosity. As a solution grows more viscous, heat transmission becomes more and more difficult. In sugar evaporators, this difficulty is not serious at low concentrations, but it assumes utmost importance in the last body where the final density is attained. It is the inevitable price of operation, and this price is directly proportional to the Brix of the sirup. For that reason, when there is a shortage of capacity, this concentration must be reduced, thus taking the penalty of a heavier load on the vacuum pans.

184. Fouled Surfaces. The greatest limiting factor to all evaporators is the fouling of the heating surfaces. With clean metal, there is always ample capacity, but soon the surfaces begin to foul, and the rating drops, very rapidly at first, then slowly progressing, until at the end of a certain period it is necessary to shut down for a cleanout as the capacity is no longer adequate for the work.

The period of operation from cleanout to cleanout varies with local conditions (see Sec. 218). Sometimes it is only three days, sometimes two weeks. In Cuba, Puerto Rico, and Santo Domingo, standard practice is to clean out every week. Many large mills are equipped with spare evaporators so that one is always available for cleaning.

METHODS USED IN EVAPORATOR CALCULATIONS

185. Rillieux's Three Principles. Consideration must now be given to the method used in making evaporator calculations to arrive at the proper selection of apparatus for a given task, as well as an accurate appraisal of the performance which may be reasonably expected. Norbert Rillieux, to whom is credited the development of multiple-effect evaporation, made a firm statement of the fundamental basis of his investigations and conclusions, which he called "principles."

First Principle. In a multiple-effect evaporator, for each pound of steam used, as many pounds of evaporation will result as there are units in the set.

Second Principle. If vapors are withdrawn from any unit of a multiple-effect evaporator to replace steam in a concurrent process, the saving of steam will be equal to the amount of vapor so used divided by the number of units in the set and multiplied by the sequence position of the unit from which vapor has been withdrawn.

Third Principle. In any apparatus in which steam or vapor is condensed, it is necessary continuously to withdraw the accumulation of non-condensable gas which is unavoidably left in the heating surface compartment.

The first principle received early recognition, and constitutes the fundamental idea of multiple-effect evaporation. It is a rough approximation, to be commended for its simplicity. The significant deviation comes from the fact that Rillieux did not realize the influence of the temperature of the solution entering the evaporator.

In the majority of cases, the juice enters the first effect at a temperature lower than its boiling point, and, therefore, the evaporation will be less than 1 lb. per pound of steam used. However, as the partially evaporated juice is transferred to succeeding cells, which operate at progressively lower temperatures owing to the flash, the evaporation will be more than 1 lb. per pound of vapor. The extent of these positive and negative variations depends on the temperature of the incoming solution, as compared with those in the evaporator, and the proportion of the solution which has to be evaporated. It is very seldom that there is a balance, and it has become customary to expect considerably less than the performance promised by Rillieux.

If each body of the multiple effect were supplied with a solution at its boiling temperature, then Rillieux's first principle would hold.

The second principle has been checked against heat balances and is substantially correct. By its use, great steam economies have been made possible. It is strange that it was not generally recognized until the beginning of the present century, and there are many sugar factories in operation, even today, where its use has been completely overlooked. Vapor pans and vapor heaters are based on Rillieux's second principle.

With the third principle, no engineer can have any quarrel, nor would he think of installing any steam-using apparatus without providing adequate venting.

186. The Heat Balance. Since Rillieux's first principle is too inaccurate for the requirements of today, other methods have been devised for arriving at the desired results. These involve a complicated series of fractions and to avoid this the writer (A. L. W.) devised a much simpler and more practical method involving the use of trial and error. The result is the so-called heat balance, which has profitably been employed during the past thirty years.

By this method the evaporator calculations follow an accounting procedure, in which everything is tabulated in columns to show the flow of heat and liquid through the succeeding bodies of a multiple effect. Each reaction is described briefly, so that it is possible to write down the entire flow for an evaporator on one sheet of paper. After this has been done, all the results are available in convenient form for use in design or analysis.

The trial and error comes in guessing at the amount of steam required for a given set of conditions and in following through with the calculations for the entire multiple effect. With this result in hand, another set of figures is made by correcting the weight of steam assumed in the first guess to compensate for the excess or deficit in the amount of evaporation required. Usually three trials are sufficient.

187. Steam Table. At the end of this chapter is an abbreviated steam table, covering the range commonly encountered in sugar factory calculations. In it are given the properties of steam most likely to be used such as pressure and vacua, temperatures, latent heat (enthalpy), and specific volumes of steam and vapor at the various temperatures.

188. Boiling Point Rise. The boiling temperature of sugar solutions is greater than that of water at the same pressure or vacuum, and the increase is called the boiling point rise, usually abbreviated b.p.r. (also called boiling point elevation or b.p.e.). Study of b.p.r. is complicated, since the function varies with the purity. (See Sec. 235.) For purposes of evaporator calculations, the table below is sufficiently accurate. The b.p.r. is exactly proportional to the weight of solids dissolved per 100 parts of water, which corre-

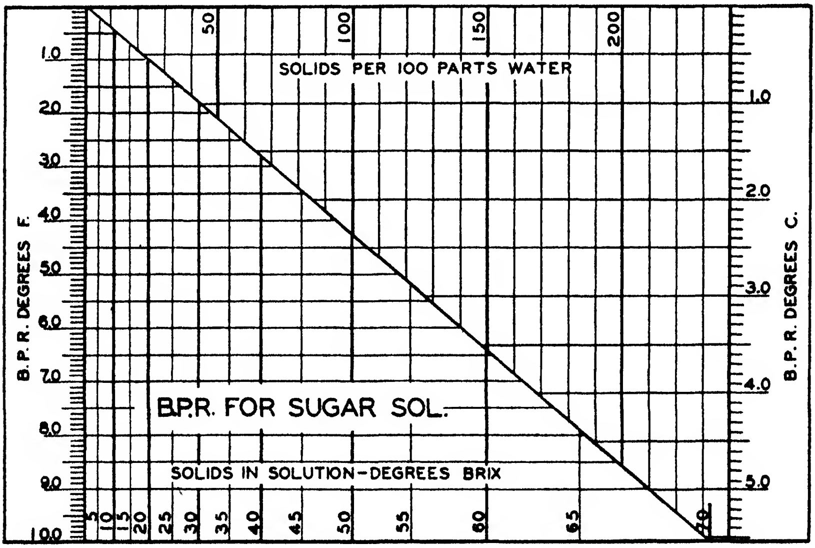

Fig. 54. Boiling Point Rise for Sugar Solutions.

sponds to Brix/(100-Brix). If a recheck is desired, it is necessary to get only one point in the straight line curve, and from this, the b.p.r. for all other densities can be read (see Fig. 54).

Degrees Brix	20.0	30.0	40.0	50.0	60.0	70.0
B.p.r., °F.	1.1	1.8	2.9	4.3	6.5	10.0[8]
B.p.r., °C.	0.6	1.0	1.6	2.4	3.6	5.5

EXAMPLES OF SUGAR FACTORY CALCULATIONS

189. Practical Application. To make practical applications of the information outlined in the preceding pages an analysis will be made with the following sugar factory conditions assumed.

Cane ground per hour	50 tons
Weight of juice per hour	100,000 lb.
Temperature of raw juice	100° F.
Temperature of heated juice	218° F.
Temperature of defecated juice	205° F.
Brix of defecated juice	16.0
Brix of sirup	70.0
Purity of sirup	86.0
Exhaust steam available	9.0 lb. gage
Vacuum obtainable	26.0 (sea level)

Five stations involve the use of heat: evaporation, heating, vacuum pans, power, and radiation. Various cases will be considered and analyzed jointly for overall performance after the individual items have been briefly outlined, since some of these are the same for all combinations. The various heat-consuming operations will be discussed in the following sections.

190. Heat to Evaporators. The ratio of evaporation is $\frac{70-16}{70} = 0.771$ or 77.1 per cent. The amount of evaporation is therefore 100,000 × 0.771 or 77,100 lb. per hour. This evaporation may be achieved by using any one of many evaporator combinations, the ultimate requirements being determined by the available steam, which will depend upon the fiber content of the cane and the moisture in the bagasse. (See chapter on boilers.) Expenditures in providing economies much in excess of the available supply of steam as determined by the fuel value of the bagasse produced are not justifiable.

191. Heat to Heaters. The load at the heaters consists in raising the temperature of raw juice slightly above the boiling point, or 218° F., from its initial of 100° F. The heat involved is $100{,}000 \times (218 - 100 = 118) = 11{,}800{,}000$ Btu. The origin of this heat will have a decided bearing on the economy of the factory, and will be elaborated in each case.

192. Heat to Vacuum Pans. Since there is 100,000 lb. of juice at 16 Brix, the solids entering the vacuum pan station per hour amount to 100,000 × 0.16 or 16,000 lb. The sirup is at 70 Brix and 86 purity. It is assumed that the

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[8] Determined experimentally by Webre for Cuban cane juice. (See also Table 12, p. 699.)

standard three-boiling system (p. 208) will be used, and that molasses will be diluted to 70 Brix. It is explained in the chapter on vacuum pans (p. 203) how low density sirup and molasses make for increased steam consumption and reduced pan capacity, and that the Brix assumed here is thoroughly practical. In this same chapter (p. 213) the solids going through the system are given in percentages of sirup solids for all strikes, and these figures are used as a basis for the following:

A STRIKE (boiled to 93 Brix)

Material	*Solids % Solids in Sirup*	*Solids Lb./Hr.*	*Brix*	*Pounds as Fed to Pan*
C sugar	11.70	1,870	100	1,870
Sirup	80.90	12,930	70	18,500
A molasses	29.40	4,700	70	6,720
Total weight				27,090
Total solids: 19,500 lb. @ 93 Brix				21,000
Evaporation				6,090

Assuming pan factor of 1.10,

Steam load = 6,090 × 1.10 = 6,700 lb.

Weight per cubic foot @ 93 Brix = 93.82 lb.

$$\text{Cubic feet per hour} = \frac{21{,}000}{93.82} = 224$$

Cubic feet *A* massecuite per 24 hours = 224 × 24 = 5,376

B STRIKE (boiled to 94 Brix)

Material	*Solids % Solids in Sirup*	*Solids Lb./Hr.*	*Brix*	*Pounds as Fed to Pan*
A molasses	28.02	4,480	70	6,400
C sugar	4.00	640	100	640
Sirup	10.99	1,758	70	2,510
Total weight				9,550
Total solids: 6,878 lb. @ 94 Brix				7,310
Evaporation				2,240

Assuming pan factor of 1.15,

Steam load = 2,240 × 1.15 = 2,575 lb.

Weight per cubic foot @ 94 Brix = 94.27 lb.

$$\text{Cubic feet per hour} = \frac{7{,}310}{94.27} = 77.5$$

Cubic feet *B* massecuite per 24 hours = 77.5 × 24 = 1,860

C STRIKE (boiled to 96 Brix)

Material	*Solids % Solids in Sirup*	*Solids Lb./Hr.*	*Brix*	*Pounds as Fed to Pan*
B molasses	23.30	3,730	70	5,330
Sirup	8.48	1,357	70	1,940
Total weight				7,270
Total solids: 5,087 lb. @ 96 Brix				5,300
Evaporation				1,970

Assuming pan factor of 1.20,

$$\text{Steam load} = 1{,}970 \times 1.20 = 2{,}365 \text{ lb.}$$

$$\text{Weight per cubic foot @ 96 Brix} = 95.17 \text{ lb.}$$

$$\text{Cubic feet per hour} = \frac{5{,}300}{95.17} = 55.6$$

$$\text{Cubic feet } C \text{ massecuite per 24 hours} = 55.6 \times 24 = 1{,}335$$

SUMMARY

Evaporation *A* pans	6,090 lb./hr.	Steam *A* pans	6,700 lb./hr.
Evaporation *B* pans	2,240 lb./hr.	Steam *B* pans	2,575 lb./hr.
Evaporation *C* pans	1,970 lb./hr.	Steam *C* pans	2,365 lb./hr.
Evaporation all pans	10,300 lb./hr.	Steam all pans	11,640 lb./hr.

193. Heat Equivalent of Power. Besides the heat consumed by the evaporators, pans, and heaters, a certain amount, corresponding to the power used in the factory by the mills, pumps, centrifugals, etc., must be considered. In the ordinary well-designed raw house, this should not exceed 1 kw. per ton per day. The example given in the test would grind 1200 tons and hence 1200 kw. would be a normal load. Since one kilowatt-hour corresponds to 3415 Btu., the heat expense is 4,098,000 Btu. per hour.

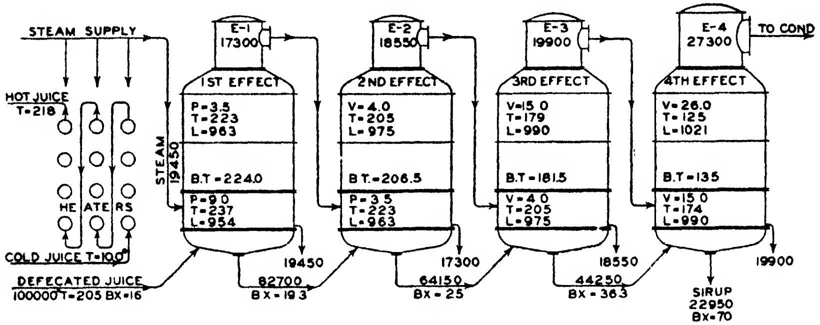
194. Radiation Losses. It is impossible to operate a sugar mill without loss of heat by radiation, and it is therefore necessary to make an allowance, which is taken arbitrarily at 2.5 lb. of steam per hour for every ton of cane ground per day. This gives 3000 lb. of steam per hour.

195. From and At Equivalent. It has been customary in steam plant practice to convert all steam used into pounds from and at 212° F. The latent heat at 212° is 970.4 Btu. per pound. Multiply the actual steam used by its latent heat and divide by 970.4. One boiler horsepower is 34.5 lb. of steam from and at 212° F.

CASE I. STRAIGHT QUADRUPLE EFFECT

The simplest and most widely used combination is the following: (1) quadruple effect evaporator, (2) heating with steam, and (3) vacuum pans operated on steam.

The heat requirements of all stations have been given above with the exception of the evaporator, which must now receive attention. The best procedure is to make a simple sketch on which operating conditions are set down as they are worked out (Fig. 55).



CASE I QUADRUPLE EFFECT EVAPORATOR

WITH STEAM HEATING

FIG. 55. Diagram of Case I.

196. Temperature Distribution. To arrive scientifically at the correct distribution of temperature differences in the various bodies of a multiple effect is a complicated task in which many variable factors have to be considered. Even after the effort has been made, actual practice seldom agrees with theory, and hence the effort hardly seems justified.

The flow of heat through the surfaces of a multiple-effect evaporator is analogous to the flow of water through pipes. An available overall difference is represented by the range from the temperature of steam in the first effect to that corresponding to the vacuum in the condenser. If the resistance to the flow of heat were equal in all the cells of the evaporator, the temperature differences would likewise be equal. Since this is not so, the distribution must adjust itself in accordance with the varying resistances.

When a multiple effect in the cane sugar industry has its total surface equally divided, experience has shown that the differences of pressures will also be equally divided, and the individual temperature differences can then be calculated accordingly. This may not be scientific, but it corresponds to practice.

Deviations from this standard will occur if any particular body of the multiple effect becomes fouled with scale or is gas bound, or if the levels of juice are improperly adjusted, etc., and the temperature difference at that point will be increased at the expense of the other bodies not so affected. The same situation obtains if the surfaces are unequally divided, increased differences occurring in the bodies having the least amount of surface. In like manner, if the work in any particular body is boosted arbitrarily, the temperature drop will become greater at that point and less at the others. Such would be true if vapors were withdrawn from the first body of a multiple effect for juice heating, provided that its surface was equal to that of the others.

In these cases, it does not mean that the evaporator will refuse to perform, but simply that it will be compelled to operate under conditions at variance with the established standard. Its capacity and efficiency then may or may not be affected adversely, depending on whether the deviation is due to faulty operation or a shift of the load, as in the last example.

By making an equal division of pressures between the various bodies of the set, the following is the result.

Position	Pressure or Vacuum	Pounds Pressure Difference	Temperature Steam or Vapor	Value of Latent Heat
Steam chest 1	9.0 lb.	5.5	237	954
Steam chest 2	3.5 lb.	5.5	223	963
Steam chest 3	4.0 in. vac.	5.5	205	975
Steam chest 4	15.0 in. vac.	5.5	179	990
Vapor belt 4	26.0 in. vac.	5.5	125	1021

(See Steam Table on p. 171.)

These data are now entered on the sketch referred to.

197. Determination of Boiling Point Rise. To arrive at the b.p.r. in the various bodies, it is necessary to make an approximation of the expected densities before the balance is calculated. This can be roughed out as follows.

Leaving the first effect,	19 Brix,	corresponding	b.p.r. = 1.0°	approx.
Leaving the second effect,	25 "	"	b.p.r. = 1.5°	"
Leaving the third effect,	36 "	"	b.p.r. = 2.5°	"
Leaving the fourth effect,	70 "	"	b.p.r. = 10.0°	"

The boiling temperature of the juice in each effect is now calculated by adding these b.p.r.'s to the corresponding vapor temperatures, and the results are set down on the sketch, as before.

198. Heat Balance. In making the calculations, it is not necessary to exceed slide rule accuracy. The completed heat balance is given as finally worked out, with a summation of the evaporation in each body, as well as the deviation from the required amount. Also there are the pounds of evaporation per pound of steam.

If the juice had entered the evaporator at a lower temperature, it would have taken more steam for the operation by the amount required to raise this temperature to the assumed figure of 205. This would have been true with open defecation where juice goes to the evaporators at about 180° F. In like manner, an opposite correction would have to be made if the juice had arrived at a temperature higher than that assumed.

HEAT BALANCE. CASE I

Body	*Description of Operation*	*Btu./Hr.*	*Juice Lb./Hr.*
1.	Steam, 19,450 lb. @ 237° F. = 19,450 × 954 =	18,550,000	100,000 Bx. = 16.0
	Deduct for heating juice 100,000 × (224 − 205) =	1,900,000	
	Available for evaporation	16,650,000	
	Latent Heat @ 223 = 963, $E_1 = \frac{16,650,000}{963} =$		17,300
	Transferred to No. 2		82,700 Bx. = 19.3
2.	Vapor from first effect	16,650,000	
	Add flash 82,700 × (224 − 206.5) =	1,445,000	
	Available for evaporation	18,095,000	
	Latent Heat @ 205 = 975, $E_2 = \frac{18,095,000}{975} =$		18,550
	Transferred to No. 3		64,150 Bx. = 25.0
3.	Vapor from second effect	18,095,000	
	Add flash 64,150 × (206.5 − 181.5) =	1,600,000	
	Available for evaporation	19,695,000	
	Latent Heat @ 179 = 990, $E_3 = \frac{19,695,000}{990} =$		19,900
	Transferred to No. 4		44,250 Bx. = 36.3
4.	Vapor from third effect	19,695,000	
	Add flash 44,250 × (181.5 − 135) =	2,055,000	
	Available for evaporation	21,750,000	
	Latent Heat @ 125 = 1,021, $E_4 = \frac{21,750,000}{1,021} =$		21,300
	Sirup from fourth effect		22,950 Bx. = 70.0

$E_1 = 17,300$
$E_2 = 18,550$
$E_3 = 19,900$
$E_4 = 21,300$
$E = 77,050$

Evaporation required	77,100 lb.
Evaporation calculated	77,050 lb.
Slide rule error	50 lb.

$$\text{Evaporation per pound of steam} = \frac{77,050}{19,450} = 3.96$$

No correction has been made for specific heat, which is less than unity. If such an allowance is made, it will be found that it is in one direction where there is heating, and in the other where there is flash, the two almost balancing. (See p. 215 for specific heats of sugar solutions.)

It is interesting to note how the steam consumption for this quadruple effect compares with that promised by Rillieux's first principle.

Steam by heat balance	19,450 lb./hr.
Steam according to Rillieux, 77,200/4	19,300 lb./hr.
Deviation	150 lb./hr.

This is a small error, but on the other hand, if the temperature of the entering defecated juice had been 180, according to the heat balance, the steam consumption would now become 22,120 lb. per hour, whereas according to Rillieux, it would remain 19,300 and the error would be 14.6 per cent, which certainly cannot be called negligible.

Summarizing the overall heat requirements of case I gives the following:

Station	*Btu./Hr.*	*Steam/Hr.* *@ 9 lb. Gage*	*Steam/Hr.* *from & at 212° F.*
1. Evaporator	18,550,000	19,450	19,100
2. Heaters	11,800,000	12,350	12,150
3. Vacuum pans	11,100,000	11,640	11,450
4. Power	4,098,000	4,300	4,220
5. Radiation	2,862,000	3,000	2,950
Total	48,410,000	50,740	49,870
Corresponding boiler horsepower			1,445

Vapors to be condensed:

From evaporator (E_4)	21,300 lb./hr.
From vacuum pans	10,300 lb./hr.
Total	31,600 lb./hr.

CASE II. QUADRUPLE WITH NO. I VAPOR HEATING

In many sugar factories the heating of the juice is done with vapors robbed from the first body of a quadruple effect. The distribution of pressures and temperature is substantially the same as in case I.

The heat balance given is calculated in the same manner as before, except that the necessary amount of heat is withdrawn from the vapors leaving the first effect for vapor heating as indicated. This is the same as case I, namely, 11,800,000 Btu. The diagrammatic flow sheet (Fig. 56) and other data are all given.

The vapor withdrawn from the first effect for heating is clearly an application of Rillieux's second principle.

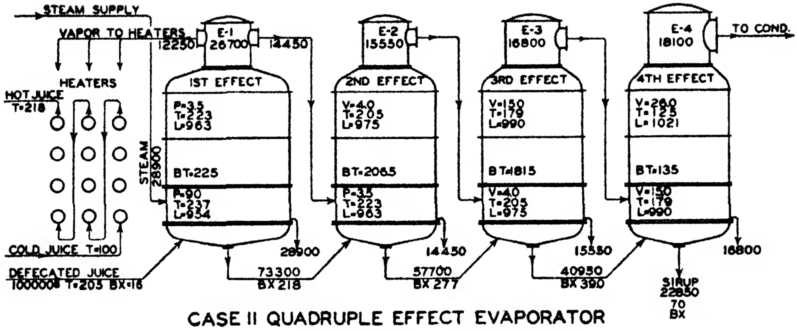
HEAT BALANCE. CASE II

Body	*Specifications*	*Btu./Hr.*	*Juice Lb./Hr.*
1.	Steam, 28,900 lb. @ 237° = 28,900 × 954 =	27,600,000	100,000 Bx. = 16.0
	Deduct for heating, 100,000 lb. × (224 − 205) =	1,900,000	
	Available for evaporation	25,700,000	
	Latent Heat @ 223° = 963, $E_1 = \frac{25,700,000}{963} =$		26,700
	Transferred to No. 2		73,300 Bx. = 21.8
2.	Vapor from No. 1	25,700,000	
	Add flash, 73,300 × (224 − 206.5) =	1,280,000	
	Available heat	26,980,000	
	Deduct for vapor heater, 12,250 lb. @ 963 =	11,800,000	
	Available for evaporation	15,180,000	
	Latent Heat @ 205° = 975, $E_2 = \frac{15,180,000}{975} =$		15,550
	Transferred to No. 3		57,750 Bx. = 27.7
3.	Vapor from No. 2	15,180,000	
	Add flash, 57,750 × (206,5 − 181,5) =	1,445,000	
	Available for evaporation	16,625,000	
	Latent heat @ 179° = 990, $E_3 = \frac{16,625,000}{990} =$		16,800
	Transferred to No. 4		40,950 Bx. = 39.0
4.	Vapor from No. 3	16,625,000	
	Add flash, 40,950 × (181.5 − 135) =	1,865,000	
	Available for evaporation	18,510,000	
	Latent Heat @ 125° = 1,021, $E_4 = \frac{18,510,000}{1,021} =$		18,100
	Sirup from fourth effect		22,850 Bx. = 70.0

$E_1 = 26,700$
$E_2 = 15,550$
$E_3 = 16,800$
$E_4 = 18,100$
$E = 77,150$

Evaporation required	77,100 lb.
Evaporation calculated	77,150 lb.
Slide rule error	50 lb.

$$\text{Evaporation per pound of steam} = \frac{77,150}{28,900} = 2.67$$



CASE II QUADRUPLE EFFECT EVAPORATOR
WITH VAPOR HEATING

FIG. 56. Diagram of Case II.

As in case I, below is the summary of the heat consumed.

Station	*Btu./Hr.*	*Steam/Hr. @ 9 lb. Gage*	*Steam/Hr. from & at 212° F.*
1. Evaporator	27,600,000	28,900	28,400
2. Heaters	None	None	None
3. Vacuum pans	11,100,000	11,640	11,450
4. Power	4,098,000	4,300	4,220
5. Radiation	2,862,000	3,000	2,950
Total	45,660,000	47,840	47,020
Corresponding boiler horsepower			1,362

Vapors to be condensed:

From evaporator (E_4)	18,100
From vacuum pans	10,300
Total	28,400

Comparison between cases I and II:

Steam consumption in case I	=	49,870
Steam consumption in case II	=	47,020
Steam saving	=	2,850
Promised by Rillieux, 12,150 × $\frac{1}{4}$	=	3,050
Rillieux error in second principle	=	+200

HEATERS AND EVAPORATORS CONSIDERED JOINTLY

199. Relation between Heaters and Evaporators. At this point, it is necessary to digress from the regular sequence of this discussion to consider the relation between heaters and evaporators and its influence on the steam economy of the factory. It is a mistake to study their performance separately, and this fact was responsible for the lapse of time between the general use of Rillieux's first principle and the second.

The juice heating load has been defined in the preceding pages, and consists of raising the temperature from an assumed 100° F. to about 218° F. In case I, this was done most simply by using steam. In case II, vapor from the first effect was substituted, with a saving of 2850 lb. of steam per hour.

200. Heat Exchanger. Looking farther into the joint operation of heaters and evaporators, the sensible heat in the sweet water[4] (condensed vapor), usually lost, can be salvaged to considerable advantage by the use of a heat exchanger, whereby the condensate is cooled by heating the raw juice in the first stage. Thus, in case I, there would be available the following:

E_1 17,300 lb. at 223° F.
E_2 18,550 lb. at 205° F.
E_3 19,900 lb. at 179° F.

Total 55,750 lb. at 201° F.

It is easily possible to cool this water to 115° F., and this would mean $55{,}750 \times (201 - 115) = 4{,}800{,}000$ Btu. This amount of heat applied to 100,000 lb. of raw juice would raise its temperature from 100.0° to 148.0°, leaving the rest to be done by some other means. This is a very useful scheme, because if sweet water is to be used for maceration it should be cooled off, at least to some extent.

201. Vapor Heating of Juice. To heat juice with vapors from the multiple effect, the most economical arrangement is what is commonly known as full compounding. Referring again to the straight quadruple, the following would be the operations:

1. Heat juice with No. 4 vapors at 125 up to 120°.
2. Heat juice with No. 3 vapors at 179 up to 174°.
3. Heat juice with No. 2 vapors at 205 up to 200°.
4. Heat juice with No. 1 vapors at 223 up to 218°.

This has been used successfully, but there are objections. First, it requires four heaters, three of which are under vacuum. Second, the complications of vapor piping, condensate drains, and air vents offer a serious obstacle. Third, it is necessary to locate the heaters close to the evaporators, which is not always possible.

202. Multiple-Effect Vapor Cells. It is not always the practice to rob the first effect of vapor to get the result desired. Very often, a separate body is used, all of whose vapors go direct to the heaters, or to some other process. In like manner, double effects are used "dead ended on the heaters." In some

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[4] The use of the term "sweet water" for condensed vapors from an evaporator is common in raw sugar factories, but the condensate rarely contains sugar. It is not to be confused with sweet waters in the refinery which are sugar-bearing wash waters. (G. P. M.)

big mills, dead-ended triple effects have been used with outstanding success. With these triples, it is customary to operate the last body under slight vacuum (3 in. to 6 in.) in order to get more capacity from the heating surface, since they usually start with 10 to 12 lb. pressure. In all these vapor cells, no attempt is made to concentrate the sirup to final density. On the contrary, the Brix should be limited to 35. The performances have been universally satisfactory and free from trouble.

CASE III. QUADRUPLE, TRIPLE-EFFECT VAPOR CELL, AND HEAT EXCHANGER

In case III the cycle is made much more economical by the use of triple-effect vapor cell, as well as a heat exchanger on sweet water, the final stage of heating being done with exhaust. This cycle is excellent where cane with very low fiber content is ground.

It is assumed that the juice is heated from 100° to 150° F. in the heat exchanger, from 150° to 205° F. in the vapor heater and from 205° to 218° F. in the exhaust heater. The heat required for this last operation will be $100{,}000 \times (218 - 205) = 1{,}300{,}000$ Btu.

The flow sheets (Fig. 57) and heat balances are all given. The main objection to this is the two evaporators instead of one. In a large house, however, this disadvantage disappears. There is a very satisfactory reduction in the steam consumption as compared with the previous cases.

Heat Exchanger Data. The hot water available for use in the heat exchanger is as follows:

E_1T = 5,210 @ 228° = 1,190,000 Btu. above 0
E_2T = 5,450 @ 218° = 1,190,000 " " "
E_3T = 5,630 @ 207° = 1,168,000 " " "
E_1Q = 13,450 @ 223° = 3,000,000 " " "
E_2Q = 14,550 @ 205° = 2,980,000 " " "
E_3Q = 15,750 @ 179° = 2,820,000 " " "

Total 60,040 @ 206° = 12,348,000 " " "

The problem is to increase the temperature of the raw juice from 100° to 150° F. by the use of this water, involving 5,000,000 Btu. The reduction in the temperature of the water would be $\frac{5{,}000{,}000}{60{,}040} = 83.2$. If all the condensate is used on the exchanger, its temperature leaving will be $206 - 83.2 = 122.8$, which allows ample margin. The differences of temperature between juice and water at the two ends of the exchanger will be:

At one end

$$206 - 150 = 56$$

At the other end

$$122.8 - 100 = 22.8$$

HEAT BALANCE. CASE III

(Triple-Effect Vapor Cell)

Body	*Specifications*	*Btu./Hr.*	*Liquor Lb./Hr.*
1.	Steam, 6,060 lb. @ 237 = 6,060 × 954 =	5,781,000	34,000 Bx. = 16
	Deduct for heating, 34,000 × (228 − 205) =	782,000	
	Available for evaporation	4,999,000	
	Latent Heat @ 228° = 960, $E_1 = \frac{4,999,000}{960} =$		5,210
	Transferred to No. 2		28,790 Bx. = 18.9
2.	Vapor from No. 1	4,999,000	
	Add flash, 28,790 × (228 − 219) =	260,000	
	Available for evaporation	5,259,000	
	Latent Heat @ 218 = 965, $E_2 = \frac{5,259,000}{965} =$		5,450
	Transferred to No. 3		23,340 Bx. = 23.3
3.	Vapor from No. 2	5,259,000	
	Add flash, 23,340 × (219 − 209) =	233,400	
	Available for evaporation	5,492,400	
	Latent Heat @ 207 = 974, $E_3 = \frac{5,492,000}{974} =$		5,630
	Transferred to quadruple		17,710 Bx. = 30.7

Original juice	100,000 lb. @ 205°
To triple-effect vapor cell	34,000 lb. @ 205°
Balance	66,000 lb. @ 205°
From triple effect	17,710 lb. @ 209°
Feed to quadruple	83,710 lb. @ 206° − Bx. = 19.1

$E_1 = 5,210$
$E_2 = 5,450$
$E_3 = 5,630$

$E = 16,290$

Evaporation per pound of steam $= \frac{16,290}{6,060} = 2.69$

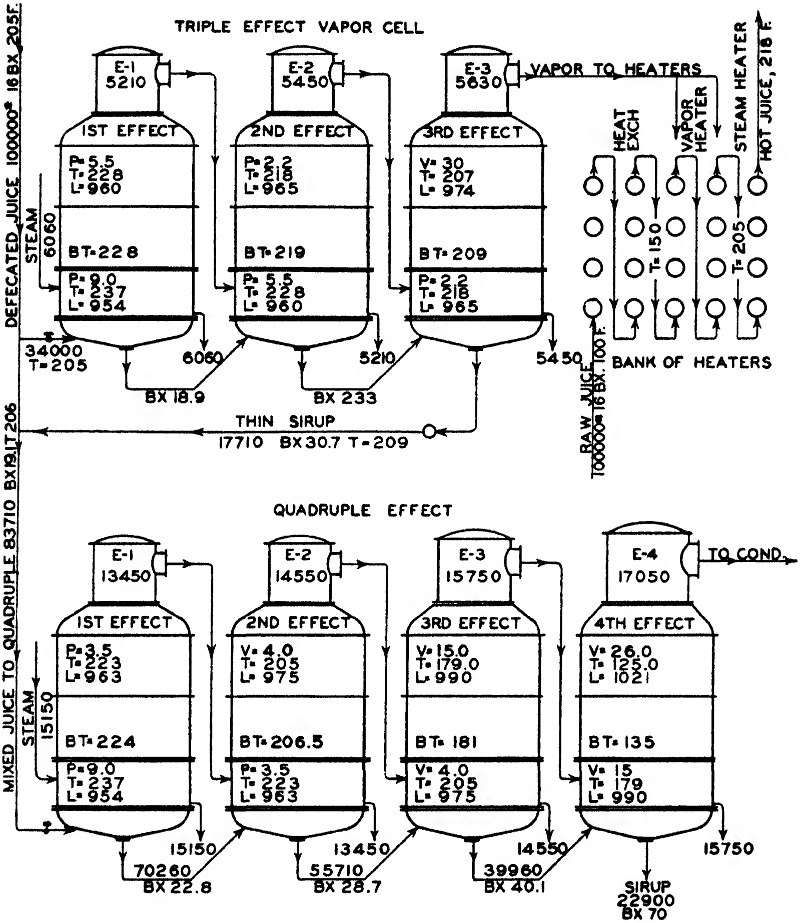
HEAT BALANCE. CASE III (QUADRUPLE)

Body	*Specifications*	*Btu./Hr.*	*Juice Lb./Hr.*
1.	Steam, 15,150 lb. @ 237° = 15,150 × 954	14,450,000	83,710 Bx. = 19.1
	Deduct for heating, 83,710 × (224 − 206) =	1,507,000	
	Available for evaporation	12,943,000	
	Latent Heat @ 223° = 963, $E_1 = \frac{12,943,000}{963} =$		13,450
	Transferred to No. 2		70,260
2.	Vapor from No. 1	12,943,000	
	Add flash, 70,260 × (224 − 206.5) =	1,230,000	
	Available for evaporation	14,173,000	
	Latent Heat @ 205° = 975, $E_2 = \frac{14,173,000}{975} =$		14,550
	Transferred to No. 3		55,710 Bx. = 28.7
3.	Vapor from No. 2	14,173,000	
	Add flash, 55,710 × (206.5 − 181) =	1,420,000	
	Available for evaporation	15,593,000	
	Latent Heat @ 179° = 990, $E_3 = \frac{15,593,000}{990} =$		15,750
	Transferred to No. 4		39,960 Bx. = 40.1
4.	Vapor from No. 3	15,593,000	
	Add flash, 39,960 × (181 − 135) =	1,840,000	
	Available for evaporation	17,433,000	
	Latent Heat @ 125 = 1,021, $E_4 = \frac{17,433,000}{1,021} =$		17,050
			22,910 Bx. = 70.0

$E_1 = 13,450$
$E_2 = 14,550$
$E_3 = 15,750$
$E_4 = 17,050$
$E = 60,800$

Evaporation required	60,810 lb.
Actual evaporation	60,800 lb.
Slide rule error	10 lb.

$$\text{Evaporation per pound of steam } \frac{60,800}{15,150} = 4.01$$



CASE III TRIPLE EFFECT VAPOR CELL
HEAT EXCHANGER VAPOR HEATERS
QUADRUPLE EFFECT

Fig. 57. Diagram of Case III.

Below is given the tabulated overall summary of the results of case III.

Station	*Btu./Hr.*	*Steam/Hr.* *@ 9 lb. Gage*	*Steam/Hr.* *from & at 212° F.*
1. Triple effect	5,781,000	6,060	5,950
2. Quadruple	14,450,000	15,150	14,900
3. Heaters	1,300,000	1,390	1,365
4. Vacuum pans	11,100,000	11,640	11,450
5. Power	4,098,000	4,300	4,220
6. Radiation	2,862,000	3,000	2,950
Total	39,591,000	41,540	40,835
Corresponding boiler horsepower			1,180

Vapors to be condensed:

From quadruple (E_4)	17,050
From vacuum pans	10,300
Total	27,350

CASE IV. MULTIPLE EVAPORATION VAPOR PANS, VAPOR HEATERS, AND HEAT EXCHANGERS

203. Vapor Pans. It is assumed in case IV that there is urgent necessity for high steam economy and hence the pans are designed to operate on low-pressure vapor. A word of warning is necessary. The load at the pan station is subject to wide fluctuations, and any cycle in which the pans are intimately tied up to the evaporators will require extra careful attention, otherwise the entire boiling house will be thrown out of equilibrium. The work of the pans will have to be rigidly scheduled to avoid vicious peaks and valleys.

204. Cycle. The cycle outlined has been in successful operation for years (at Clewiston, Florida) and consists of the following:

1. Heat exchanger on sweet water (condensed vapor).
2. Juice heating with second-effect vapors.
3. Pan operation with second-effect vapors.
4. Quadruple effect evaporation.

In order to operate successfully with this scheme, it is necessary to have exhaust steam at about 15 lb. gage. The liquor side of the second effect is at about 2 lb. gage at which its vapors are usable for the purpose contemplated.

205. Pressure Regulation. Exhaust steam must be held constant at 15 lb. If there is not enough exhaust in the mains, live steam is automatically admitted to maintain the pressure. If this exceeds 15 lb., it must be relieved to the atmosphere also automatically. If the vapor pressure in the second effect drops below 2 lb., exhaust steam is automatically admitted to boost it to that point. If, on the other hand, this pressure rises excessively, it is relieved to the atmosphere automatically. This regulation of the 15-lb. and the

2-lb. systems is the indispensable element, and it must be thoroughly reliable over the entire capacity range.

206. Pan Operation. The pans preferably should be equipped with mechanical circulation to perform satisfactorily on as low as 2 lb. steam pressure. The load at this station has been given for the example at 11,640 lb. per hour, but even with most careful operation, this demand will fluctuate as much as 6000 lb. on either side of the average, hence the precautions suggested, for the evaporator must continue to function at a substantially uniform rate irrespective of these disturbances.

207. Heat Balance. The heat balance and flow sheets are all given in full detail, and need no explanation (see Fig. 58).

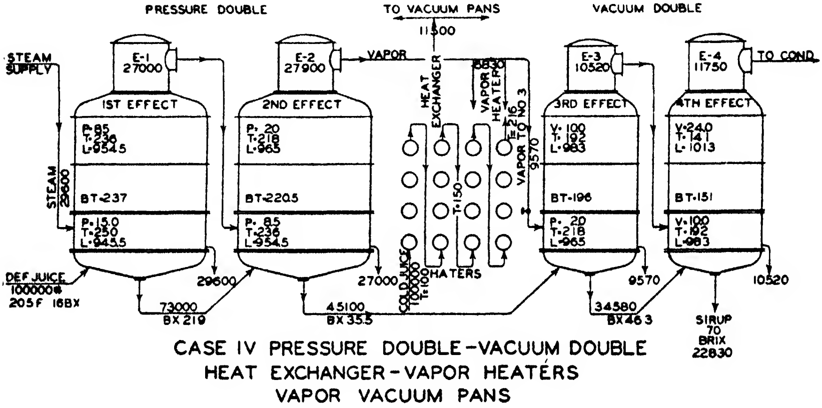


Fig. 58. Diagram of Case IV.

208. Heat Exchanger Data. The hot water available for use in the heat exchanger is as follows:

$$\begin{aligned}
E_1 &= 27{,}000 \text{ @ } 236^\circ = 6{,}370{,}000 \text{ Btu. above } 0\\
E_2 &= 27{,}900 \text{ @ } 218^\circ = 6{,}090{,}000 \quad " \quad " \quad "\\
E_3 &= \underline{10{,}520} \text{ @ } 192^\circ = \underline{2{,}020{,}000} \quad " \quad " \quad "\\
&\ 65{,}420 \text{ @ } 221^\circ = 14{,}480{,}000 \quad " \quad " \quad "
\end{aligned}$$

As in case III, it is assumed that 5,000,000 Btu. are absorbed from the condensate by the juice at this station. The change in this water is $\frac{5{,}000{,}000}{65{,}420} = 76.5^\circ$. Since the initial temperature is 221, the outgoing water will be at $221^\circ - 76.5^\circ = 144.5^\circ$ F. This is a very easy exchanger load, because the temperature difference will be:

At one end

$$144.5 - 100 = 44.5^\circ$$

At the other end

$$221.0 - 150 = 71.0^\circ$$

HEAT BALANCE. CASE IV

Body	*Specifications*	*Btu./Hr.*	*Juice Lb./Hr.*
1.	Steam, 30,600 lb. @ 250° = 30,600 × 945.5 =	28,950,000	100,000 Bx. = 16.0
	Deduct for heating 100,000 × (237 − 205)	3,200,000	
	Available for evaporation	25,750,000	
	Latent Heat @ 230° = 954,5, $E_1 = \frac{25{,}750{,}000}{954.5} =$		27,000
	Transferred to No. 2		73,000 Bx. = 21.9
2.	Vapor from No. 1	25,750,000	73,000 Bx. = 21.9
	Add flash, 73,000 × (237 − 220.5) =	1,205,000	
	Available for evaporation	26,955,000	
	Latent Heat @ 218° = 965, $E_2 = \frac{26{,}955{,}000}{965} =$		27,900
	Transferred to No. 3		45,100 Bx. = 35.5
3.	Vapor from No. 2	26,955,000	
	Add flash 45,100 × (220.5 − 196) =	1,105,000	
	Available heat	28,060,000	
	Deduct for heaters and pans	17,700,000	
	Available for evaporation	10,360,000	
	Latent Heat @ 192 = 983, $E_3 = \frac{10{,}360{,}000}{983} =$		10,520
	Transferred to No. 4		34,580 Bx. = 46.3
4.	Vapor from No. 3	10,360,000	
	Add flash, 34,580 × (196 − 151) =	1,555,000	
	Available for evaporation	11,915,000	
	Latent Heat @ 141 = 1,013, $E_4 = \frac{11{,}915{,}000}{1{,}013} =$		11,750
	Sirup from No. 4		22,830 Bx. = 70.0

$E_1 = 27{,}000$
$E_2 = 27{,}900$
$E_3 = 10{,}520$
$E_4 = 11{,}750$

$E = 77{,}170$

Evaporation required	77,100 lb.
Actual evaporation	77,170 lb.
Slide rule error	70 lb.

$$\text{Evaporation per pound steam} = \frac{77{,}170}{30{,}600} = 2.52$$

Following is the summary of the heat requirements of case IV:

Station	*Btu./Hr.*	*Steam/Hr. @ 15 lb. Gage*	*Steam/Hr. from & at 212° F.*
1. Evaporator	28,950,000	30,600	29,800
2. Heaters	None	None	None
3. Vacuum pans	None	None	None
4. Power	4,098,000	4,330	4,220
5. Radiation	2,862,000	3,025	2,950
Total	35,910,000	37,955	36,970
Corresponding boiler horsepower =			1,070

Vapors to be condensed:

From evaporator (E_4)	11,750
From vacuum pans	10,300
Total	22,050

The economy of this arrangement is very remarkable indeed. It has been used in connection with the refining of raw sugars at the mill itself as fast as they are made, and it is very evident that with normal fiber content in cane there is enough fuel available for all operations.

The scheme is not complicated and expensive, but it requires more careful supervision than the conventional arrangement in the ordinary sugar mill. Since the pans operate with very low steam temperature the quality of sugar is much improved, and danger of heat injury is at a minimum.

Summary of Cases Studied. Below is given an interesting and instructive summary of the various cases as studied above.

Cases	Steam/Hr. from and at 212° F.	Boiler Horsepower
I. Straight quadruple	49,870	1,445
II. Quadruple and No. 1 vapor heating	47,020	1,362
III. Quadruple, triple vapor cell, and heat exchanger	40,833	1,180
IV. Pressure double effect, vacuum double effect, vapor heaters, heat exchangers, and vapor pans	36,970	1,070

GENERAL COMMENTS

209. Bagasse Fuel. In ordinary sugar factory reports, it is customary to give the following information about bagasse:

1. Fiber in cane	Example	10.0%
2. Moisture in bagasse	Example	48.0%
3. Sucrose in bagasse	Example	3.0%
4. Purity last mill juice	Example	70.0%

A considerable error is involved in assuming that fiber in cane is the same as solids in bagasse. To be more specific, take the cases considered, in which 50 tons of cane per hour were ground.

Moisture in bagasse	48.0%
Solids, non-fiber, in bagasse	4.3%
Total, exclusive of fiber	52.3%
Fiber to make up 100%	47.7%

$$\text{Weight of fiber alone} = 50 \times 2,000 \times 0.10 = 10,000 \text{ lb.}$$

$$\text{Weight of bagasse/hr.} = \frac{10,000}{0.477} = 20,964 \text{ lb.}$$

With good boilers an efficiency of 66⅔ per cent can be attained, giving 2.55 lb. of steam from and at 212° F. per pound of bagasse, and hence for this example, 53,458 lb. per hour.

210. Other Cycles. There are many combinations of evaporators, heaters, and pans used other than the four cases outlined in the preceding discussion. It is felt that with the elaboration provided as a guide, the reader should be able to work them out in detail if he wishes. In particular, one specific case should be mentioned.

211. The Pauly or Pre-Evaporator. In many mills, the quantity of exhaust steam is insufficient to supply the demands of the boiling house, and this deficiency is made up by feeding live steam into the exhaust. Where the quantity of this live steam is considerable and where its use is uninterrupted, it provides an opportunity to effect additional steam savings by interposing an evaporator between the live steam system and the exhaust system, the juice vapors entering the exhaust mains. This is the so-called Pauly or pre-evaporator. In a few plants, the cycle has been supplemented with thermo-compressors, by means of which juice vapors are withdrawn from the liquor space and compressed back to the pressure in the steam chest, thus increasing the economy.

The successful use of the Pauly in the cane sugar industry seems to be limited to large factories where it can be kept in operation continuously. The frequent starting and stopping of any evaporator complicates operation, tends to promote fouling, and involves serious heat losses.

Another objection to the Pauly is the high temperatures prevailing, which often result in additional coagulation of organic matter with rapid fouling and color increment. On the whole, better results can be secured with more desirable combinations. In the beet sugar industry, where there are fewer heat-sensitive compounds, the Pauly has been widely applied.

212. Limits of Steam Economies. It should also be emphasized that it is *absolutely useless to cut down the steam consumption of the boiling house to a point lower than the amount of exhaust steam produced by the prime* *movers*. The excess of exhaust is lost by blowing to the atmosphere, vitiating the entire project.

213. Condenser Loads. Another interesting feature about the use of economical steam cycles that is frequently overlooked is that they are always accompanied by a corresponding reduction in the amount of vapors to the condensers, and hence an equivalent reduction in the demand for injection water. Thus, referring to the examples worked out in the preceding pages, it will be seen that the vapors to be condensed are as follows:

Case I	Straight quadruple	31,600
Case II	Quadruple with No. 1 vapor heating	28,400
Case III	Quadruple, triple vapor cell, and heat exchanger	27,350
Case IV	Pressure double effect, vacuum double effect, vapor heating, heat exchanger, and vapor pans	22,050

214. Evaporator Ratings. The amount of heating surface required to perform the work outlined should theoretically be in the form of a coefficient of heat transmission but it is much simpler to give these capacities in pounds of water evaporated per square foot per hour under standard operating conditions. Among most manufacturers these ratings have been standardized as follows: with steam at not less than 5 lb. gage, vacuum at not less than 26 in., sirup at not over 65° to 70° Brix, and the evaporators normally clean, the rate of evaporation per square foot per hour is:

For triple effects	10.5 lb.
For quadruple effects	8 lb.
For quintuple effects	6 lb.
For vapor cells	6 lb.
For Pauly or pre-evaporator	6–8 lb.

215. Heating Surface for Quads. The amount of evaporation in the standard quadruple in the example is 77,100 lb. Allowing 8 lb. per sq. ft. per hour, this would require 9630 sq. ft. For practical purposes, it would be 10,000 sq. ft. The total surface would be the same whether vapor heating is used or not. In that event, however, the distribution of surface would not be equal, but more or less in proportion to the condensate in each steam chest. Referring to case II, consider the following data:

		Calculated H. S.	Actual H. S.
Condensate in No. 1	28,900	3,830	4,000
Condensate in No. 2	14,450	1,910	2,000
Condensate in No. 3	15,500	2,050	2,000
Condensate in No. 4	16,800	2,210	2,000
Total	75,650	10,000	10,000

216. Heating Surface Defined. The correct method of calculating the heating surface of pans, evaporators, and heaters was established in the Evaporator Test Code published by the American Society of Mechanical Engineers in 1921, and endorsed by the American Institute of Chemical Engineers. Article 29 of this code is as follows:

> Heating surface will be calculated as the surface in contact with steam or other heating medium.

Properties of Saturated Steam at Different Vacua and Pressures

Inches Vacuum *	Temp. °F.	Cu. ft./ Lb.	Latent Heat Btu.	Vacuum or Pounds	Temp. °F.	Cu. ft./ Lb.	Latent Heat Btu.
28.0	101.15	339.6	1035	10 in.	192.23	39.21	983
27.5	108.70	275.2	1030	9 “	194.52	37.4	981
27.0	115.06	231.9	1027	8 “	196.73	35.8	980
26.5	120.55	200.2	1024	7 “	198.87	34.3	978
26.0	125.38	176.7	1021	6 “	200.94	33.0	977
25.5	129.75	158.1	1019	5 “	202.92	31.8	976
25	133.77	143.0	1017	4 “	204.85	30.6	975
24	140.64	129.0	1013	3 “	206.71	29.5	974
23	146.78	104.5	1009	2 “	208.52	28.6	973
22	152.16	92.3	1006	1 “	210.28	27.7	972
21	157.00	82.6	1003	0 “	212.00	26.8	970
20	161.42	74.8	1001	1 lb.	215.3	25.2	968
19	165.42	68.5	998	2 “	218.5	23.8	965
18	169.14	63.1	996	3 “	221.5	22.5	964
17	172.63	58.6	994	4 “	224.4	21.4	962
16	175.93	54.5	992	5 “	227.2	20.4	961
15	179.03	51.2	990	6 “	229.8	19.5	959
14	181.92	49.0	989	7 “	232.4	18.6	957
13	184.68	45.5	987	8 “	234.8	17.8	955
12	187.31	43.2	985	9 “	237.1	17.1	954
11	189.83	41.1	984	10 “	239.4	16.5	953

* At sea level.

References

Webre and Robinson, *Evaporation.*
McAdams, *Heat Transmission.*
W. L. Badger, *Heat Transfer and Evaporation.*
E. Hausbrand, *Evaporating and Condensing Apparatus.*
Walker, Lewis, and McAdams, *Principles of Chemical Engineering.*
J. C. Olsen, *Unit Processes and Principles of Chemical Engineering.*

CLEANING EVAPORATORS: INSULATION, ETC.

(G.P.M.)

217. Evaporator Scale. The scale which forms on the tubes of the evaporators is a very poor conductor of heat. Its composition varies somewhat with that of the cane, and in some localities it forms very much faster than in others. The tubes of the first body, which always contains thin juice, scale little compared with those of the other bodies. The last body of a multiple effect contains the thickest and most obstinate scale.

The following analyses of the scales indicate their composition in the different pans of a quadruple effect: [5]

	First Pan, Per Cent	Second Pan, Per Cent	Third Pan, Per Cent	Fourth Pan, Per Cent
Calcium phosphate	57.85	56.98	15.02	7.49
" sulfate	2.02	1.92	0.54	1.65
" carbonate	3.25	4.68	19.55	9.93
" silicate	7.86	13.31	0.71	7.02
" oxalate	.....	.....	11.32	11.27
Iron oxide	2.03	1.53	2.31	2.58
Silica	7.79	7.43	39.26	54.34
Combustible matter	20.37	13.41	11.04	5.08

These analyses are corroborated by Bogtstra [6] who also found calcium phosphate in predominance in the first bodies and silica in the last body.

218. Cleaning Evaporator Tubes. The tubes of the evaporator must be cleaned at regular intervals (normally once a week) to remove the accumulation of scale, which greatly reduces the efficiency of the effects (see Sec. 184). Common practice is to boil for several hours a volume of caustic soda or soda ash solution or both, sufficient to cover the tubes, after which the alkali is run back to an iron storage tank. The effects are then washed with water and boiled with dilute hydrochloric acid which is discarded after use. At the end of the manufacturing season it is usually necessary to give the tubes a thorough scraping.

The amount of chemicals used and the time of boiling were found by Keller [7] to vary widely in Louisiana. Caustic soda and soda ash are generally used together (the soda ash aids in dissolving the silicate scale in the last body), in proportions of two-thirds to three-quarters of caustic soda and one-third to one-quarter of soda ash. The concentration of alkali ranged from 1.5 to 24 per cent with an average of about 4 per cent in the greater number of plants. Most of the factories boiled the caustic 3 to 6 hours. The hydrochloric acid solution ranged from 1 to 3 per cent by weight and was generally boiled 1.5 to 2.5 hours.

The boiling out with alkali and acid is always done at atmospheric pressure with a vent to the roof. This gives a higher reaction temperature and prevents corrosive gases from entering the next calandria.

In Hawaii a method of spraying the tubes with caustic soda, soda ash, or a mixture of both has had favorable reports.[8] Soda solution of 30° Baumé is pumped into the bodies of the evaporator through ordinary garden spray

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[5] Prinsen-Geerligs, *Kobus Archief,* 1900, 694.

[6] *Archief,* 1932, p. 911; *Intern. Sugar J.,* December, 1932, p. 462.

[7] *Sugar J.,* September, 1940.

[8] N. E. Wright, *The Planter,* 79, No. 23 (1927).

nozzles placed so that the solution falls in a fine rain over the tubes. A small amount of steam in the calandria serves to keep the soda hot. The soda drains back to the supply tank and the spraying continues for about two hours, after which the effect is washed with water. Acid is used subsequently if needed.

A similar idea using a revolving spray is described by Clarke[9] in Hawaii and by Feringa[10] in Java. According to these writers, there is a saving in the quantity of caustic soda solution (400 gal. of 25°–35° Baumé solution as against 10,000 gal. by the ordinary method) and reduced steam consumption as well as a time saving. This practice of spraying tubes with strong alkali has also been found efficacious in Louisiana and Cuba.

Vegetable products, sold under various names (Algo, Algaloid, etc.) have been reported in Australia as very effective in boiling out evaporators, even when used in dilute solutions (0.05 per cent).[11] These preparations soften the scale rather than dissolve it, and it is then readily washed from the tubes without scraping.

Cleaning evaporator tubes with fermented molasses has been practiced in Queensland (Australia) and other countries but not to any extent in the West Indies and Louisiana. Molasses of 18° Brix is allowed to ferment spontaneously at 25°–30° C., the pH falling from 6.0 to 3.9.[12] In practice this ferment is boiled in the evaporator bodies following the juice on liquidation, and then is allowed to stand sixteen hours, after which the tubes are sprayed with a fresh supply of ferment. According to Chappell[13] the results are such that only a light brushing once during a season is necessary. The use of organic acids formed by decaying mangrove fruit has also been suggested, but no general adoption seems to have resulted.

The steam side of evaporator tubes also fouls with soot and oil from exhaust steam and requires cleaning at the end of each season. A quantity of kerosene is added to the steam drum of the calandria, and this is slowly floated upwards by adding water until the drum is full. This is repeated by lowering and raising the level several times. Then the caustic soda solution is admitted to the drum and boiled. The use of carbon tetrachloride for this purpose has also been advocated, but it should be used with care because of the toxic properties of the fumes.

219. Insulation of Evaporators and Vacuum Pans. Heat losses of considerable magnitude will occur unless the bodies of evaporators and vacuum pans are insulated. The general practice is to use either hair felt an inch or more thick covered with wood lagging, or asbestos-magnesia blocks 1½ in. to 2 in. thick held in place with wire netting and smoothed over with asbestos cement. Kieselguhr blocks have also been used for insulation, and an eco-

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[9] *Rept. Assoc. Hawaiian Sugar Tech.*, 1929.

[10] *Intern. Sugar J.*, March, 1931, p. 129.

[11] *Intern. Sugar J.*, April, 1931, p. 17.

[12] Cameron, *Intern. Sugar J.*, November, 1933, p. 422.

[13] *Proc. Queensland Soc. Sugar Cane Tech.*, 1937, p. 33.

nomical insulating material has been made by mixing dried refinery press cake in which there is a high percentage of kieselguhr with sufficient cement to permit molding.

Sandera[14] gives an extensive study of the value of insulation of evaporators, pans, and pipelines (live steam, exhaust steam, and hot juice) and shows that a loss of 2.2 per cent of the total heat consumption may occur if 10 per cent of all surfaces are not insulated. Naturally, the hotter the surface, the greater the need for insulation. Sandera also investigated the value of aluminum paint for covering heating surfaces and showed that radiation losses were 10 to 25 per cent lower for both lagged and naked surfaces when the surfaces were covered with this type of paint. The insulating value of aluminum paint has also been proved in many studies by American paint manufacturers. This is of interest because of the extensive use of aluminum in recent years for painting all types of surfaces in American factories and refineries. (See also Lyle, *Technology for Sugar Refinery Workers*, London, 1941, p. 102.)

220. Settling the Sirup. After the concentration of the juice to a sirup in the multiple-effect evaporators, it is pumped to combined settling and storage tanks. A considerable quantity of impurities that were soluble in the thin juice are insoluble in the sirup. As has been explained, part of these impurities deposit themselves upon the heating surface of the evaporator, forming a hard scale. Those which remain in suspension were formerly removed by settling and decantation in sirup-settling tanks.

Large modern factories do not have tank space to permit settling the sirup, and any removal of the sediment is largely incidental to cleaning the tanks. In white sugar manufacture, particularly in sulfitation plants in Louisiana, the common practice of settling and decanting the sirup (see p. 109) improves both the sugar and the molasses.

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[14] *Intern. Sugar J.*, June, 1935, p. 227.

CHAPTER 10

PRESERVATION OF THE JUICE AND SIRUP DURING SHUTDOWNS

221. Storing of Juice and Sirup during Shutdowns. It may be necessary in the event of the breakage of the machinery or delay from other causes to store juice and sirup several hours, or even days. Sirup will usually keep under tropical conditions from thirty-six to forty-eight hours without the use of a preservative, provided that it is at least 54.3° Brix (30° Baumé), is stored in clean tanks, not in the vicinity of fermenting sugar solutions, and the pH is 6.8 or above. In modern factories, sirup is evaporated to higher densities (60° to 70° Brix) which still further improves the keeping qualities. Raw juice will deteriorate in a very short time without a preservative, and in the event of a shutdown all juice should be limed, heated, and sent to the defecators. The method of manufacture influences the keeping qualities of juices and sirup, those treated by the sulfur processes remaining sweet much longer than those prepared with lime only.

The length of time which defecated juice will keep without preservatives depends on the temperature at which it is stored and the reaction of the juice. McAllep[1] reports that juice limed to a pH between 7.6 and 8.3 (tested after heating) and held at a temperature not higher than 180° has frequently been kept for twenty-two hours with no acidity developing sufficient to cause inversion of sucrose. McAllep[2] also reports that H. S. Walker kept juices over a twenty-four or thirty-six hour shutdown without preservatives by reducing the temperature of the stored juices to 180° F. and not permitting it to fall below 160° F., below which point bacterial action will develop.

222. Use of Preservatives. During shutdowns the use of preservatives, particularly formalin, was formerly quite general, but in the past decade the use has been discontinued almost entirely in Louisiana, and largely in Cuba except in extreme cases. It has been found preferable to evaporate all thin materials to the sirup stage, or if this is not possible to maintain the temperature of clarified juice between 160° and 180° F. as suggested by McAllep and Walker.

Formaldehyde. Formalin, which is a 40 per cent solution of formaldehyde in water, is a cheap and effective preservative for juice and sirups during shutdowns. It was first advocated by Spencer in Cuba in 1903 and was general practice until recent years as explained above.

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[1] *The Planter,* **72**, No. 6 (1924).

[2] *Loc. cit.*

The proportions recommended are as follows. Clarified juice, made by the sulfur process, may be stored in clean tanks for thirty-six hours and probably longer by the addition of 3 cc. of a 40 per cent solution of formaldehyde per cubic foot of juice (about 1 part formalin to 10,000 parts of juice). Juice clarified by the ordinary process requires considerably more than this quantity of formaldehyde, usually from 6 to 8 cc. (from 1:5000 to 1:4000).

Owen[3] recommends the use of formaldehyde in the above proportions when milling deteriorated juices from frozen canes, as such juices may infect the whole plant and the infection may persist even when milling is resumed on normal windrowed cane.

Spencer preserved 30° Baumé sirup during a period of seven days by the addition of 6 cc. of 40 per cent formaldehyde solution per cubic foot (1:5000). Possibly less formaldehyde would have served, but it was not deemed advisable to incur risk. The capacity of the storage tanks was 5000 gal. each.

Without removing the scum or mud, juice may be preserved twenty-four hours in the defecators by the use of formaldehyde. The measured quantity of the preservative should be mixed with the juice immediately on starting to fill the defecator. With closed separators of the Dorr type the formaldehyde should be added to the pump tank and pumped through the heaters to insure thorough mixing.

Spencer found that there was always a slight deterioration of juice even in the presence of formaldehyde. This was undoubtedly due to the high temperatures at which the juices were carried and can be largely prevented by maintaining a temperature not higher than 180° F. on any juice carried over, as reported by McAllep and Walker in the work cited above. Insulation with dry bagasse of tanks used for storage of juices to keep the temperature from dropping below 160° F. is advocated by McAllep. Under these conditions the use of the preservative is of doubtful value.

It has been shown by Norris[4] and Meade[5] and corroborated by Owen bacteriologically that formaldehyde in too small amounts (say 1:50,000 or 1:100,000) will actually increase the deterioration due to micro-organisms, so no attempt should be made to economize on the preservative. The cost of formaldehyde in barrels is so small that it would usually be advisable to use it in even larger quantities than is indicated above. Most of it disappears from the juice in the evaporation. However, because some distillers have objected to molasses from factories in which formalin was freely used, this objection to its use has been advanced.

Other Preservatives. Other disinfectants such as hypochlorite of lime ("chloride of lime") and chlorine have been advocated as juice preservatives and as sanitary agents around mills, gutters, etc.[6] These materials may be

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[3] *Sugar Bull.* 16, No. 8 (1938).

[4] *Hawaiian Bull.,* No. 23.

[5] *Proc. 8th Intern. Congr. Applied Chem.* (1912).

[6] Mascaró, *15th Annual Proc. Assoc. Cane Sugar Cuba Tech.* (1941), p. 169.

of value in extreme cases but in general it appears that the best practice is to avoid chemical disinfectants in the process itself and to depend on the proper reaction, concentration, and temperature to prevent deterioration of materials. Hosing down mills with hot water at frequent intervals, the avoidance of souring or fermenting residues, scrupulous cleanliness throughout the process, and proper temperature control will all give better results generally than dependence on antiseptics.

CHAPTER 11

CRYSTALLIZATION OF THE SUGAR[1]

223. The Vacuum Pan Station. The function of the vacuum pan is to develop and grow sugar crystals from the sirup or molasses fed to it during operation. The desirable qualities of raw sugar are discussed in Chapter 15 and are influenced to a great extent by the design and operation of the vacuum pans. Sirup arriving from the evaporators is usually 60° to 65° Brix, and even 70° Brix, thus permitting the maximum use of steam in multiple effect, with corresponding fuel economy. (See Sec. 257.)

224. Types of Vacuum Pans. There are in general use two types of vacuum pans, coil and calandria. The design and accessories are similar, the difference being in arrangement of the heating surface.

Coil Pans. The coil pan was formerly in exclusive use. It is a vertical single-effect evaporator operating under vacuum (see Fig. 59). The heating surface comprises six or seven copper coils to which live steam at 50 to 90 lb. pressure is admitted. These coils are independent, and usually are built of 4-in. copper tubing in the form of a conical spiral, high at the outer periphery and low in the center, conforming to the shape of the bottom and providing good drainage for the condensed steam. The coils are supported and fastened on braces with appropriate saddle pieces to avoid bruising the copper as a result of vibration, expansion and contraction.

The following are the characteristics of a good coil pan:

a. The length of the coils should not be greater than 100 diameters, preferably less. This means that multiple-strand coils must be used.

b. The coils should be properly spaced to insure good circulation. The distance from one turn of the spiral to the next should be at least 3 in. in bottom coils and 5 in. in the top coils, even if the heating surface has to be reduced.

c. Large space, at least one-fourth the diameter of the pan, should be left open in the center (downtake) for free downward return of the massecuite.

d. Vapor liberation space should be ample above maximum level of massecuite.

e. A heating surface of 1 sq. ft. per cu. ft. massecuite is usual in raw house work with 1.5 sq. ft. per cu. ft. for refining.

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[1] This chapter was written in collaboration with Alfred L. Webre, M.E., Consulting Engineer with the United States Pipe and Foundry Company, Burlington, N. J., and the writer acknowledges his indebtedness for the valuable assistance of both Mr. Webre and his Company. (G. P. M.)

f. A feed valve in the distributing feed pipe in the bottom of the pan should be so designed as to insure proper mixture of feed liquor or sirup with massecuite. This is *most important.*

The following fittings and auxiliaries are common to both coil and calandria pans:

A large discharge valve at the bottom whose diameter is from one-eighth to one-sixth that of the pan.

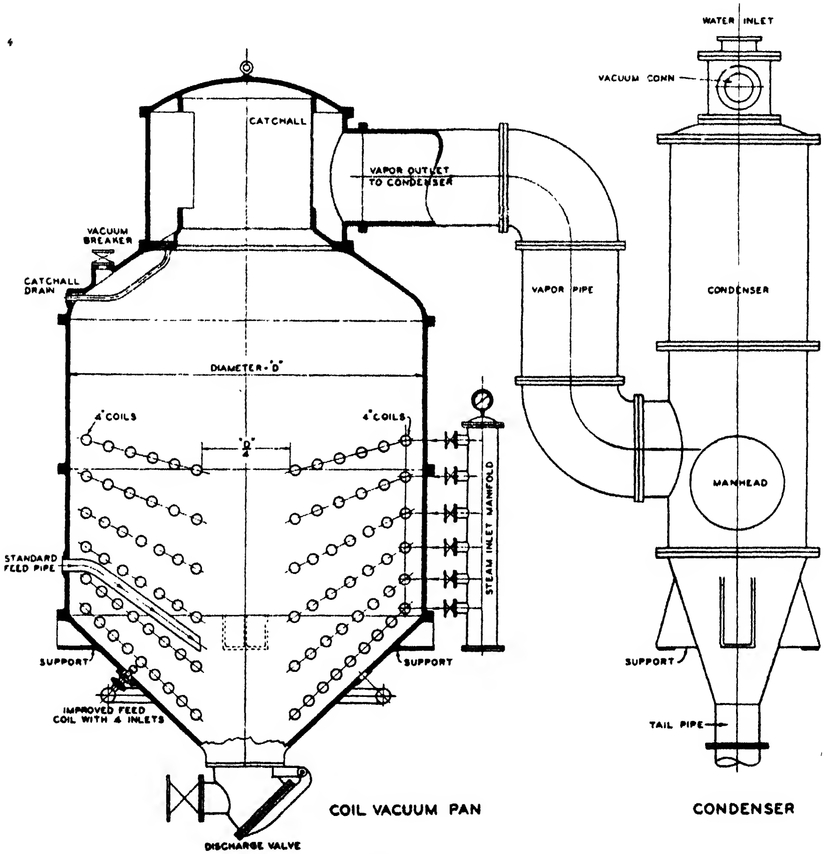

Fig. 59. Coil Pan.

A saveall or catchall at the top of the pan which is either in the dome of the pan itself or between the dome and the condenser. This serves the purpose of slowing down the vapors, thereby reducing entrainment of sirups to a minimum. (See Sec. 169.)

A condenser which may be one of several types, and is supplied with cold water for the purpose of condensing the vapors, thus maintaining vacuum (See Sec. 170.)

A vacuum pump to which the condenser is connected by a 6-in. or 8-in. pipe. The pump is used to raise vacuum at the beginning of the strike and to remove non-condensable vapors during boiling. (See Sec. 171.)

A vacuum breaker for use just before discharging the pan. There are sight glasses or lunettes through which to observe the progress of the boiling; a proof stick for taking samples; vacuum gage; thermometer, and steam gage. The apparatus is so arranged that the pan boiler may vary the temperature of the boiling liquor by increasing or decreasing the vacuum through adjustment of the injection water.

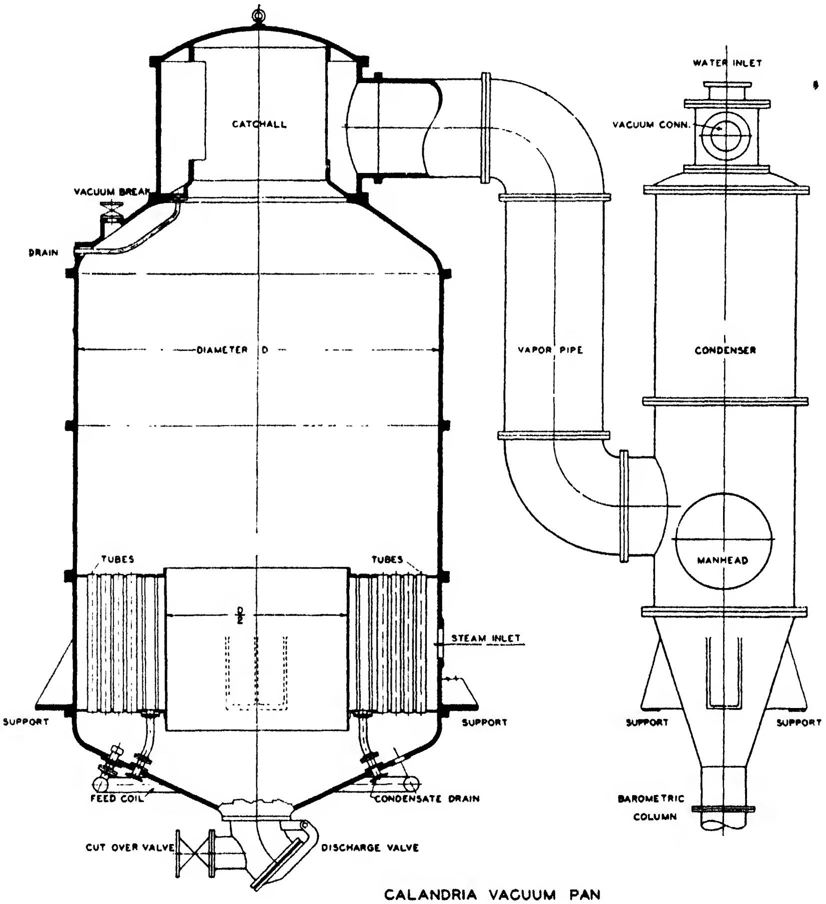

Fig. 60. Calandria Pan.

Calandria Pan. The calandria pan, which is gradually displacing the coil pan, is a vertical evaporator with tubes about 4 in. in diameter, and from 42 in. to 60 in. high. (See Fig. 60.) In the center is a large circulating pipe or downtake, and in the past its restricted size has prevented proper circulation. Without a mechanical circulator (see Sec. 230) this downtake should be *one-half the pan* diameter; otherwise, the performance will be greatly inferior to that of coil pans.

With natural circulation, it is not practical to carry the level of the strike over 6 ft. above the calandria. The ratio of heating surface to capacity is greater than for coil pans, running to 2 sq. ft. per cubic foot of strike.

225. Advantages and Disadvantages of Coil and Calandria Pans. Webre [2] has recently given figures which show that (with natural circulation) the coil pan has much better circulation than the calandria pan, particularly at the end of the strike when the temperatures are high. Since the color increment is proportional to the product of this temperature by the time element, it can be seen that the advantage is unquestionable, because the contact temperature is lower and the time element is shorter.

The average supersaturation for the coil pan is higher than for the calandria pan, and hence the growth of the crystals should be faster, and the danger of fines and conglomerates incident to unsaturation much less.

A detailed description of factors affecting circulation in coil pans, with suggestions for improvement in design based on work in Queensland, are given by Smith.[3] Vertical and horizontal pitch of coils, "dish" of the coils, clearance at side walls, removal of obstructions from center wells, feed distribution, and "circulation ratio" were some of the points studied.

The advantage of the calandria pan is that it operates with low-pressure steam, or juice vapors from the evaporator, thus permitting large economies. The upkeep is low as compared with coils, their drain pipes, and supports, which are subject to breakage by expansion, contraction, and the heavy stresses imposed by the ebullition of massecuite.

A defect of design in some calandria pans is the cone bottom with a tendency to form an idle pocket, which does not mix with the remainder of the mass. This is readily overcome by extending the downtake to within 15 in. of the bottom. A recent design is the "saucer bottom" which is streamlined to aid circulation.[4] The saucer type bottom with the 20° angle bottom shown in Fig. 61 reduces the "graining volume" of the pan, i.e., the volume necessary to cover the calandria, to 28 per cent as compared to 33 per cent in the older designs with deeper cones.

Some calandrias have conical tube sheets. This provides drainage and avoids the downtake extension. Other designs have a flat upper tube sheet, and a conical lower one to eliminate the dead pocket. These conical arrangements are more difficult to build and repair. Some pans have floating calandrias and the express pan had inclined tube sheets as well. The objection to this type is poor circulation, part of the down current coming through the small center downtake, and the rest in the annular space between the drum

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[2] Address before *Am. Chem. Soc.*, Detroit, April, 1943.

[3] *Intern. Sugar J.*, March, 1938, p. 101; April, 1938, p. 140.

[4] Walter E. Smith, *Intern. Sugar J.*, January, 1935, p. 20.

and the shell of the pan. Conical and floating calandrias are not common, the general type being the flat calandria first described.

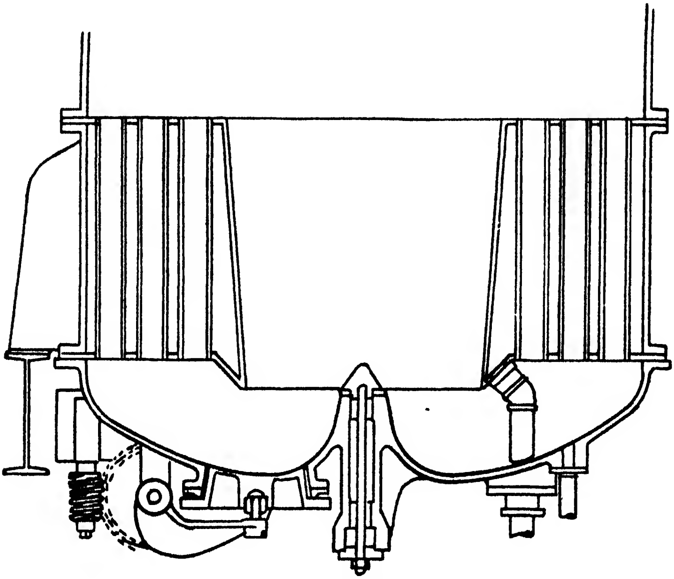

FIG. 61. Saucer-Bottom Pan.

226. Surface Condenser. The use of surface condensers in the vapor pipe before the regular condenser is not uncommon in refinery pans. Such a surface condenser serves as a catchall to prevent entrainment and also conserves heat which is otherwise lost in the condenser water.[5] The plan has been of greatest value where fresh water is scarce.

227. Definitions Used in Pan Boiling. The concentrated juice from the evaporators is called sirup or meladura (Spanish). The concentrated mass of crystals and molasses form the massecuite, and each boiling is called a strike. When a portion of a strike is removed from the pan, and the remainder is left as a base or nucleus upon which to boil another strike, the portion so left is termed a cut, or footing.

Where two or more pans are worked side by side they are often connected to one another by large pipes, with suitable valves, for transferring massecuite in either direction. These are called cut-over pipes.

When a grained strike reaches a certain state, diluted molasses may be drawn into the pan instead of sirup, and it is then said to be "boiled in" or "boiled in on grain sugar."

When the liquor in the pan has been concentrated to a certain density and has passed saturation, it is said to have reached "proof." The proof may be

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[5] *Intern. Sugar J.*, March, 1931, p. 101 (abs.).

"strong" or "weak" as the liquor is of greater or less density and the massecuite is said to be "tight" or "loose."

PAN CIRCULATION

NATURAL AND MECHANICAL

228. Importance. The most important characteristics of vacuum pan operation which determine the quality of the sugar produced are circulation and temperature conditions. A thorough investigation of these features was made by Webre. Below is given the gist of the findings and conclusions.

a. The first problem was the speed of massecuite traveling through the tubes of a well-operated calandria pan with a downtake one-half the diameter. This velocity calculated from the observations on a *C* strike was:

First hour	1.53 ft.	per	second
Second hour	0.63 "	"	"
Third hour	0.15 "	"	"
Fourth hour	0.03 "	"	"
Fifth hour	0.02 "	"	"
Sixth hour	0.01 "	"	"

b. Then experiments were carried on to determine the temperature conditions of the massecuite passing through the tubes at such slow velocities. To accomplish this, a sensitive thermometer was mounted in the side of the pan above the upper tube sheet, and in line with the center of a tube. Into the top of the tube was inserted a ferrule about 6 in. high, with a hole in its side for the free passage of the thermometer in its proof stick mounting, providing readings of the variations of temperature between the center of the tube and at its inner periphery.

The results were startling. Even at the beginning of the strike, there were observable differences of 4° F. between the two points in question, which gradually increased as the circulation slowed down, reaching a peak of 30°.

It was concluded that in the tubes of a calandria pan or at the surfaces of a coil, in order for ebullition to take place, temperatures must obtain which correspond to the vacuum lowered by the superposed hydrostatic head because of the weight of the massecuite above the point under observation. To this must be added the boiling point rise at the purity and density in the pan. Figure 62 emphasizes this. Ordinates show temperatures both Fahrenheit and centigrade but plotted in the reverse order. Abscissas show vacua in inches, as well as absolute pressures in both inches and centimeters of mercury. The plot is based on 100 purity.

The first curve shows saturation vapor temperature. The second curve shows the boiling point of massecuite at the surface of ebullition. Each successive line shows this boiling point corrected for the reduced vacuum due to the superposed pressure caused by 1 ft. of additional massecuite. As this temperature is increased, a point is soon reached where the massecuite, instead of carrying a normal supersaturation of 1.20, will revert to saturation and recede below it. The dotted line shows the critical point of saturation, above which massecuite is supersaturated and below which it is unsaturated.

When the surface is submerged under about five feet of massecuite, boiling can take place only by forcing the temperature so high that the solution be-

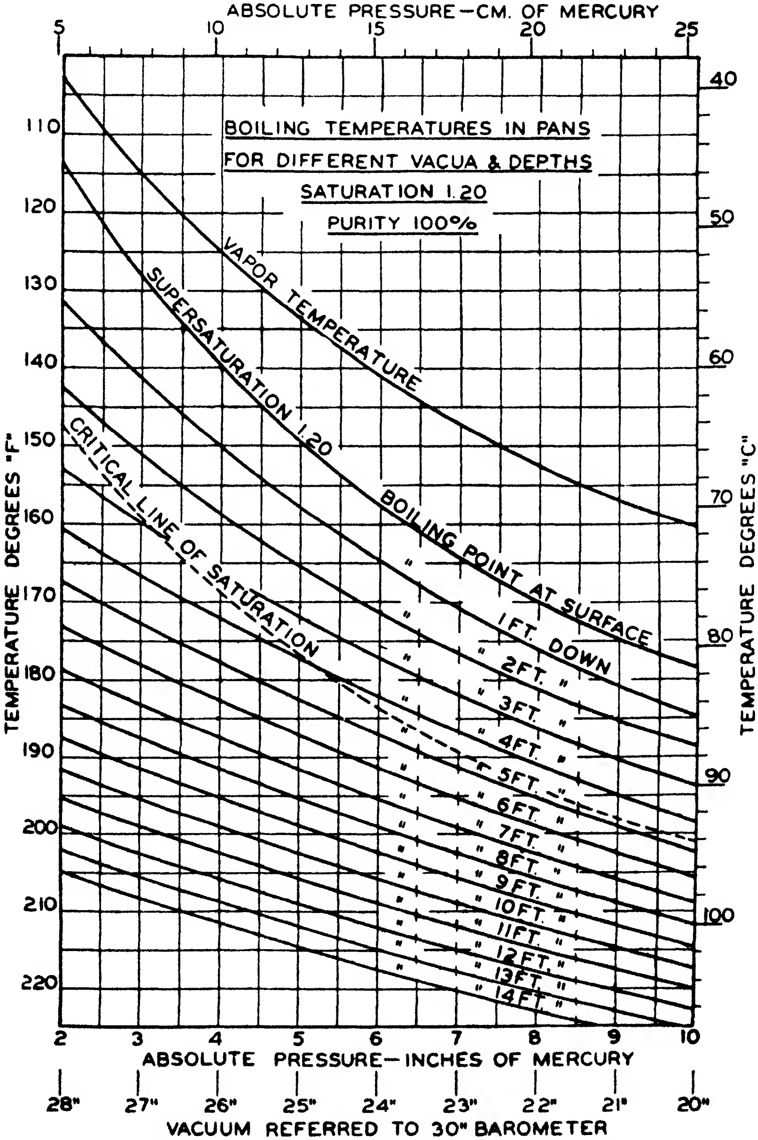

Fig. 62. Boiling Temperatures in Pans for Different Depths.

comes unsaturated, thus dissolving part of the crystals. It is not surprising that control of pan operations is difficult, and remarkable that it has been possible to do good work under the conditions imposed. The reason is in the time element required for redissolving to occur.

229. Forces Causing Natural Circulation. Supplementing this, consider the variations of temperature recorded during the investigations. What causes natural circulation in vacuum pans? When the level of the strike is low, circulation is induced by the formation of vapor bubbles in the tubes of the calandria, thus reducing the weight, as compared with the balancing column in the downtake, which has no bubbles. The difference in the specific gravity of these columns causes upflow in the tubes and downflow in the downtake.

It has been shown that as the massecuite leaves the tubes of the calandria on its way up it contains many spots of different temperatures, the general average of which is represented by the reading of the pan thermometer. As the mass proceeds upward, when these spots reach a level where the local vacuum corresponds to their boiling point, a flash takes place with the release of a vapor bubble. This volume displacement causes a sudden readjustment of position permitting the vapor liberated to come into contact with some of the cooler massecuite, when an immediate condensation takes place.

This procedure is repeated on the way up, gradually lowering the temperature of the hot spots by raising that of the cold spots, ultimately attaining a general average indicated by the pan thermometer. This average exists in uniformity at the level of uninterrupted ebullition limited to a zone including 12 in. below the surface of the massecuite in the pan. This is the main force causing natural circulation, for whereas the individual displacements are of small volume and duration, they are of infinite number and extend from the tubes themselves to the very top of the mass. It can be seen, therefore, that the weight of the agitated solution in the outer periphery of the pan is lightened as compared with the downward-moving central stream which is free from flash spots.

Two dangers are involved. One is the fact that if the local temperatures are too high, there is likelihood of color increment. The other is that a point of unsaturation is reached as explained, wherein sugar actually redissolves into the molasses or sirup.

230. Mechanical Circulation. It was mentioned that improvements had been effected in calandria pans by increasing the diameter of the downtake. This was followed by the reintroduction of the old idea of circulation by mechanical means, which had failed many years ago, evidently owing to improper proportioning and inadequate powering. Figure 63 shows the Webre design.

The apparatus consists of a heavy central shaft driven and supported from the top, and extending into the downtake. A number of screw elements are provided at different elevations. The lowermost circulation element is at the bottom and consists of a hollow drum to the outside of which are attached

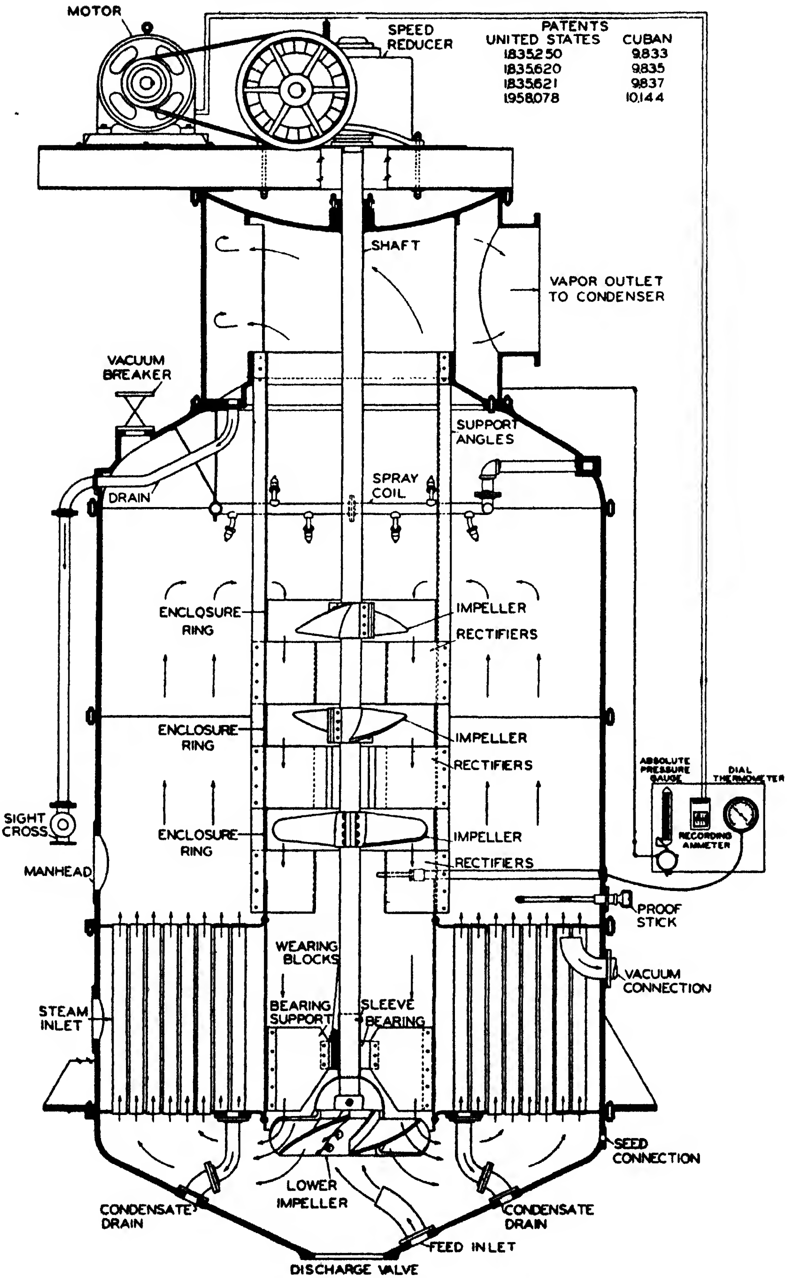

Fig. 63. Webre Circulator Pan.

multiple vanes. The upper screws are similar to ship propellers, and are enclosed in cylindrical rings to prevent radial flow away from the center. Above and below each of the upper screws are vertical rectifiers to overcome rotation of the massecuite. The lower drum has holes under the vanes, and feed is admitted from below and forced to pass through the orifices, thus providing a very satisfactory mixture.

Usually the circulators are motor driven, and a recording ammeter is provided located near the proof stick. The readings of this instrument are inversely proportional to the fluidity of the mass and hence provide a very useful element of control.

The speed of circulation is such that the entire contents of the pan are turned over at least twice per minute, under which conditions there will be very little local overheating or segregation of the mass due to dead pockets.

The advantages of mechanical circulation are:

a. Thorough and uniform distribution of feed as it enters the circulating mass, avoiding local dilution.

b. Good control of mass densities as indicated by a recording ammeter attached to the motor drive.

c. Adequate rapid movement of the mass, depending only on controlled mechanical equipment.

d. Minimum variations of local temperatures.

These characteristics have brought about practical advantages in both the raw house and the refinery, which may be outlined as follows:

a. Improved crystal formation.

b. Better pan performances, with lower steam pressures, larger capacities, and simplified means of control.

c. At the beginning of a strike, the circulation does not increase the rate of evaporation, but at the end it is six to eight times as fast, thus decreasing the time element when temperatures are highest.

d. Owing to less heat injury the color increment in strikes is lower.[6]

UNDERLYING PRINCIPLES OF PAN CONTROL AND THE USE OF INSTRUMENTS

231. Introductory. Up to twenty years ago, knowledge of vacuum pan operation was incomplete and unsatisfactory, and dependence was based on the experience and rule of thumb of the sugar boiler. It was Dr. Claassen who suggested supersaturation as the control element and put it into practice in Europe. His work was followed by that of Dr. Thieme (*Studies in Sugar Boiling*, New York, 1928), who developed it for the cane sugar industry in Java. The developments achieved since are derived from the pioneering of these two men. (See also discussion of "Theory of Crystallization" by Davies, p. 217).

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[6] A somewhat different design of mechanical circulator is described by Walter E. Smith, *Intern. Sugar J.*, April, 1933, p. 148. The advantages of forced circulation are discussed at length.

232. Theoretical Considerations. Before discussing the use of Dr. Claassen's data, another concept of the same problem is worth considering. Research done in Sweden revealed the real secret of the control of all crystallization and established the following principles.

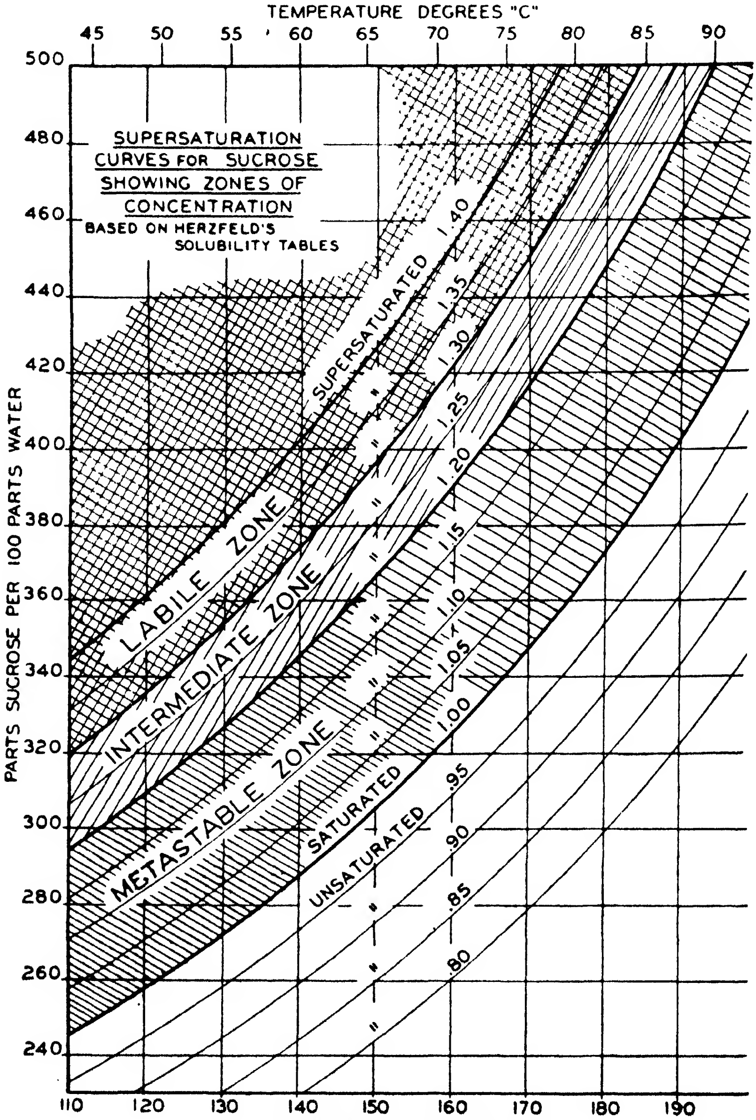

Fig. 64. Supersaturation Curves for Sucrose.

No crystals are formed, grown, or deposited from a solution 100 per cent saturated. For this, the solution must be supersaturated, which means that more solids must be in solution than could be dissolved by water at the given temperature. The degree of supersaturation has a remarkable influence on crystallization, and this can be divided into three phases:

a. The first or metastable phase includes a zone of concentration in which existing crystals grow in size, but no new ones form.

b. The second phase, just above the metastable, is the intermediate, within which not only existing crystals grow, but also new ones form.

c. Finally, beyond the intermediate is the labile phase, in which crystals form spontaneously, without the presence of others. (Figure 64 is good only for pure sucrose.)

This theory applies to all solutions from which crystallization takes place. As the purity decreases the supersaturation at the metastable line is higher. For example, the upper limit of the metastable zone for crystallizer strikes may be 1.40 or above, depending on local conditions. Means for determining the limits of the various zones of concentration are, therefore, of paramount importance. As far as the sugar industry is concerned, the suggested procedure is as follows.

233. Determination of Critical Points. To determine the saturation point, with sirup concentrating in a pan, place sharp sugar crystals in the proof stick and allow them to be exposed to the sirup. As soon as the corners of these crystals cease acquiring rounded edges as observed by a microscope, saturation has been reached. The Saturascope described on p. 536 also serves well to make this determination.

To determine the metastable line, separating the metastable phase from the intermediate phase, with a strike boiling in the pan, increase the concentration until new grain begins to appear. This is the upper limit of the metastable zone, and the point determined is on the metastable line.

The labile line is the demarcation between the intermediate zone and the labile zone. A point on this line is secured while making grain by the old method of "letting it come in." As soon as the microscopic nuclei appear, the labile zone has been reached.

These data are used in the regular routine of making sugar, knowingly or not. Pans must be seeded while the concentration is in the metastable zone. Also, after grain has once been obtained, the massecuite must be maintained within that zone until the end of the strike. If the concentration is allowed to go below, sugar crystals will dissolve. If it goes above, false grain will immediately appear.

To make grain by shocking, seed in the metastable zone and push the concentration into the intermediate zone to induce the formation of new grain in the presence of others (shock). After sufficient nuclei have been secured, the strike must be brought back to the metastable zone, as mentioned in the above paragraph.

To make grain by the old method of "letting it come in," the labile line must be exceeded.

234. Need for Instruments. For accurately controlled pan work, it must be possible to determine in which of these three zones the operation is being carried on and in which part of each individual zone so that the feed or the evaporation can be adjusted accordingly. Thus it is indispensable to have some quick means of determining the concentration, or some indication closely related to concentration.

The ideal instrument giving a direct reading of supersaturation has been designed by A. L. Holven and is now available (see Sec. 240). A number of alternative devices have been used and the one which offers the easiest application is the boiling point rise combination.

235. Boiling Point Rise and Supersaturation. The boiling point rise (b.p.r.) (also called boiling point elevation, b.p.e.) is the difference between the temperature of a boiling solution and the saturation temperature of the vapors leaving it. Three important facts must be remembered. (1) The b.p.r. decreases as the temperature is lowered. (This has been thoroughly established by A. L. Holven,[7] and is in contradiction to Dr. Claassen's data.) (2) The b.p.r. increases as the purity decreases. (3) At a given purity and temperature, the b.p.r. is exactly proportional to the solids dissolved per 100 parts of water; hence, if the b.p.r. at saturation is known, it becomes very easy to determine the supersaturation by simply dividing the observed b.p.r. by the b.p.r. at saturation.

Example. B.p.r. observed, 25; b.p.r. saturation, 20.

$$\text{Supersaturation} = \frac{25}{20} = 1.25$$

236. Standards for B.p.r. The determination of the correct b.p.r. at saturation for various purities and temperatures is a problem. Holven has given formulas deduced from his research which enable us to arrive at what is believed to be a satisfactory standard, and the chart in Fig. 65 was computed accordingly. The *purities are true purities and apply to the liquid part of the strike only*. This means that the purities to be considered are those of the molasses in the strike at the time of the observation.

The determinations made by Holven were based on a certain composition of non-sugars, and since these are subject to considerable variations, it would be too much to expect absolute accuracy. Certain allowance will have to be made in the field, but even though the data are not precise, the discrepancy is proportional, and compensation is not a difficult matter, as will be indicated later.

Determination of the correct b.p.r. in the pan, while making a strike, is no easy matter. Since it is the difference between two temperatures, each must be taken accurately, or the conclusions will be misleading.

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[7] *Ind. Eng. Chem.*, April, 1936, p. 454.

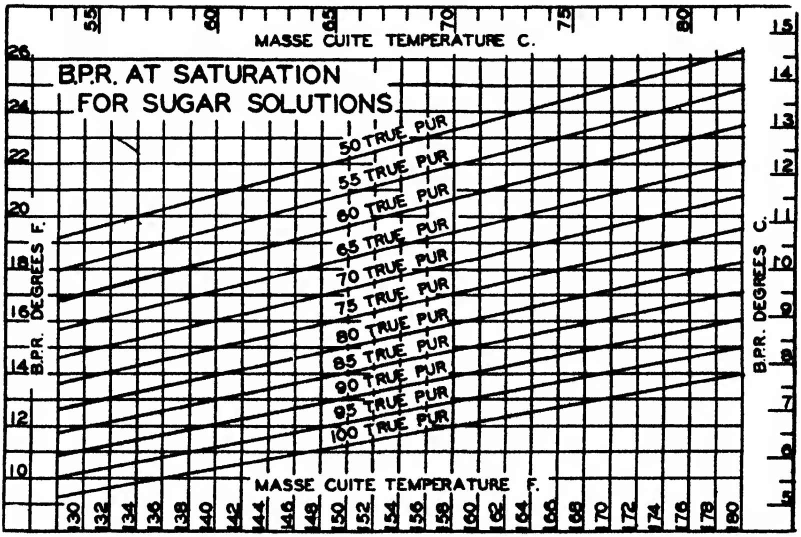

Fig. 65. Boiling Point Rise for Sugar Solutions of Different Purities.

237. Determining Massecuite Temperature. The massecuite temperature cannot be read satisfactorily by the ordinary angle thermometer with which vacuum pans are equipped. The temperature at the boiling surface of the mass is what is needed, and this is inaccessible to the instrument, which usually indicates a higher temperature, since the massecuite has just passed through the heating surface before reaching it. Best results are obtained by making the observation in the downtake through which the circulating mass returns below the heating surface after having released its heat by flashing at the boiling level.

This necessitates either a thermocouple, or a mercury-operated dial thermometer, with the sensitive element located in the center of the downtake. The dial size recommended is 12 in. with an indicated temperature range from 120° to 180° F., and an overtravel to at least 225° F., to avoid injury to the instrument while steaming out the pan.

238. Determining Vapor Temperature. A thermometer element inserted into the vapor leaving the pan will not give desired results, because the vapor is superheated and the readings will be too high by the amount of the superheat. There are two satisfactory ways to determine the correct saturation vapor temperature. The original was to have a small, closed vessel, called a pilot pan, arranged for boiling water and discharging its vapors into the massecuite pan. A thermometer inserted into the vapor pipe of this pilot pan will give the correct saturation vapor temperature provided this vapor pipe is large enough to have substantially no friction loss. Many installations have failed because they overlooked this important detail.

Another way to obtain the correct saturation vapor temperature is to use an absolute pressure gage instead of a vacuum gage, and to have this calibrated to read absolute pressure in inches of mercury as well as the corresponding water vapor temperatures. This instrument is really a specially designed barometer indicating the absolute pressure in the pan.

The combination of the absolute pressure gage and the dial thermometer works very well. There are charts which can be attached to the dial thermometer, by means of which, knowing the vapor temperature from the absolute pressure gage, the b.p.r. can be read without subtraction. Other charts can be plotted for various purities by means of which supersaturation can be observed.

The charts selected for the thermometer must correspond to the true purity of the mother liquor or molasses at the time of observation. Since a considerable range is included, it is evident that this will require the use of several of them during the boiling of a strike. This unwanted complication is avoided by selecting a chart that corresponds approximately to the average purity of the molasses from the beginning to the end of the operation, and by accepting the fact that the indications will be apparent and not true supersaturation.

It is not difficult to work this combination. The smear points are soon located on the charts, and once the operation has been explored by trial, it becomes routine work. In making grain by any method, supersaturation control is invaluable. It is much better to have steady conditions, particularly as regards vacuum and feed temperature.

239 Micromax Instruments. An excellent combination used in Hawaii has two thermocouples at the points named (downtake and pilot pan) and records the b.p.r., from which it is made to control an electric feed valve regulating the concentration to hold the desired b.p.r. This is the basis of automatic vaccum pan operation practiced there. Automatic pan control is possible only when most of the other variables have been eliminated. This means constant vacuum, constant steam pressure, constant feed density, and constant feed temperature. The work in Hawaii has been very successful. A detailed description of full automatic pan control as used in Hawaii is given by Fabirs.[8] Among the devices described are: thermostatic regulation of temperatures of massecuite and vapor; vacuum control by automatic regulation of the water to the condenser; control of the density of the massecuite by regulating the flow of the feed, depending on the b.p.r.; thermostatic control of the temperature of the feed (molasses or sirup), and constant density control of the same materials; constant rate of evaporation by having the volume of condensate discharged by the trap regulate the steam flow to the calandria; and finally, the control of final density by the amount of power required for the Webre circulator.

240. Direct Measurement of Supersaturation. A. L. Holven has worked out a method for the continuous automatic measurement of super-

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[8] *Intern. Sugar J.*, May, 1938, p. 184.

saturation.[9] The application of this principle to actual pan boiling operations is covered by patents,[10] but the theoretical considerations developed by Holven should have a profound effect on all pan control methods using the boiling point rise principle. An extended discussion of his findings cannot be given here, but the most significant conclusions may be summarized as follows:

a. Constant supersaturation lines are substantially linear for all supersaturations within the range of absolute pressures encountered in sugar boiling practice, not only for solutions of pure sucrose but also for impure sugar solutions.

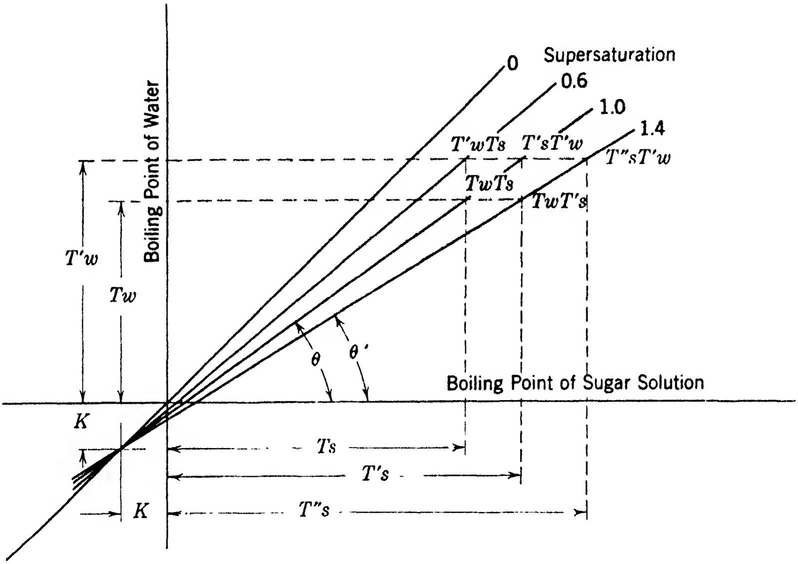

FIG. 66. Supersaturation as a Function of Boiling Points of Water and Sugar Solutions.

b. Constant supersaturation lines converge to practically a common point of intersection, and deviations can be compensated for easily.

c. Any degree of sucrose supersaturation may be represented as a line whose slope or tan θ value serves as an index of the degree of supersaturation.

Holven has shown that supersaturation is equivalent to a *ratio* of the differences between the boiling point of water and a reference temperature, and the boiling point of sugar solution and a reference temperature, when both are boiling at the same absolute pressure. Expressed as a formula this is $\frac{T_w - K}{T_s - K} = \tan\theta$ when T_w is the boiling temperature of the water, K the reference temperature, and T_s the boiling point of the sugar solution at the same

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[9] *Ind. Eng. Chem.,* October, 1942, p. 1234.

[10] U.S. patents 2,135,511, 2,135,512, and 2,263,847.

absolute natural pressure. Tan θ represents the slope of the curve of constant supersaturation lines as shown in Fig. 66.

The basic circuit for the measurement of supersaturation is shown in Fig. 67. The temperature-measuring element is a resistance thermometer placed in a pilot pan connected by means of a vapor pipe to the vacuum pan. If this resistance is included in one arm of a Wheatstone bridge circuit *W*, as shown, the other resistances R_1, R_2, and R_3 can be so proportioned with respect to each other that the potential difference between the points *E* and *D* is at all times proportional to $T_w - K$. Likewise, a similar resistance thermometer mounted in the vacuum pan and connected in a second Wheatstone bridge circuit *M* and similarly proportioned with resistances R'_1, R'_2, and R'_3 will provide a quantity representative of $T_s - K$.

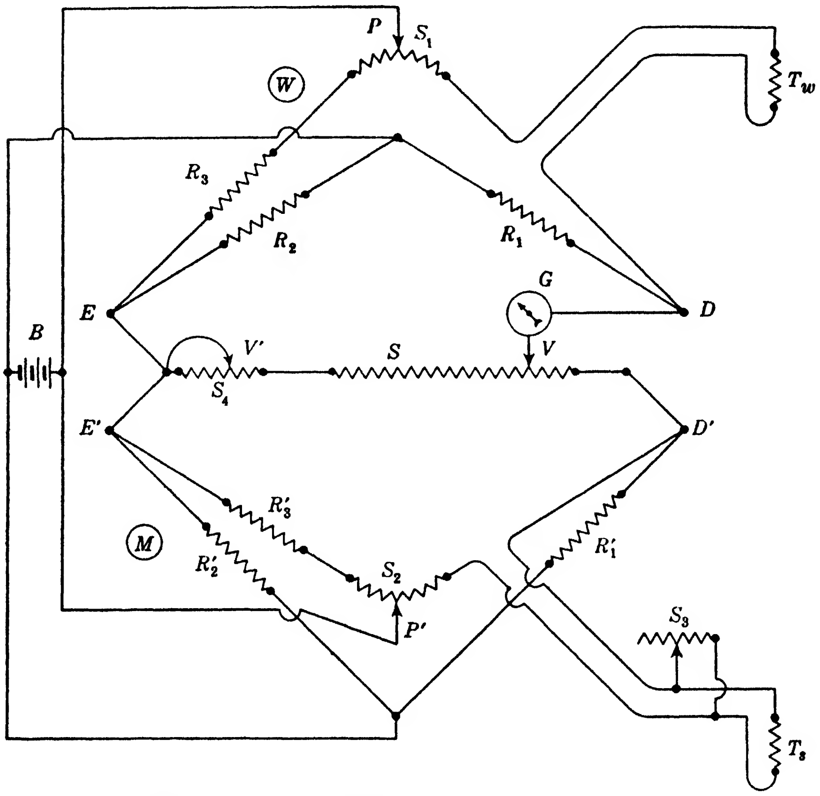

FIG. 67. Basic Circuit for Measurement of Supersaturation (Holven).

By opposing the potential differences developed in the respective Wheatstone bridges in a simple potentiometer circuit, their ratio, which is indicative of the supersaturation coefficient, can be read directly on the slidewire S as a measurable value.

Compensation for the effect of purity has been obtained by providing an adjustable high-resistance shunt S_3 across the resistance thermometer immersed in the boiling sugar solution. With a 100° purity sugar liquor, the full amount of this resistance S_3 is in circuit. As the purity of the sugar liquor decreases, the amount of resistance in the shunt S_3 is correspondingly reduced so that the effective resistance of the $P'D'$ arm of the Wheatstone bridge (which includes T_s and S_3 in parallel) is reduced to the value which T_s would have if immersed in 100° purity sugar boiling at the prevailing absolute pressure.

By incorporation of the above-described circuits into a standard recording instrument, a direct measure of the coefficient of supersaturation is obtained on a suitably calibrated slidewire.

The practical application is possible by utilizing any one of a number of standard recording instruments that will provide a measure of the potential differences established. Several supersaturation recorders based on these principles have been in successful operation for a number of years.

241. Electric Conductivity Instruments. Another instrument that has found extensive application is the electric conductivity apparatus, designed orginally by Dr. Peter Honig of Java. It is both recording and indicating. The principle involved is based on the fact that the conductivity of a sugar solution is proportional to its water content, which is closely related to the concentration, and therefore the supersaturation. One must be sure so to mount the electrodes that they get a fair sample of the strike. Alternating current is used with adjustable voltage. The readings are in milliamperes of current flowing between the electrodes. Different voltages are used for different purities. The best-known apparatus for indicating the electrical conductivity in pans is the Cuitometer the use of which has been described in detail by Alewyn[11] and Honig.[12] Stare[13] has shown that the conductivity method may be used in refinery pan boiling by the use of larger electrodes and by providing means of adjusting the voltage between the electrodes instead of using a constant voltage as is done in the Java apparatus.

242. Refractometers. Refractometers are used in Europe. They are attached to the shells of the pans for their element of control. The readings are proportional to the solids in solution, and hence a simple straight line relation can be established between the refractometer readings and supersaturation. The difficulty is to find a satisfactory location for fair sampling. (For a description of the use of the Zeiss refractometer in raw sugar pan boiling see S. J. Saint, *Intern. Sugar J.*, June, 1932, p. 227.)

243. Dittmar-Jantse. The Dittmar-Jantse instrument records the difference of pressure between two points: (1) the vapor pressure in the dome

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[11] *Intern. Sugar J.*, September and December, 1933, pp. 352, 465.

[12] *Intern. Sugar J.*, March, 1933, p. 111.

[13] *Intern. Sugar J.*, January, 1934, p. 19.

of the pan, and (2) the vapor pressure exerted by water in a closed container submerged in the massecuite in the downtake near the bottom. The indications of the instrument are closely related to the solids in solution, and hence to supersaturation. (See also Tromp, *9th Annual Proc. Assoc. Cane Sugar Tech. Cuba* [1935]; Gonzalez Maiz, *12th Annual Proc. Sugar Tech. Cuba* [1938].)

244. The Brasmoscope. The Brasmoscope is based on the b.p.r. principle and consists of a combination of an absolute pressure gage and an angle thermometer set very close to one another. Tables for various purities and depths of strikes, with connecting lines between the two instruments, are arranged so that when the readings coincide with the same lines, a supersaturation of 1.20 obtains. This arrangement has been abandoned for the more accurate methods described above.

PROCESS OF SUGAR BOILING

Since the same principles are involved and the same problems encountered, whether the pans are controlled by hand or by instruments, the operation by the two methods will be discussed concurrently, pointing out the essential considerations from both standpoints. Thus, the operator who is unaccustomed to the use of instruments will learn more quickly, and the one who knows instrument control only will appreciate more fully the task of his less-fortunate fellow technician. The work differs little whether a coil or a calandria pan is used; the only allowance to be made is that the one has a number of superposed heating elements, the other, only one.

The operation must be planned with a definite objective, namely, to have a certain volume of sirup at a certain temperature when grain is made in the pan. This volume will depend upon whether fine, coarse, or intermediate sugar is desired, the coarser the grain, the less the volume. It is advisable to establish a fixed routine and always to make grain under the same known conditions. The pan temperature at this stage should not be too low, because the viscosity will be greater, and the control more difficult. A vacuum of 25 in. at sea level is a good standard and will give a graining point of between 150° and 160° F. The normal procedure would be about as follows:

The pan is closed and the vacuum raised, the full amount of injection being turned on the condenser, because the rate of evaporation with low-density sirup at the beginning will be very high. As soon as vacuum has risen sufficiently to draw, open the charge valve, and take in enough sirup to cover the calandria, or several coils, if a coil pan is in use. Steam is now turned on full, and evaporation proceeds very rapidly. Bearing in mind the level required at the graining point, it will probably be necessary to draw in more sirup, as the volume decreases.

The heating surfaces on which steam is turned must always be covered with sirup, otherwise caramelization will take place. With a coil pan, as concentration progresses and graining density and volume are being approached, it may be necessary to shut off some of the upper coils which are about to run dry.

If the full graining charge is taken at the beginning, the hydrostatic head over the heating surface will slow down the operation, and danger of entrainment will be greater. Naturally, the initial volume of sirup required to produce the fixed volume at the graining density will vary greatly according to the Brix of the available sirup (see Fig. 75, p. 213).

245. Graining: Old Method. The progress of the concentration under fixed conditions mentioned above is followed by hand by drawing a sample through the proof stick and noting its proof, viscosity, or feel. Since viscosity is inversely proportional to temperature, it will be appreciated that accuracy in this determination is promoted by always carrying out the process at the same vacuum. By taking a sample between the thumb and the forefinger, and separating them, it is easy to note the length of the sirup string before it breaks. Grain will not come in by the old method until this string is longer than can be spanned between the fingers of one hand. With sirup at 83° purity, and instrument control, the graining point will be at about 27° b.p.r., or a supersaturation of 1.75, in the labile phase wherein crystals form spontaneously. (See Sec. 232.)

Grain will now come in rapidly, and it will soon be necessary to decide when the quantity is adequate. This is determined only by the judgment of the skilled sugar boiler, based upon observation of a sample spread out on a piece of glass plate. Here a good magnifier or microscope is of considerable help. When there is enough grain, further formation is arrested by taking in a charge of sirup and decreasing the vacuum 3 or 4 in. With hand control, the sense of touch and the appearance of a proof-stick sample determine the progress of the grain formation. The object is to return to the metastable phase, in which existing crystals grow, but no new ones form. With instruments, however, it is possible to be precise. The b.p.r. should be lowered to 19, corresponding to a supersaturation of about 1.20.

246. Shock Seeding. A method which has rapidly gained favor in refining and cane factory work was introduced by Zitkowski in beet sugar plants. It consists of concentrating to above saturation and then drawing into the pan a small quantity (a pound or so) of sugar dust. It was believed at first that this dust served as the nucleus of grain formed by its introduction, but it is now agreed that it constitutes a shock to the supersaturated sugar solution, whereby the formation of new grain is induced much sooner than in the former procedure. In fact, theoretically, the shock should be applied as soon as saturation has been attained, which means in the metastable phase. A very regular grain results from this method if properly employed.

With hand control, the time at which to shock is determined by the string length of a proof-stick sample, tested between the thumb and the forefinger. This length should be about 1 in. With instrument control, the powder should be drawn in just beyond the saturation point. This can be taken as b.p.r. 16 or S.S. 1.10 with 83° purity.

A common mistake is to shock too late, assuming that the formation of grain will be more uniform. Such a procedure involves the danger of conglomeration due to excessive concentration, and grain formed by shocking too late will be no better than that made by "letting it come in." It is not necessary to have absolute uniformity of nuclei to get commercially uniform sugar. The rate of sucrose absorption by any given crystal is proportional to its surface, which varies with the square of its linear dimension whereas the volume and the weight vary with the cube of the linear dimension. Hence, it is seen that the surface of a crystal as compared to its weight is inversely proportional to its size. In other words, for a given weight of sugar, the smaller the crystals the greater will be the accreting surface, and therefore the faster the rate of growth. This results in an automatic correction of initial grain irregularities, since the small ones grow faster than the large ones.

It is therefore easy to see that if the final strike shows considerable variation of grain size, this is evidence that either new small grains were drawn into the pan with the feed, or that the strike was smeared (false grain) after the footing was made by allowing the supersaturation to go too high. This can happen by (1) a sudden increase of vacuum, (2) allowing evaporation to proceed too fast, (3) air leakage through the foot valve, and (4) admission of cold feed into a hot pan while the strike is going up.

Grain will not appear immediately upon the introduction of powdered sugar as recommended herein. It is advisable to admit a minimum quantity of air with the shock to avoid disturbing the temperature equilibrium. When grain does begin to form after a few minutes, it will be necessary to decide when to stop it by proof-stick sample examination as before.

The grain will be all in at b.p.r. 18.5 to 19 (supersaturation 1.25–1.30), so the change of concentration to revert to the metastable is very small indeed, being a matter of perhaps 1° b.p.r. In this case, it is much better to effect the change by feeding sirup and allowing the vacuum to remain undisturbed. As a matter of fact, the best results have been obtained by holding the vacuum constant during the entire operation.

Until grain has been fully developed, it is urgently necessary not to allow the supersaturation to go too high, because even before false grain appears, conglomeration will take place, after which nothing can be done to correct this except to melt the sugar.

247. Pan Seeding. The best method of graining a pan is that known as pan seeding or full seeding. It consists of adding at the proper moment the full amount of grain of predetermined size to equal the number of grains in the finished strike. In this case, no grain is formed in the pan at any time, and the concentration is held in the metastable or crystal-growing phase. Seed is introduced as soon as the saturation point has been passed, and before the metastable line is reached. To determine the proper amount of fines of a given grist to introduce into a pan to make a strike of sugar of a certain size:

a. Find the weight of sugar expected from the strike.

b. Count and weigh about 500 crystals of this grade sugar.

c. Count and weigh 500 crystals of the seed powder to be used.

d. Dividing (*c*) by (*b*) and multiplying by (*a*) gives the weight of seed.

After this has been determined and tried, minor corrections can be made to take care of variations. After the procedure has been established, standardization can eliminate the personal equation, and the operation may then be duplicated by anyone. (See also Webre, *Proc. 6th Congr. Intern. Soc. Sugar Cane Tech.* [Baton Rouge, 1938], p. 945, for more detailed discussion of pan seeding.)

This method of pan seeding is now used universally by refineries in the production of large-grain sugars such as sanding, manufacturer's standard, medium, and coarse. With the right amount and size seed for the grain, these "specialty" sugars can be produced with a much more regular crystal, entirely free from conglomerates, which is practically impossible by the older methods.

It was mentioned above that the proper time to seed is when saturation is exceeded. With instruments, this will be very easily determined. Thus, with 83° purity and a strike temperature of 150° F., the b.p.r. at saturation is 14.5° F. Allowing a margin of 1.5°, the proper point would be when the b.p.r. reaches 16° F. at which the supersaturation is 1.10. To make this determination by hand it is suggested that a gram of coarse sugar be placed in the notch of the proof stick, and wetted with sirup in the pan and withdrawn. If, on inspection with a magnifier, the corners of the crystals are rounded, saturation has not been reached. If the corners remain sharp and square, saturation has been passed, and seeding, or shocking, may proceed. The Saturascope (p. 536) may also be used for this purpose.

248. False Grain and Conglomerates. It is necessary to digress a moment to elaborate on certain difficulties likely to be encountered in operation at this point, unless pan seeding is used. When the formation of grain is arrested by raising the temperature, by dilution, or by both, if the process is carried too far, it is easily possible to overshoot the mark, dropping the mass below saturation and dissolving all the grain. This would necessitate a fresh start. Even if all the grain is not dissolved, part of it may be destroyed, thus leaving an insufficient quantity and requiring the formation of additional nuclei to make up the loss.

Fundamentally, after the grain has been secured, the concentration must be brought back to the metastable or crystal-growing phase, where it must remain for the duration of the strike.

If at any time the concentration is carried too high, new unwanted crystals will form. These are called false grain or smear. They must be dissolved by dilution with water, sirup, or low-density molasses. Even before false grain appears conglomeration will take place, which is most undesirable.

Conglomerates—mounted grain, married grain, agglomerates, rolled grain—all mean the same thing, a grouping of a number of crystals which then grow together as one. Once formed, conglomerates cannot be separated into their component elements, and will so remain to the end of the strike. As all sugar men know, these group crystals are objectionable, as impurities and dirt lodge between the many seams, thus making it impossible properly to wash the sugar, and yielding a raw product of poor filterability, not to mention the increased chance for bacterial infection. In refined and direct consumption sugars, the impurities held in the conglomerates lower the quality of the sugar as a whole and make thorough drying in the granulator more difficult.

Certain important facts should be emphasized regarding conglomeration. First, conglomerates form much more readily at higher purities. Second, low purities never conglomerate. Third, in selecting fines for seeding, if these are conglomerated, the resulting final product will be conglomerated also, since conglomerates are never destroyed. Fourth, conglomeration takes place at the upper edge of the metastable zone, just before the occurrence of false grain. In other words, if false grain has been formed, so have conglomerates.

For the reasons given, fines used for seeding should be made by crushing coarse sugar. This has a number of advantages. (1) These broken pieces will not be conglomerated. (2) They will revert to perfect shape very rapidly owing to physical forces compelling definite crystal form, and hence, fines so made for seeding will grow much more rapidly in the pan than powdered sugar secured from the dust box of a granulator. The maximum rate of growth thus occurs when it is needed the most. This is not theory only but has been actually proved in plant practice.

If grain is started by seeding low-purity molasses (say 60) with fines made by crushing large crystals, there will probably be no conglomeration whatever. This is a new field that is now being developed with outstanding success.

249. Bringing the Strike Together. After grain has been secured by whatever method, the most difficult and important step is to bring this mass together, as the nuclei are widely dispersed and in a critical condition owing to the restricted accreting surface they present for sucrose absorption. Here, automatic vacuum control will help for, if the evaporation is too fast as revealed by a rise in the b.p.r. or supersaturation, or as found by the sense of touch if hand operation is used, the temptation is to shut off steam, thus slowing down the rate of evaporation. Without vacuum control, and without changing the water supply to the condenser, this will result in an immediate increase in vacuum, accompanied by a corresponding decrease of temperature, and there is imminent danger of the formation of false grain or conglomerates. This change will be shown by instruments, but the reaction is too rapid to record before it is too late to prevent.

250. Movement Water. In some countries, if the rate of evaporation is too fast for the rate of crystal growth, instead of reducing the steam flow with the danger outlined above, overconcentration is avoided by feeding "movement water." As the crystals develop, the water is gradually reduced until it can be discontinued altogether, when the rate of sucrose absorption by the nuclei corresponds to the rate at which it is being made available by evaporation. Water involves additional steam consumption, and the practice is easily abused by the sugar boilers, hence it has been discouraged.

With mechanical circulation as well as vacuum control, grain development becomes easy, because circulation is adequate, irrespective of the rate of evaporation, and if the supersaturation tends to rise excessively, steam can be reduced or shut off altogether if necessary. The temperature cannot change because the vacuum is constant.

251. Tightening the Mass. It is not advisable to increase the volume until the crystals have grown enough to fill most of the void spaces occupied by the sirup or mother liquor. It is an interesting and curious phenomenon that the rate of crystal growth is faster with a tight strike than a loose one. When crystals are close together, they rub, and remove the partially exhausted sirup adhering to their surfaces, and replace this film with a fresh one. Especially with high purities, it has been observed that when a strike is tightened within reasonable limits, upon reaching a certain critical point, the b.p.r. will actually go down, showing that the fluidity of the mass has been decreased, not because of increased concentration of the liquid, but because the volume of the sirup approaches the theoretical volume of the voids between the crystals.

In practice, this optimum point is indicated as a phase just preceding heavy shaking of the pan caused by lack of fluidity. If the pan vibrates excessively the strike is too tight and must be fed faster. In refinery practice, when this stage is reached, control instruments are of questionable value, as the supersaturation of the sirup which they indicate is no longer the controlling factor. There is still room for study on this point. With raw pans, the instruments are always usable, and their indications are reliable.

The great value of instruments is that they eliminate the personal equation, substituting definite readings for the sense of touch of the sugar boiler. The purpose of either method is to control the operation so that the maximum rate of crystal development is obtained, and the formation of conglomerates and false grain is avoided. One of the worst errors is to allow the strike to become too loose, and then to try to correct this mistake too fast. Invariably the result will be false grain.

As the development proceeds, the rate of sucrose absorption by the crystals becomes greater than the rate at which it is being made available by evaporation, all indicated by instruments, or by "feel," and it is now possible gradually to increase the vacuum. Massecuite takes time to respond to a change, and until equilibrium is established, the instruments will not give the corresponding indications. As the volume of the strike increases, the evaporating rate will be reduced which results in a correspondingly smooth increase of vacuum if the setting of the condenser is allowed to remain undisturbed, assuming that each pan has its own condenser and that the vacuum is not automatically controlled.

During the making of a raw strike, compensation must be made for the gradually decreasing purity. If the instruments are based on supersaturation, it may be necessary to change the charts. If the b.p.r. is the control element, it will have to be increased gradually, reaching a maximum at the end of the strike.

252. Final Concentration. When the volume of the massecuite has grown to the full capacity of the pan, the final concentration is arrived at by shutting off the feed and allowing evaporation to proceed until the proper Brix is attained. By hand, this is determined by taking a sample from the proof stick and forming a cone or ball of massecuite in the palm of the hand or on the ball of the thumb and noting how quickly it flattens. It takes a great deal of experience to do this, but it is surprising how accurate a good sugar boiler can be. They rarely miss by more than one-half point Brix.

With control instruments the point is determined by attaining a certain b.p.r. or supersaturation. With mechanical circulation, the reading of the horsepower developed by the drive provides an accurate and reliable test after proper calibration.

If, on the other hand, the strike has been carried loose, there is imminent danger of forming false grain when the final concentration is given. To avoid this, it will be necessary to proceed very slowly. In many instances it has been necessary to shut off part of the steam at this point, and *not to allow the vacuum to rise while this is accomplished.*

253. Steaming Out or Washing Out. After the pan has been discharged, the massecuite adhering to the inside must be completely removed before the beginning of the next strike, otherwise the crystals may disturb the graining operations described previously. It was formerly general practice to admit steam into the pan to heat and dissolve this grain, but recently water sprays have been found to be more effective and desirable, as they keep the heating surfaces cleaner and make less color.[14] The sprays should be so placed as to reach all parts of the heating surfaces and shells, and the water should strike with considerable force. A single 2-inch spray nozzle in the dome of the pan, properly placed and directed, is effective.

254. Temperature during Boiling. There is a conflict of opinions regarding the temperatures at which massecuite should be boiled in vacuum pans. The tendency now is toward cooler pans because of less inversion and color. Normal practice has been to make refined white sugar at about 180° F., and plantation whites somewhat cooler. However, many refiners today insist on maximum temperatures of 145°–150° F.

High-test raw sugars are usually grained at 160° F. although this is far from being universal. It was pointed out in the preceding discussion that the viscosity is less at higher temperatures, and therefore the crystallization simpler. However, high temperatures always promote a color increment which is to be avoided. It is customary to boil low-grade raw strikes as cool as possible both to promote exhaustion of molasses and to avoid "froth fermentation" (see Sec. 286) which is usually not present below 150° F.

[14] Salinas, *4th Annual Proc. Assoc. Cane Sugar Tech. Cuba,* 1930.

Refinery soft brown sugars are boiled with the maximum vacuum obtainable, which gives a soft feathery grain.

255. Pan Feeding by Drinks. In this discussion it has been assumed that the feeding of the pans is done continuously, without which instrument control is of little value. The old method was to feed discontinuously, by "drinks." In this case, the concentration is in periodic oscillation between low supersaturation and high supersaturation. In view of present knowledge, this method is not to be recommended because, not only does it cause loss of time but also the danger of forming conglomerates and false grain is much greater.

256. Charts. The above discussion covers in a general way the technic of making grain, and the method of developing this grain to make a finished strike. If a dial thermometer and absolute pressure gage are used as control instruments, it will be necessary to make proper adjustments and allowances according to the purities. If supersaturation is the control element, it will be necessary to select a chart that corresponds approximately to the average true purity of the sirup or molasses in the strike. Readings by these charts will not truly represent supersaturation, but a figure closely related to it. It will be realized that, in all but refinery white strikes, the purity of the molasses will go down progressively as the massecuite builds up, from sight glass to sight glass, and it is suggested that a schedule be made and posted on each pan for the guidance of the sugar boiler. The schedule should show the operating apparent supersaturation at each sight glass level for each kind of strike, as well as the supersaturation when the final concentration is given. If the b.p.r. is used instead of supersaturation, a similar schedule can be made answering the same purpose. With this set of simple instructions, the problem of sugar boiling is greatly simplified.

257. Brix and Evaporation. In the past, the advice of sugar boilers has been followed blindly regarding the concentration of the feed sirup or molasses. Recently, prevailing densities approach 70 Brix. The apparent Brix of the strikes themselves in a raw sugar factory are about as follows:

Grain strikes	90.0	
A strikes	92.0	
B strikes	93.5	
C strikes	96.0	and sometimes up to 100.0

Assume that 100 lb. of solids are introduced into these pans as sirup or molasses, and concentrate this to the final Brix of the various strikes as outlined above. The chart in Fig. 68 shows the evaporation at various densities for the different strikes and graphically indicates the fact that the steam consumption with 55° Brix sirup or molasses is 100 per cent greater than where 70° Brix is fed to the pans.

The only legitimate objection to high Brix sirup is the possibility of scaling the tubes of the evaporators. As regards molasses, there is likewise no objection. The critical point is that all the smear crystals contained in the molasses leaving the centrifugals should be completely dissolved. This is effected through dilution and agitation, supplemented with heating to at least massecuite temperature. After such a treatment, there will be no sugar crystals left at 70° Brix. (See Sec. 294 for automatic dilution and heating.)

Many sugar boilers insist on a lower Brix because it is easier to manipulate the pans. It is known that circulation is brought about by evaporation, and that if there is less evaporation, there will be correspondingly slower circulation, and this would apply particularly to low grades. With mechanical circulation, the argument does not hold, because circulation is effected by mechanical means and is the same regardless of the rate of evaporation.

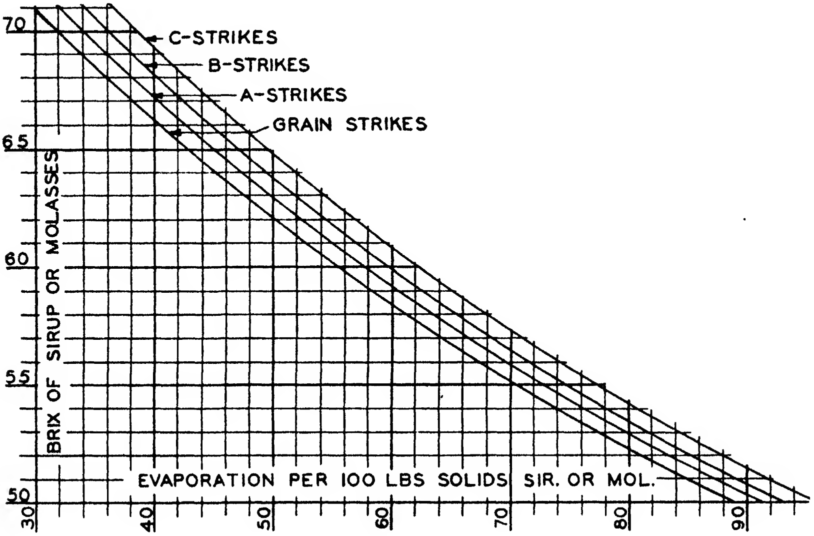

FIG. 68. Evaporation per 100 lb. Solids.

Many mills operate successfully with both sirup and molasses at 70° Brix at the pans, and there is no excuse for demanding 55°, which involves double the amount of steam and apparatus.

PAN BOILING SYSTEMS

258. Change in Methods. Since earlier editions of this book were written, the methods of sugar boiling and the handling of massecuites recommended at that time have been generally adopted. These consisted of: (1) the production of only one grade of sugar; (2) the use of seed grain in high-test massecuites; (3) the use of crystallization-in-motion apparatus; (4) the discontinuance of string-proof boiling with the accompanying hot-room procedure.

The disappearance of "molasses" sugars from the market (Sec. 359) and the production of "centrifugal" sugar only has been attended also by a steady increase in the test of the raw produced.[15] Where 96° polarization was the aim fifteen years ago, 97.0° is common in Cuba today, and the Hawaiian Islands are regularly producing raws of 97.5° to 98.0°. Pan boiling systems have been modified so as to give these higher polarizations.

259. Designation of Massecuites. In raw sugar practice the different grades of massecuite are designated either by letters *A, B, C,* etc., or by first, second, third, etc. Lettering is more general in Hawaii and Java and numbering in Louisiana and Cuba, though lettering is also quite common in the last-mentioned country. The molasses purged from the massecuites are designated by the same symbols, i.e., a *B* massecuite yields *B* molasses; a third massecuite, third molasses, etc. Refinery massecuites are usually designated by names: liquor strike, sirup strike, high remelt, low remelt, etc.

With the complications introduced into pan practice by boiling-in molasses many find it simpler to designate massecuites by names rather than number, e.g., sirup strike, mixed strike, crystallizer strike, etc. The various names and numbers used will be given in describing the different methods of pan boiling.

The boiling back of molasses has been practiced in Louisiana for nearly fifty years, especially in making grocery sugars. The mixed massecuite was often termed grain seconds. With the advent of crystallizers the present methods of boiling were developed simultaneously in Louisiana, Cuba, and Java. The work in the East Indies was apparently based entirely upon European methods, and that in Cuba upon Louisiana and beet sugar methods.

260. General Considerations. In describing the different pan boiling systems it will be understood that purities given are only average figures, which will vary through fairly wide ranges, depending upon the purity of the original sirup, the equipment of the factory, and other considerations. The purities of the various massecuites are generally arranged with a view to giving a fixed purity to the molasses that goes to make up the bulk of the crystallizer massecuite. Purities of 46° to 52° represent the average range under Cuban conditions.

The purity to be given the final or crystallizer massecuite depends upon several factors, viz., vacuum pan capacity, crystallizer and centrifugal capacity, and the desired exhaustion of the final molasses. The lower the purity of this massecuite the longer is the time required in boiling the strike. A very low-purity massecuite must remain a longer time in the crystallizers in order to lower the molasses purity by crystallization of the sugar. Where molasses is sold upon a basis of a certain sugar content, this also must be considered in fixing the purity of the second massecuite. For obtaining final molasses

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[15] Dr. Spencer was largely responsible for the development of these improvements, particularly in Cuba and Puerto Rico. He introduced the use of seed made from crystallizer strikes, also the double-purge method of purifying this seed; he was the first to make a single high-grade product of uniform composition, and the boiling methods originated by him were so practicable that they spread rapidly, resulting in the fulfillment of his prediction made in 1910 that "within a few years the string sugars will almost cease to be produced." (G. P. M.)

of apparent purities below 30°, early in the season in the tropics, and from 30° to 35° when the cane is ripe and of high purity, the massecuite should be reduced to below 60° purity. Where the pan, crystallizer, and centrifugal capacities are large, the purity may be reduced to 56° or even lower with correspondingly lower final molasses purity.

The other massecuites may be purged immediately after leaving the vacuum pan and should produce the same grade of sugar, although with molasses of widely different purities. The crystallizer massecuite is purged after remaining from one to four days in the crystallizers, depending on the type of apparatus. This massecuite yields a sugar that was formerly sold as seconds but is now used for seed grain as described below.

It should be noted that boiling-in molasses on the first strike should be accomplished after no more sirup is required. The molasses must be free of sugar crystals and should be warmer than the massecuite in the pan.

261. Massecuites Boiled with Seed. Grain, mingled into a magma with some convenient vehicle such as sirup or molasses upon which to build additional sugar, is called seed. Its use is general for all systems of boiling. The evolution of this practice is typical of the efforts that have been put forth in sugar making to produce a raw of better quality. The earliest form of the method was in Java and the Hawaiian Islands where the strike was started upon a footing of magma formed of string sugar mingled with sirup. A sugar of good grain and test may be made in this way, and the factory may produce a single grade of product without remelting the string sugar. However, the objectionable feature is that the nucleus of the raw sugar crystal (the string sugar) is of low test, and sugar so made will not give a high purity washed sugar in the affination process of the refinery (Sec. 349). The filterability will be low (see Sec. 350), and the sugar will not decolorize well in the char filters.

The next modification (introduced by Spencer in Cuba) differed only in the source of the seed. The grain sugar obtained from the crystallizer strikes was mingled with first or second molasses to form a magma and this was used as a footing for starting first massecuites or mixed strikes. It was soon recognized that the use of molasses for mingling the seed introduced color and impurities into the higher massecuites, hence sirup is now used for mingling. Another advance (also introduced by Spencer) is the double purging of the sugar for seed. The sugar purged from crystallizer strikes is mingled with a low-purity molasses and sent to a separate set of centrifugals, where it is repurged, washed, and then mingled with sirup (meladura) for use as seed in high-grade strikes (see Sec. 290). Seed sugar, repurged in this way, is practically of the same test as the grain which builds upon it, so that the resulting raw is uniform throughout its crystal structure. Magma for seed is stored in a crystallizer in motion and is always available without loss of time. In this respect it is preferable to cutting massecuite from another pan.

262. Selection of Boiling System. Multiple strikes must be resorted to in any sugar boiling system, whether beet sugar, cane factory, or refinery, and the problem in the cane factory resolves itself into the selection of the procedure which is best from the following viewpoints: (1) the refining quality of the sugar produced, (2) its keeping quality, (3) the purity of the final molasses, (4) the amount of reboiling required (cubic feet of massecuite per 100 lb. sugar produced), and (5) flexibility and simplicity as a factory operation.

It is evident that the purity of the original sirup will be a great factor in the selection of a boiling system. In the tropical countries where sirup purities range above 85° more boilings are necessary than in the sub-tropics such as Louisiana, where purities as low as 75° are not uncommon. In the discussion of the boiling systems outlined below, the higher purity sirups of the tropical countries are used as illustrations. Webre has calculated the weight of massecuite for the different boiling systems described, and these are shown in the chart in Fig. 75. The percentage of recirculation of molasses for the different systems is also shown by Webre in Fig. 74.[16]

263. Two-Boiling System. A diagram of this method is shown in Fig. 69. All the massecuites from which commercial raw sugar is to be produced

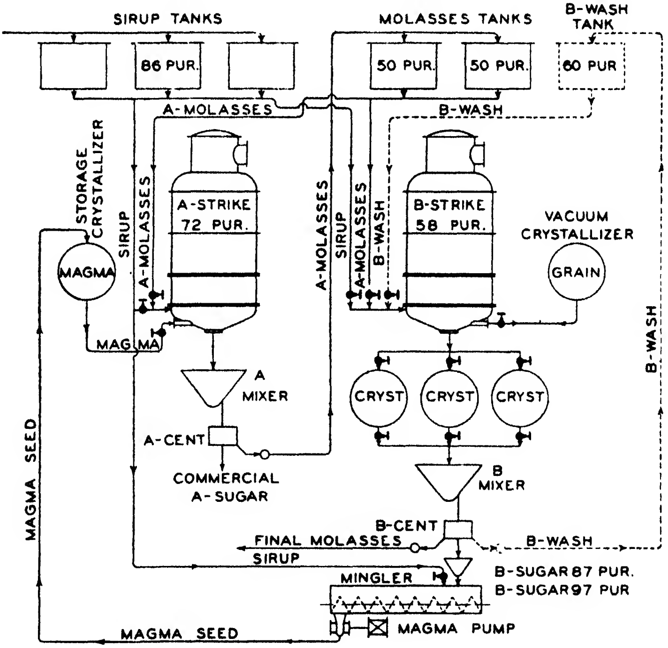

FIG. 69. Diagram of Two-Massecuite System.

are reduced to the same purity (from 70° to 74°) with molasses from previous strikes so as to give a second molasses of about 52° or lower. This

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[16] See also Webre, *Massecuite, Molasses and Sugar* (Spanish and English), U.S. Pipe & Foundry Company, 1930, 132 pages.

second molasses is boiled back into a massecuite of 58° formed on grain made from sirup. This massecuite goes to crystallizers (as do all final massecuites in modern practice) where it remains until cooled. Then it is separated into sugar and final molasses in the centrifugals. The sugar from the crystallizer strikes is mingled with sirup and taken back for seed into the commercial sugar strikes.

The two-boiling method has only the advantage of simplicity to commend it. The disadvantages are that all the output of sugar is from relatively low-test massecuite, with a detrimental effect on refining and keeping quality; also depending upon the purity of the original sirup, a large proportion of the molasses must be reboiled putting that much extra work on pans, crystallizers, and centrifugals, with increased fuel consumption. (See Figs. 74 and 75.) For these reasons the method has been entirely discontinued in Hawaii, where it was formerly very general, and is in use in only a few factories elsewhere.

264. Three-Boiling System. This method, with various modifications, is used (Fig. 70) by most of the Cuban houses and generally throughout the tropics. A first massecuite is boiled from sirup and first molasses to a purity of 80°–83°; a second massecuite, frequently called a mixed massecuite, is boiled from sirup and first molasses to about 70° or 72° purity; and a third massecuite, known as crystallizer massecuite (where the term "mixed" is used for the second boiling) is made at about 58° purity. In actual practice

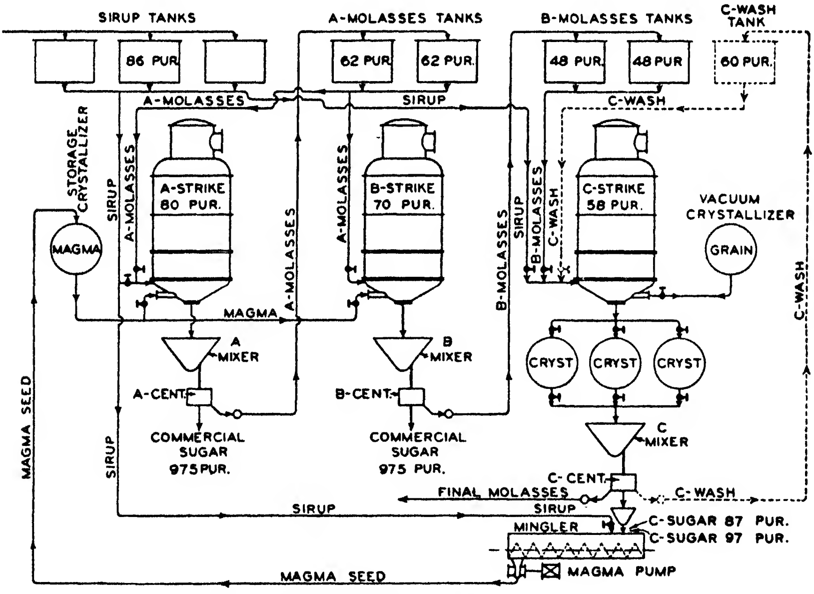

FIG. 70. Diagram of Three-Massecuite System.

the first massecuite is generally boiled on seed grain from the crystallizer strikes mingled with sirup, whereas the mixed strikes are boiled in the same manner, or on a footing from a cut from a first massecuite. The crystallizer massecuites are usually started on a footing of sirup.

The sugar from the first and second massecuites is purged immediately after leaving the vacuum pan and should produce the same grade of sugar. It may be necessary to wash the sugar from the second (mixed) massecuite to accomplish this. The crystallizer massecuite is purged after remaining in the crystallizers from one to four days and yields final molasses and a sugar which may be mingled with molasses, repurged, and washed to give a sugar practically equal to the first sugars, in which form it may be used as seed.

With high-speed centrifugals (see Sec. 291) equipped with exact separation of wash and low-grade molasses, it is possible to dispense with repurging and obtain the same result. The crystallizer sugar may be washed directly in the high-speed machines to 96° polarization or above. The wash is returned to the pan floor for reboiling, and the molasses is turned out as final molasses. The use of the high-speed centrifugal will give these results irrespective of whether the three- or four-boiling system is used.

The three-massecuite method, when double-purged crystallizer sugar mingled with sirup is used for seed, is subject to only one criticism. If the purity of the original sirup is above the desired purity of the first massecuite, the amount of first molasses used to reduce the massecuite purity entails just that much extra boiling, which may amount to from 5 to 10 per cent of extra pan and centrifugal work. With high sirup purities (86° to 88°), such as are met with in tropical practice, this is generally true. (See Figs. 74 and 75.) Where double purging is not used, or where molasses instead of sirup is used for mingling the seed, the objections already referred to are valid; viz., that impurities from the low-grade strikes are introduced into the high-grade strikes.

265. Four-Boiling System. This is shown diagrammatically in Fig. 71 and consists of using the natural drop in purities without boiling any lower-purity molasses back into higher strikes. (This principle of never reversing the downward tendency of the stream of impurities has been the basis of refinery and beet-house boiling systems for many years.) The first strike is started on seed mingled with sirup and completed with sirup only; the second and third strikes are started on similar seed and then take in all the molasses of the preceding strike, but all the crystallizer strikes are boiled on a footing of sirup boiled to grain (not from seed). The figures as shown in the diagram are for an original sirup purity of 86°, but it is evident that with a change in this purity the other purities will change accordingly, the only predetermined purity being that of the crystallizer strike which is held at approximately 58° in Cuban practice. If the sirup purity approximates 80° or lower, only three boilings are needed to reduce the molasses sufficiently, and the system becomes identical with the three-boiling method.

With unusually high sirup purities (say 90° or above), five boilings may be necessary to accomplish the reduction of the molasses to the proper degree.

The four-massecuite method with double purging of crystallizer sugars for seed was developed in Cuba by William B. Saladin, formerly with the Cuban-American Sugar Company. The resulting sugars are of even composition and polarize 96.5° to 97.5° without resorting to washing (except for sugars from third massecuites which require a quart or so of water per charge). The system eliminates reboiling, and the cubic feet of massecuite per 100 lb. sugar produced is lower than for any of the other systems of boiling so far described. The separation of three grades of molasses causes no great difficulty in actual practice.

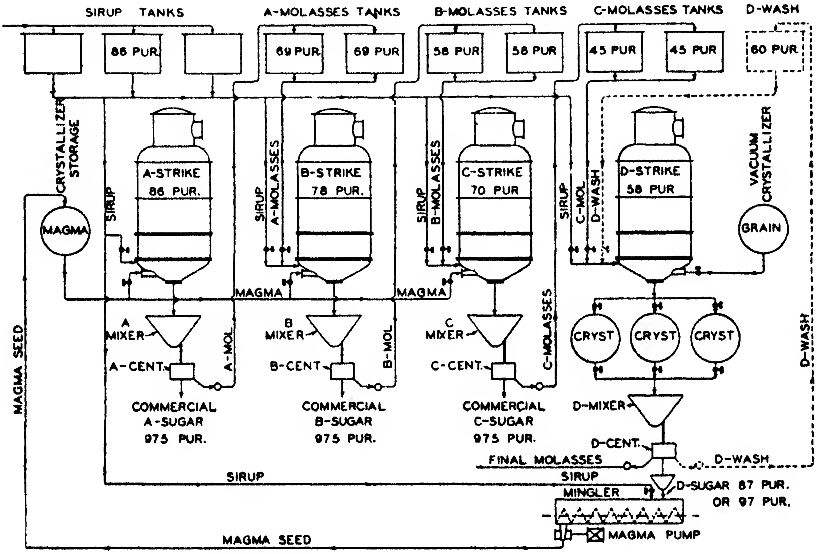

Fig. 71. Diagram of Four-Massecuite System.

Diago[17] gives extensive figures to show that the four-massecuite method produces sugars of better filterability and better color than do the systems previously described.

266. Other Systems. Recent improvements have been used to develop modifications of the systems discarded. The improvements are: (1) making grain on molasses of about 60° purity and (2) double purging or washing the low grades to 97° purity in high-speed centrifugals.

267. New Two-Boiling System. The procedure for the new two-boiling system follows (Fig. 72). The *A* strike takes all the sirup and utilizes all the *B* sugar as seed. There is boiling back of *A* molasses to be able to remove all the available sugar in one operation. Since the *A* molasses must

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[17] *Proc. 7th Annual Cane Sugar Tech. Cuba* (1939), p. 203.

be brought down to a purity of 58°, the *A* strike purity must be held to 78.5° which involves boiling back. This 78.5° is very close to the 80° purity of the *A* strike in the three-boiling system. When the original purity of the sirup is above 80°, this system would not seem to be advantageous.

The grain is obtained by seeding *A* molasses mixed with *B* wash with uniform fine-grain powdered sugar (see Sec. 247), the combination giving 60° purity. There is no difficulty if the pans are provided with control instruments and mechanical circulation. The technic of handling the strike after grain has been made is the same as before.

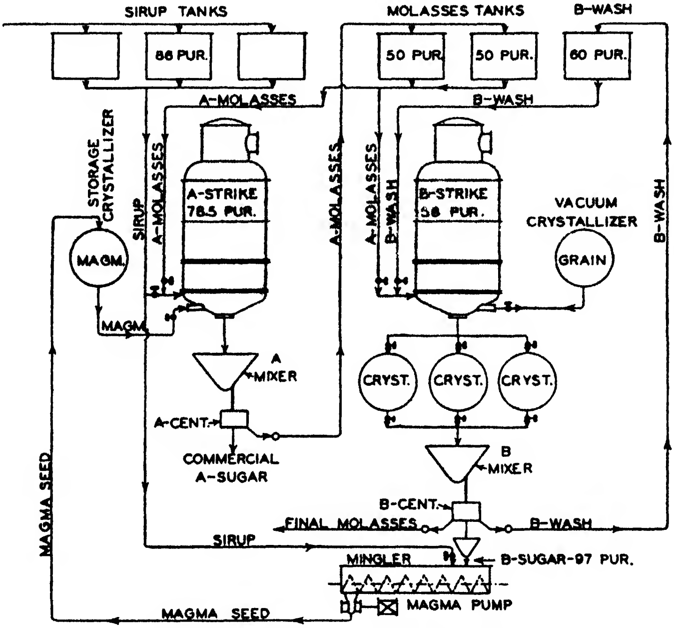

FIG. 72. Diagram of New Two-Massecuite System.

From the crystallizers, the low-grade massecuite is reheated by the use of revolving coils in the mixers. If the machines are high speed, after a conventional period, water is switched on and the wash separated by automatic sirup troughs. The *B* sugar, now of 97° purity, is mingled with sirup, and used as footings for the *A* strikes.

Gomez[18] gives a modification of this method in which he "shock-seeds" instead of "pan-seeds" molasses of 60°–65° purity. Results in actual practice are given which show the practical advantages.

A further discussion of the new two-massecuite method and Gomez's modification by Yearwood and Davies[19] calls attention to the freedom from con-

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[18] *Intern. Sugar J.*, September, 1941, p. 270.

[19] *Intern. Sugar J.*, February, 1942, p. 36.

glomerates in the *C* sugar grained from molasses of 65°–70° purity. The grain is much more regular than in the older methods of graining on high-test sirup.

Gomez' work was done with sirup purities of about 81°. It is evident that with higher-purity sirup the same methods might be employed with a three-massecuite system. Both Gomez and Davies emphasize the value of a projection apparatus for examining the proof in this type of boiling. Davies also recommends a refractometer for use at the pan station.

268. New Three-Boiling System. In the new three-boiling system, similar procedures are used (see Fig. 73). Grain is made by seeding a mixture of molasses at 60° purity. The *C* sugar is purged and washed to 97°. The various strikes are made up as follows.

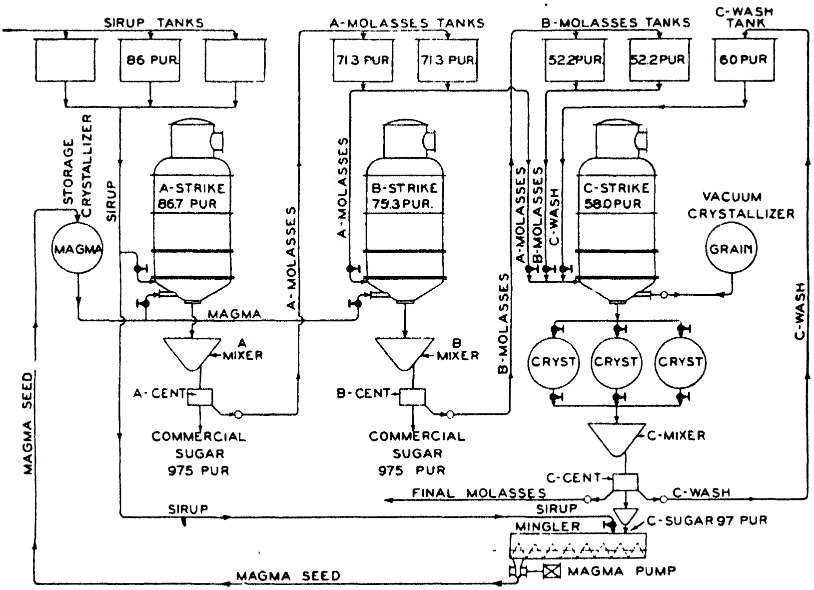

FIG. 73. Diagram of New Three-Massecuite System.

The *A* strike uses all the sirup, and 97° purity *C* sugar made into a magma with sirup. The purities of the strike and the out-going *A* molasses vary accordingly. There is no recirculation. The *B* strike is made up of *A* molasses plus *C* sugar mingled with sirup. A certain amount of *A* molasses is retained for the *C* strike in order to be able to maintain the set purity of 58°.

The *C* strike is made up of all the *B* molasses plus the wash from the *C* centrifugals plus the *A* molasses retained from the *A* strike. Grain is made by seeding a 60° purity mixture as described above. All objections have been eliminated from this system. About 75 per cent of the sugar comes out of the *A* strike, leaving the difference from the *B* strike.

269. Comparative Results with Various Systems. The tabulation on p. 214 shows the percentage of sugar derived from each strike as well as

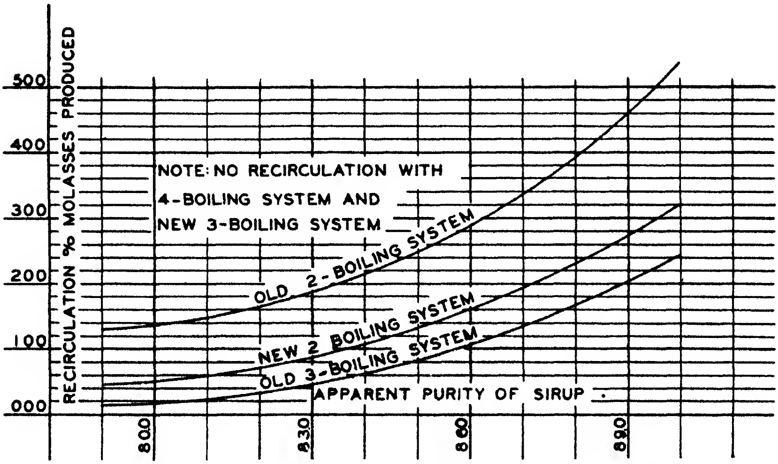

Fig. 74. Molasses Recirculation for Various Boiling Systems.

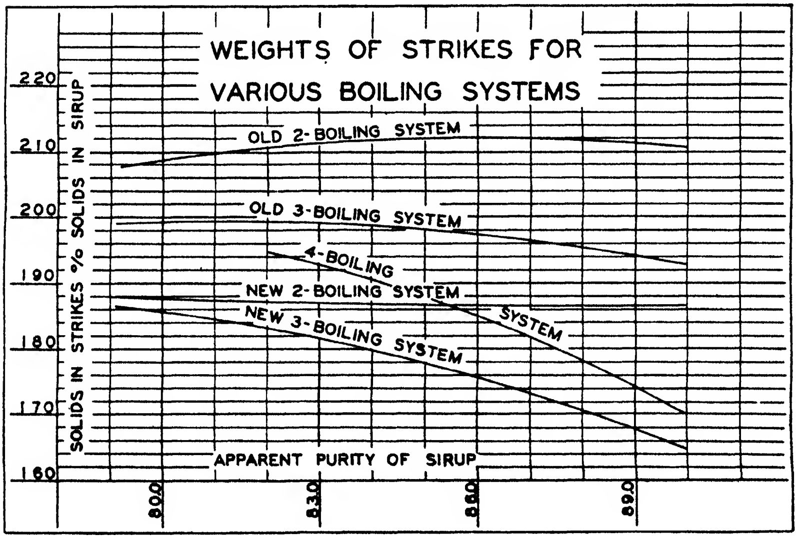

Fig. 75. Weights of Strikes for Various Boiling Systems.

the purities of the strikes for the various boiling systems discussed. The new three-boiling system has the advantage.

The recirculation curve, Fig. 74, shows the percentage of molasses boiled back in the *A* strikes. The old two-boiling system is the worst offender.

Next comes the new two-boiling system. Finally, the three-boiling system is lower than the other two. There is no recirculation by the four-massecuite and the new three-massecuite system.

Figure 75 shows the solids appearing in strikes per 100 lb. of solids in the sirup, as the purities vary. The advantage again lies with the new three-boiling system.

MASSECUITE PURITIES AND PERCENTAGE OF SUGAR PRODUCED FOR THE VARIOUS BOILING SYSTEMS AT DIFFERENT SIRUP PURITIES

Sirup Purities	80.0	83.0	86.0	89.0
Two-boiling system				
A massecuite purity	72.0	72.0	72.0	72.0
Per cent total sugar	100.0	100.0	100.0	100.0
Three-boiling system				
A massecuite purity	80.0	80.0	80.0	80.0
Per cent of *A* sugar	63.0	69.4	76.6	84.0
B massecuite purity	70.0	70.0	70.0	70.0
Per cent of *B* sugar	37.0	30.6	23.4	16.0
Four-boiling system				
A massecuite purity		83.4	86.0	88.7
Per cent of *A* sugar		29.4	44.0	59.8
B massecuite purity		78.2	78.2	78.2
Per cent of *B* sugar		38.8	31.8	23.4
C massecuite purity		70.0	70.0	70.0
Per cent of *C* sugar		31.8	24.2	16.8
New two-boiling system				
A massecuite purity	78.5	78.5	78.5	78.5
Per cent of *A* sugar	100.0	100.0	100.0	100.0
New three-boiling system				
A massecuite purity	82.2	84.0	86.7	89.3
Per cent of *A* sugar	82.1	77.6	74.2	73.1
B massecuite purity	70.3	72.4	75.3	79.0
Per cent of *B* sugar	17.9	22.4	25.8	26.9

270. Boiling to String Proof. This practice has disappeared from present-day factories, which are almost always equipped with crystallizers. The method is variously termed boiling to string proof, smooth, blank, etc., the molasses so boiled generally being 58° to 60° purity.

The addition of carbonate of soda or of lime to low-grade massecuites, particularly string-proof strikes, has been advocated by many, and previous editions of this book stated that soda ash is preferable to lime. An extended study by Grogan[20] in Trinidad gives results which indicate that the use of soda ash in any low-grade massecuite is of doubtful value, in fact actually harmful. He found the lime to have the following advantages: (1) quicker

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[20] *Intern. Sugar J.*, July, 1932, p. 265.

maturing of massecuites, leading to increased recovery and better quality of sugar; (2) elimination of foaming in the pan, reducing possible losses by boiling over; (3) lower cost of the chemicals. He also found that the losses by inversion were excessive in string-proof boiling in the absence of proper pH control.

Molasses is boiled to string proof by drawing it into the pan and boiling it down to the proper density. The determination of this density, by the usual tests of a sample drawn to a string between the finger and thumb, is a matter requiring much judgment and experience. The proof varies with the purity of the molasses, and the kind of containers used in crystallizing the sugar. In this crystallization at rest, as distinguished from the modern process of crystallization in motion, the crystals formed slowly settle to the bottom of the car or tank, gaining slowly in size, but never becoming large. Such sugars range in polarization from 80° to 90°, varying in test with the quality of the molasses and the skill displayed in the pan boiling. A suitable density for the massecuite is approximately 89° to 90° apparent Brix.

SPECIFIC HEAT OF SUGAR SOLUTIONS

Exact figures for the specific heat of sugar solutions are not generally required for routine calculations. The subject has been studied by many investigators, the work of Gucker and Ayres being the most recent.[21] The specific heat which they found for a 65 per cent sucrose solution at 20° C. is 0.6406; for a 50 per cent solution, 0.7213; for 30 per cent, 0.8299; and for 10 per cent, 0.9428. These values and others which they give are almost a straight-line relationship. For practical purposes a direct interpolation gives close approximations for all intermediate strengths of solutions. Earlier studies show figures differing from these by 1.5 per cent.

[21] *J. Am. Chem. Soc.*, **59**, p. 447; *Intern. Sugar J.*, May, 1941, p. 154 [abs.].

CHAPTER 12

CRYSTALLIZATION IN MOTION

I. THEORY AND PRACTICE, PRE-CURING TREATMENT, AND THE EXHAUSTION OF FINAL MOLASSES

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THEORY OF CRYSTALLIZATION IN MOTION

The lowest-purity strike from any of the various boiling systems described in the previous chapter is not in the proper condition to be sent immediately to the centrifugals. The low purity and high viscosity of the massecuite retard the growth and development of the crystals and in order that the exhaustion of the molasses may be nearly complete the temperature of the massecuite must be slowly reduced.

Crystallization in motion is the operation in which a massecuite, after being discharged from the pan, is slowly stirred while it cools from pan temperature to one nearer that of the surrounding atmosphere. Since the solubility of sucrose in the mother liquor decreases with decrease in temperature, sucrose is forced out of solution, and the recovery is thereby enhanced. Continuous stirring is necessary to ensure that fresh crystal nuclei are not formed and that the sucrose is deposited on the existing crystal surfaces. The conditions of the operation are therefore in direct contrast to the isothermal ones which exist in the vacuum pan, although the mechanism of sucrose deposition is the same.

Crystallization in motion is generally applied only to the low-grade massecuites. Recently the advantages of its utilization for higher-purity massecuites, in order to reduce the number of boilings required, have been discussed by Praeger[1] and by Bernard.[2] (See Sec. 285.)

The entire success of the operation depends on adequate control of the factors influencing the solubility of sucrose. These, in turn, mainly influence the rate of deposition of sucrose with the decrease of temperature. A discussion of some theoretical factors influencing solubility follows.

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[1] *Intern. Sugar J.*, **42**, 286 (1940).

[2] *Intern. Sugar J.*, **42**, 327 (1940).

271. The Solubility of Sucrose in Pure and Impure Solutions. The solubility of sucrose in impure solutions may be expressed as a ratio, known as the saturation coefficient.

$$\left.\begin{array}{l}\text{Saturation}\\ \text{coefficient}\end{array}\right\} = \frac{\text{Sucrose/100 water in impure solution of } R \text{ purity at } t^\circ}{\text{Sucrose/100 water in pure solution at } t^\circ}$$

The solubility of sucrose in pure aqueous solution was first established by Herzfeld,[3] but the values of sucrose per 100 water which he published are now accepted with some doubt, especially those for temperatures exceeding 60° C. The literature on the subject is ably reviewed by Verhaar.[4]

The solubility of sucrose in solutions containing cane juice impurities is determined not only by the temperature but also by the purity of the liquor and the nature of the impurities. Thieme [5] in Java and Birkett [6] in Trinidad did not attach any practical significance to the effect of temperature on the saturation coefficient. Thieme [7] gives a table of saturation coefficients for different purity ranges only, and Birkett [8] presents a formula calculated from a statistical examination and relating the true purity, x, to the saturation coefficient, y. It is:

$$x = 178.64y - 80.79$$

In each case it is shown that the saturation coefficient decreases with decrease in purity.

The additional effect of temperature on the saturation coefficient is reported by deVries,[9] who demonstrates that at any one purity the saturation coefficient decreases with decrease in temperature. He also claims that at any one purity there is a constant difference between the saturation concentration of the impure solution, b_t, and the saturation concentration of a pure sucrose solution, s_t, throughout the temperature range. Therefore once this difference, Δb, is established, the saturation concentration of an impure solution at any temperature may be obtained by simple addition: $b_t = s_t + \Delta b$. Davies and Yearwood [10] observe similar variations in the saturation coefficient with variations in temperature for 60° purity Trinidad material, but are unable to agree with deVries that Δb is constant throughout the temperature range. In examining the effect of the nature of the impurities on the solubility of sucrose in 60° purity materials, they show [11] that non-sugars in molasses from different factories on the same island are such as to produce radical differences in the saturation coefficient.

272. Mechanism of Deposition. Silin [12] regards crystallization as taking place in two phases. The first is the diffusion of sucrose towards the

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[3] *Z. Ver. deut. Zuckerind.*, **42**, 181, 232 (1892).

[4] *Archief*, **1**, 325 (1940); *Intern. Sugar J.*, **43**, 50 (1941).

[5] *Studies in Sugar Boiling*, 1928, p. 74.

[6] *Intern. Sugar J.* **37**, 95 (1935).

[7] *Loc. cit.*

[8] *Loc. cit.*

[9] *Archief*, **1**, 543 (1941).

[10] *Intern. Sugar J.*, **44**, 153 (1942).

[11] *Intern. Sugar J.*, **44**, 164 (1942).

[12] *Intern. Sugar J.*, **38**, 32 (1936).

crystal face through a motionless layer of the mother liquor, which is absorbed or jelled on the crystal surface. The rate of diffusion is proportional to the difference in concentrations. The second is the transformation of the sucrose into the crystalline condition, and the rate is proportional to the square of the difference in concentrations.

273. The Velocity of Crystallization of Sucrose. The factors affecting the rate at which sucrose is deposited on crystal surfaces have been examined by Smolenski and Zelenzy.[13, 14] They show that at 95° purity the velocity of crystallization is about half that of pure solutions, and at 80° purity about one-ninth. Silin[15] states that the velocity does not depend on crystal size, quantity of crystals (16.7 to 41.1 per cent), or speed of mixing, provided the crystals are kept in suspension. He reports that beet molasses of 55° purity crystallizes 64 times slower than a pure solution. The question of stirring is discussed by Savinoff[16] who comes to the conclusion that the velocity of crystallization which takes place under industrial conditions is far from the maximum and that a considerable increase could be obtained by intensive mixing. Dubourg and Saunier[17] state that the linear growth of a crystal is proportional to the time and independent of the size, that the rate of crystallization is diminished with increase in pH value, and that glucose inhibits the crystallization of sucrose.

274. Viscosity. Claassen[18] has shown that viscosity does not prevent sucrose from crystallizing, but that it does retard the velocity. King[19] has reported the relationships for Philippine material between viscosity, concentration, temperature, and purity. Micheli and Gyulay[20] present results showing the relationship between viscosity, temperature, concentration, supersaturation, and purity. The viscosity increases with increase in concentration, decrease in temperature, and decrease in purity. On curves of constant supersaturation with increasing temperature, Micheli and Gyulay show that viscosity first decreases and then increases, indicating a temperature of minimum viscosity. This had been previously observed by Bennet and Nees,[21] among others, for saturated solutions.

FACTORY PRACTICE

275. Theory Applied to Practice. Consideration of the above theoretical discussion shows that when a massecuite is cooled, sucrose is forced out of solution by three factors, each of which reduces its solubility in the mother

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[13] *Intern. Sugar J.*, **36**, 448 (1934).
[14] *Intern. Sugar J.*, **37**, 318 (1935).
[15] *Intern. Sugar J.*, **37**, 403 (1935).
[16] *Nauchine Zapiski*, **7**, 416 (1929).
[17] *Facts About Sugar*, **34**, 6, 33 (1939).
[18] *Intern. Sugar J.*, **1**, 250 (1899).
[19] *Intern. Sugar J.*, **35**, 187 (1933).
[20] *Proc. 6th Congr. Intern. Soc. Sugar Cane Tech.* (1938), p. 1094.
[21] *Ind. Eng. Chem.*, **22**, 91 (1930).

liquor. They are (1) the decrease in temperature, (2) the decrease in purity due to progressive exhaustion, and (3) the decrease in saturation coefficient due to decreasing temperature and decreasing purity. As in pan boiling, the requisite driving force required to bring about deposition on the crystal surfaces is the maintenance of an adequate degree of supersaturation. An opposing force is set up by the viscosity of the liquor. With progressive cooling and exhaustion of the mother liquor, the viscosity has been shown to increase, but it tends to decrease because of the decrease in concentration of the mother liquor. The net result in practice, however, is an increase in viscosity during cooling.

The theoretical requirements for proper crystallizer control therefore demand that the rate of cooling should be such that sucrose is deposited by the maintenance of a suitable supersaturation to overcome viscosity. When the rate of cooling is accelerated so that the deposition of sucrose cannot keep pace with the decrease in solubility of sucrose, the degree of supersaturation is increased and may attain a value sufficient to form fresh nuclei or false grain. False grain is especially dangerous at this stage because, as Honig and Alewijn[22] have shown, small crystals grow more rapidly than large ones. Since in the crystallizer false grain seldom grows large enough for proper separation from the mother liquor in the centrifugals, the result is that either the sugar will not cure or the small grains will pass through the screen with the molasses, and losses are emphasized. The maintenance of the mother liquor in the zone of metastable supersaturation, as defined by Ostwald and cited by Schweizer,[23] is therefore of as much importance in crystallizer work as in pan boiling.

Saint[24] states that supersaturation does not fall during cooling and that crystallization is entirely due to the decrease in the solubility of sucrose.

276. Operation. After the massecuite has been struck from the pan, it is run into a crystallizer, the construction and types of which are described later in this chapter. Each crystallizer should be large enough to take one whole strike. This avoids the possibility of running hot massecuite onto other massecuite which has already been partially cooled. Such a procedure is objectionable, and may lead to the re-solution of sucrose, false grain, or a mixture of crystal size which would result in curing difficulties. A bulk of massecuite cools slowly, and if kept in constant motion by stirring, the rate of deposition of sucrose can be safely accelerated by hastening the rate of cooling. Such accelerated cooling is not possible with the air-cooled type of crystallizer, unless fan equipment is used; hence, air-cooled crystallizers should be located in a cool part of the factory so that the cycle time is as short as possible.

With forced cooling, usually by water coils, the rate of temperature drop can be accelerated and controlled by regulating the flow of water. Prac-

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[22] *Intern. Sugar J.*, **33**, 595 (1931).

[23] *Intern. Sugar J.*, **35**, 385 (1933).

[24] *Trop. Agr.*, **8**, 3 (1931); *Intern. Sugar J.*, **33**, 288 (1931).

tice varies from opening the water full as soon as the massecuite is struck to the use of water cooling for the last twelve hours of the cycle only. Elliot[25] advocates a rapid lowering of the temperature so that the supersaturation is increased to accelerate deposition.

Although crystallization in motion is a subsidiary operation to pan boiling, the proper manipulation of the crystallizer station to suit local conditions is of the utmost importance. But the station has its limitations. Skorbilin[26] states that poor pan work cannot be rectified by good crystallizer work. Bromley[27] estimates that of the total low-grade crystallization which takes place, only 30 per cent is accomplished by the crystallizer. Saint[28] attempted to increase the recovery of sucrose in the crystallizer by submitting three similar massecuites to different treatments, but he was unable to show any difference.

Van der Ent[29] followed the course of massecuite exhaustion in a crystallizer and made observations with suitably placed thermometers and a pair of electrodes. He thereby suggested that in controlling the rate of cooling not only should the temperature of the massecuite be considered but also conductivity measurements. The conductivity method has already been applied successfully to pan boiling control, but this is the first recorded instance of its application to crystallizer control. He also emphasizes that the addition of water or of diluted molasses slows down exhaustion considerably.

Salinas[30] shows that considerable inversion takes place in the crystallizer, but he is not supported by Saint.[31] Early research has shown that while in the crystallizer the massecuite becomes heavily infected with microorganisms. Recent work has been carried out by Millstein and others.[32]

PRE-CURING TREATMENT

277. Dilution versus Heating. Viscosity is one of the main factors influencing the rate at which molasses can be separated from the crystal sugar under constant mechanical conditions. Hence any expedient which will reduce the viscosity will increase the rate of curing. After the massecuite has been cooled in the crystallizer, the mother liquor is still supersaturated with respect to sucrose. Its viscosity can therefore be reduced, without re-solution of sucrose, by one or both of two procedures: dilution or heating. The practice of adding water to highly concentrated massecuites prior to curing was well established in the industry before the more

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[25] *Assoc. Hawaiian Sugar Tech.*, 1936, p. 261.

[26] *Nauchine Zapiski*, **5**, 203 (1927).

[27] *Assoc. Hawaiian Sugar Tech.* 1937, p. 49; *Intern. Sugar J.*, **40**,, 441 (1938).

[28] *Trop. Agr.*, **10**, 68 (1933); *Intern. Sugar J.*, **35**, 309 (1933).

[29] *Intern. Sugar J.*, **43**, 354 (1941).

[30] *Intern. Sugar J.*, **21**, 147 (1929).

[31] *Intern. Sugar J.*, **33**, 288 (1931).

[32] *Facts About Sugar*, **36**, 3, 28 (1941).

recent researches were published. But all operators admitted that with the crude methods then employed, excessive solution of sucrose took place, resulting in an increase in the purity of the molasses. The practice of heating, or warming, the massecuite is of more recent origin. It was suggested as a result of work on the sucrose status of massecuites immediately before curing. Pre-curing treatment, as understood today, is based on adequate knowledge of the sucrose solubility characteristics of the mother liquor. The terminal point of dilution or of heating is the concentration at which the liquor is just saturated at that temperature or the temperature at which the liquor is just saturated at that concentration. Warren[33] and Barker[34] were among the earlier workers who saw the practical advantages of warming to the saturation temperature as opposed to diluting to the saturation concentration. Since then much work has been reported. Mitchell and Behne,[35] in a laboratory investigation, determined the effect of viscosity on centrifugaling. They show that dilution to the saturation concentration may result in a lower viscosity than reheating to the saturation temperature, but they admit that dilution is not a simple operation in the factory. Glick[36] concludes that it is almost impossible to add water to a massecuite in the factory without re-solution of sucrose, and he gives general notes on the purity rise obtained due to warming and dilution. McCleery's results[37] favor warming since he finds it difficult to control water additions. He points out that in warming there are two important factors: (1) no local overheating should be permitted, and (2) there should be no loss of massecuite temperature while curing. He suggests control by means of properly placed thermometers and the Saturascope[38] (see Sec. 668). In another paper, McCleery[39] gives factory data on the treatments used in Hawaii together with capacities of equipment, heating surfaces, etc. Micheli and Gyulay[40] show that the viscosity of some molasses is lower at the saturation concentration than it is at the saturation temperature.

Jenkins[41] in following up the work of Mitchell and Behne[42] shows that it is impracticable to dilute to a supersaturation less than 1.09 but that re-solution can be definitely avoided by reheating with water at the saturation temperature of the mother liquor. McCleery[43] has studied the purity-viscosity relationships from 30° to 55° C. saturation temperatures on Hawaiian final molasses and determined the working viscosity for each factory.

[33] *Sugar News,* **10,** 317 (1929).
[34] *Intern. Sugar J.,* **33,** 610 (1931).
[35] *Intern. Sugar J.,* **40,** 470 (1938).
[36] *Intern. Sugar J.,* **37,** 158 (1935).
[37] *Assoc. Hawaiian Sugar Tech.,* 1934, p. 127; *Intern. Sugar J.,* **37,** 198 (1935).
[38] *Intern. Sugar J.,* **38,** 189 (1936).
[39] *Intern. Sugar J.,* **37,** 279 (1935).
[40] *Loc. cit.*
[41] *Intern. Sugar J.,* **42,** 259 (1940).
[42] *Loc. cit.*
[43] *Assoc. Hawaiian Sugar Tech.,* **40,** 377 (1939); *Intern. Sugar J.,* **39,** 363 (1937)

Waddell[44] states that either reheating with live steam or rapid mixing of dilution water will result in a higher recovery. Claassen[45] advocates the addition of water. Stevens[46] has patented a hot mingling process and equipment on which Gutleben[47] reports. Hadden[48] also has a patent for similar equipment. Penning has patented[49] an apparatus for a continuous operation of cooling and heating low-grade massecuites. Ralston's heating system is shown in Fig. 76.

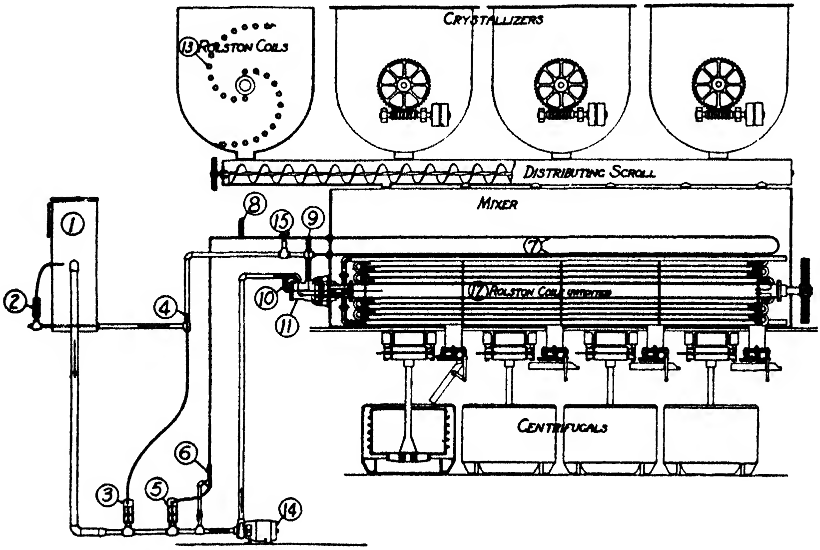

FIG. 76. Ralston Crystallizers and Heating Coils.

278. Heating Preferable. It therefore appears that the consensus of practical opinion is in favor of warming. This may be carried out wherever convenient, in the crystallizer, or between it and the centrifugals. It is obvious that in order to obtain the benefit of the reduction in viscosity, the temperature must be maintained up to the time that the basket is spinning, as emphasized by McCleery.[50] The cooling coils in the crystallizer, special coils in the gutter, or special coils in the mixer have been used separately or in combination for warming. Excessive heating is as dangerous as excessive dilution, but as has been shown, heating is easier to control than dilution. Generally, the heating medium is warm water, such as evaporator con-

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[44] *Intern. Sugar J.*, **40**, 377 (1938).
[45] *Intern. Sugar J.*, **41**, 32 (1939).
[46] *Intern. Sugar J.*, **39**, 199 (1937).
[47] *Intern. Sugar J.*, **40**, 282 (1938).
[48] *Intern. Sugar J.*, **39**, 243 (1937).
[49] *Intern. Sugar J.*, **41**, 323 (1939).
[50] *Assoc. Hawaiian Sugar Tech.* (1934), p. 127; *Intern. Sugar J.*, **37**, 198 (1935).

densate, of suitable temperature. A thermostatically controlled tank acts as a supply for the coils. (See Fig. 76.)

THE EXHAUSTION OF FINAL MOLASSES

Though control of the boiling process appears to be accurate enough when based on a consideration of purities, it is recognized as not being altogether satisfactory for measuring the degree of exhaustion of the final molasses. On fundamental grounds, it is obvious that both the concentration and the temperature to which the massecuite is cooled will vitally affect the degree of exhaustion. Geerligs [51] showed in his classic thesis that the reducing sugar content and the ash content were of some influence in determining the amount of sugar which could be extracted. He suggested that the reducing sugar-ash ratio should therefore be considered in conjunction with the purity figure. This ratio is based on the observation that reducing sugars salt out sucrose from molasses and that ash increases the amount of sucrose retained in solution. Sijlmans [52] states that the effect of ash can be ignored and that only reducing sugar exerts any influence. In place of the reducing sugar-ash ratio he suggested reducing sugar per cent non-sugars. This criterion was used in Java until 1929, when Thieme [53] after some extensive experiments concluded that there was only a slight correlation between reducing sugar per cent non-sugar and exhaustability. He found that the original reducing sugar-ash ratio was a more valuable guide, that ash per cent non-sugar was better, and that alkali per cent non-sugar was the most valuable of all. When temperature and concentration are constant, he states that exhaustion depends on the relation of inorganic to organic non-sugars, the alkalis exerting the greatest influence. Since the determination of the alkalis is tedious, ash per cent non-sugars is suggested for control purposes. Thieme [54] also states that cultural conditions of the cane exert a greater influence on exhaustability than clarification, and discusses [55] the 1929 conditions in East Java when the factories were yielding high-purity molasses. Later, Thieme [56] suggests as a reference point of exhaustability the purity of the molasses at 85° Brix saturated at room temperature. This is chosen because no Java factory is able to attain it. Davies [57] discusses the requirements of a measure of exhaustion, and after showing that at one factory the ash per cent molasses did not change significantly over four years, he suggests as a practical measure for use in the control laboratory the correlation between total sugars per 100 water and

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[51] *Sugar Cane* (1893), p. 146.

[52] *Archief,* **111,** 413 (1942).

[53] *Archief,* **38,** 115 (1930); *Intern. Sugar J.,* **33,** 244 (1931).

[54] *Loc. cit.*

[55] *Archief,* **38,** 713 (1930); *Intern. Sugar J.,* **33,** 131 (1931).

[56] *Archief,* **39,** 361 (1931); *Intern. Sugar J.,* **33,** 408 (1931).

[57] *Intern. Soc. Sugar Cane Tech., Bull. 121* (1932); *Intern. Sugar J.,* **34,** 242 (1932).

per cent water. Sijlmans[58] made a statistical study of the analyses of molasses from ninety Java factories. Using the solubility tables of Jackson and Silsbee for pure sucrose and sucrose-invert sugar mixtures, he compiled a table which represents the theoretical solubility for Java molasses. The table is based on sucrose per 100 water at $t°$ and invert sugar per 100 water. When the sucrose per 100 water in a molasses is equal to that shown in the table, then the molasses is in equilibrium. He then proceeds to give a formula which expresses the practical exhaustability. Behne[59] applied Sijlmans' work to Queensland molasses and concluded that it is of considerable value. Birkett[60] considered the crystallization of sucrose from cane juice theoretically and practically, using phase rule methods and diagrams. He demonstrates the practical application. Micheli and Gyulay[61] object to phase rule methods because under factory conditions equilibrium is not attained. They devised a practical crystallization test and proposed:

$$\text{Exhaustability } (E_0) = 100Q_L$$

where Q_L = lowest purity obtainable in a well-equipped factory.

$$\text{State of exhaustion } (E) = \frac{100 - Q}{100 - Q_L}$$

where Q = actual purity of molasses.

They state that under the conditions of their test with any one material, only one factor, concentration, has any effect on purity. They expressed the concentration as the impurity-water ratio because it is not affected by crystallization, and it can be determined without separating the crystals. With their molasses, a rise of 0.2 in the impurity-water ratio led to a decrease of one unit in final purity. At constant impurity-water ratio (3.5) the final purity is dependent on the saturation coefficient.

II. TYPES OF CRYSTALLIZERS (G.P.M.)

279. Air-Cooled Crystallizers. Crystallizers first came into use in the beet sugar industry in Europe about 1880, but the cane sugar industry did not adopt the process generally until about twenty or twenty-five years later. The earliest type of crystallizers consisted of either U-shaped, open, horizontal containers or horizontal, closed cylinders. In either type a shaft and helix rotated slowly (one turn every two or three minutes). These crystallizers depended entirely on air cooling by radiation. Many are still in use but during the past fifteen years the rapid-cooling types described below have superseded these to a great extent.

[58] *Archief* (1934), p. 167; *Intern. Sugar J.*, **36**, 437 (1934).
[59] *Intern. Sugar J.*, **38**, 174 (1936).
[60] *Intern. Sugar J.*, **38**, 12 (1936).
[61] *Intern. Sugar J.*, **38**, 460 (1936).

280. Water-Cooled Crystallizers. More modern types of crystallizers (either open or closed) are water cooled and may be divided into four general classes:

1. Water-jacketed crystallizers in which the cooling surface is entirely on the outside of the vessel.
2. Crystallizers with fixed circulating coils.
3. Crystallizers with rotating circulating coils in which the cooling surfaces are part of the stirring apparatus.
4. Rotating crystallizer with fixed cooling coils.

Detailed studies of various types of crystallizers by Tromp[62] and Farnell[63] give diagrams and descriptions of most of the designs now in use. To illustrate the principles involved in the general classifications the more common designs will be referred to.

Since control of supersaturation in a crystallizer is by cooling, it must be uniform and gradual without local excess cooling. In crystallizers provided with water-cooled coils or surfaces such local overcooling may result. To prevent this it has been recomended that the cooling water be passed in series through the units, the water entering the coolest crystallizer and leaving the hottest. Tromp[64] gives a discussion of the theory of counterflow and intermittent cooling which may be consulted.

Water-Jacketed Crystallizers. An early arrangement consisted of an extra shell on the outside of a cylindrical crystallizer through which water could be circulated. Some of these are still in use but this type is no longer built as the first cost is greater for the water jacket than for stationary coils.

Stationary Cooling Coils. A number of designs of this type have come into use during the last fifteen or twenty years, the best known being the Kopke type in which the coils are at right angles to the shaft and placed about 18 in. apart with the paddles turning between the fixed coils. Early reports[65, 66] showed that molasses of 3° or 4° lower purity could be secured in about half the time with a crystallizer equipped with these cooling coils as compared with the ordinary type, but these reports seem somewhat optimistic for fixed coils. Usual practice is to cool the massecuite to 90°–95° F. before sending it into the centrifugals. In refinery work the Kopke coils have not given such good results as are reported by the writers referred to above; in fact, in one refinery a comparison over a long term between old-style water-jacketed crystallizers and crystallizers equipped with Kopke coils shows the coils to be no more effective than the water jacket.

Crystallizers with Revolving Coils. In this form the circulating coils of different types are attached to a hollow rotating shaft through which water is circulated at the proper temperature, thereby bringing the cooling surface

[62] *Intern. Sugar J.*, April, 1934, p. 154.

[63] *Intern. Sugar J.*, July, 1934, p. 264.

[64] *Loc. cit.*

[65] Keller, *Proc. 2d Congr. Intern. Soc. Sugar Cane Tech.* (Havana, 1927).

[66] Biela, *Hawaiian Sugar Tech.*, 1930.

in more direct contact with the entire mass. There are many designs, one of which was patented by Herisson of South Africa and is widely used in Natal. Cooling is reported as very effective.[67] The Ralston crystallizer is another rotating type. It has a hollow shaft and a cooling surface of tubes with a spiral distribution lengthwise of the entire crystallizer (Fig. 76).

The Kilby crystallizer has a cylindrical coil about two-thirds the crystallizer diameter which is cut in two. One section is mounted eccentric to the shaft so that the edge of the turns just clear the shell of the crystallizer; the other half is also eccentric but 180° away. This crystallizer is rugged and may be used for cooling very heavy massecuite (Fig. 77).

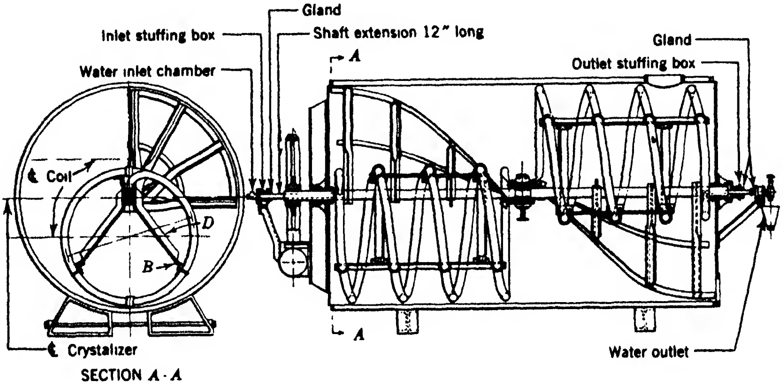

Fig. 77. Kilby Crystallizer.

281. Werkspoor Continuous Crystallizer. An ingenious design is that of the Werkspoor which is intended for continuous operation, the hot massecuite entering at one end and leaving at the other. It consists[68] of an open container, either U shaped or bottle shaped, having a revolving cooling and stirring system with a shallow drum of large section riveted to which are a number of cooling pockets. The pockets are coupled by suitable pipe connection inside the central drum and through them cooling water circulates (Fig. 78). The transportation of the massecuite from one end of the crystallizer to the other is effected by a screw-shaped element independent of the cooling pockets. The cooling water is circulated in the opposite direction to that of the massecuite, resulting in true countercurrent action. A uniform fall in the temperature during the cooling period is reported. This design has been tried in several factories in Java and the results reported are said to be even better than with the LaFeuille crystallizer described below.[69]

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[67] *Intern. Sugar J.*, February, 1938, p. 62.
[68] R. Bonath, *Intern. Sugar J.*, June, 1930, p. 306.
[69] *Intern. Sugar J.*, June, 1931, p. 283.

Werkspoor for White Sugars. Experiments by Stare in Swedish beet sugar refineries[70] showed that the Werkspoor continuous crystallizer can be used in a rather novel manner for white sugar work. Refined sugar massecuites are mixed with a saturated sugar solution (saturated at 90° C. or 194° F.) made by dissolving second sugar in the washings from the white sugar centrifugals, and the whole run through the Werkspoor. A high output of refined is obtained, namely, 69 per cent of solids in one boiling. In this way the total water evaporated is reduced from 81.4 to 50.4 per cent of the sucrose entering the refinery and the total magma purged reduced from 46.0 to 29 per cent of the sucrose input. Capacities are placed at 100 tons white sugar massecuite per twenty-four hours in each Werkspoor of 120 cu. ft. capacity having 500 sq. ft. cooling surface. The actual cooling time is one hour.

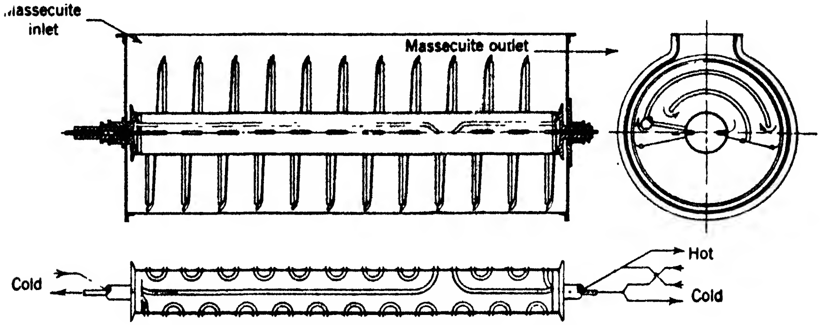

Fig. 78. Werkspoor Continuous Crystallizer.

282. LaFeuille Rotary Crystallizer. This is an improved form of crystallizer consisting of a rotating shell containing cooling tubes. The cooling surface consists of 2-in. tubes extending the full length of the crystallizer parallel to the axis of rotation divided into two or four coils arranged to make the temperature uniform all through the massecuite. The crystallizer is stopped while the filling and emptying are done through suitable shear gate valves. Extensive comparisons made in Java by Honig and Alewyn show the LaFeuille to be about six times as effective as a standard type crystallizer without cooling coils, cooling being complete in five or six hours. However, these same Java authorities found later[71] that, following very rapid cooling in the LaFeuille, continued stirring at ordinary temperatures gave improved results: greater yield of crystals, lower-purity molasses, and less sticky sugar. In some cases this after-mixing period of four to seven hours more than doubled the crystal yield obtained by cooling in the LaFeuille. It would appear from this that the cooling in the LaFeuille may be too rapid for the crystallization to keep pace with the increasing supersaturation.

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[70] See Farnell, *Intern. Sugar J.*, July, 1934, p. 269.

[71] Honig and Alewyn, *Intern. Sugar J.*, November, 1931, p. 546 (abs.).

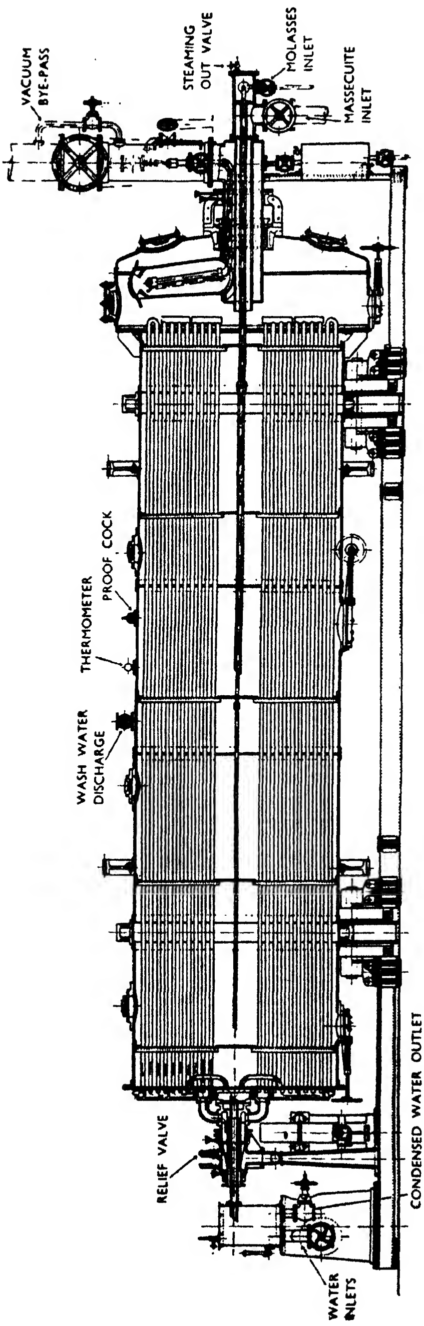

Fig. 79. LaFeuille Crystallizer Pan.

Because of the very high first cost of the LaFeuille its use as a crystallizer has been limited.

LaFeuille Crystallizer Pan. A more recent development of the LaFeuille serves not only as a crystallizer but also as a vacuum pan (Fig. 79). The general appearance is much the same as the original LaFeuille crystallizer, but it is fitted with vacuum and steam connections and there are numerous mechanical improvements.[72] Several mills in the Philippines have these crystallizer pans and have reported that lower-purity molasses, less volume of molasses, and easier purging of lower grades result.

283. The Pitcairn Double Helix Crystallizer. The Pitcairn crystallizer operates with air cooling only. The idea developed from observation that ordinary open U-type crystallizers if only half full of massecuite would cool much more rapidly because of exposure of the massecuite to air, evaporation, etc. W. G. Hall suggested superimposing one helix on another to obtain the same results with a full crystallizer. The arrangement gives the following advantages: [73]

1. Complete circulation and an even distribution of temperature.
2. Evaporation of water during cooling which favorably influences growth of the crystals.
3. Rapid cooling due to exposure to the air.
4. Purging of massecuite at higher temperature with same exhaustion of molasses.
5. More effective addition of water or molasses, if such procedure is carried out.

284. Cooling in Circulator Pan (Webre).

Where densities do not exceed 96° Brix the mechanical circulator pans [Sec. 230] have been used for cooling. The cycle takes from 5 to 6 hours, reducing the temperature from 155° F. to 110° F. The method of cooling, which is covered by patents, is simple and avoids temperature shocks. When the strike is finished, steam is shut off, the condensate drains are closed as well as the vents, and the calandria is filled with water slightly warmer than the strike. Vacuum is now raised on the upper part of the calandria, and the massecuite being circulated mechanically inside of the tubes dissipates its heat by causing the water to boil under high vacuum. The cooling is smooth, easy, continuous and without shock, as the temperature of the water is dependent upon the vacuum in the calandria, which progresses slowly to a maximum at the end of the operation.

This plan, though successful, has many limitations because of the extra pan capacity required and the excessive power used during the cooling process.

285. High-Grade Massecuites to Crystallizers. This possibility has been discussed by Honig and Alewyn [74] for Java and more recently by Praeger for Queensland practice.[75] The latter finds that an increased drop of 6° of

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[72] LaFueille and Kaar, *Intern. Sugar J.*, October, 1933, p. 387.

[73] R. C. Pitcairn, *Intern. Sugar J.*, May, 1931, p. 234.

[74] *Intern. Sugar J.*, June, 1931, p. 283.

[75] *Intern. Sugar J.*, August, 1940, p. 286.

purity may be obtained for each grade of massecuite with the Werkspoor crystallizer, eliminating one boiling from the four-boiling system without any reboiling of molasses. He believes that the colder sugar purged from the cooled massecuites will be less likely to harden on storage; Praeger also suggests after-cooling of high-test strikes in the Webre circulator pan. (See Sec. 284.)

The elimination of one boiling would result in 15 per cent less total massecuite according to Honig and Alewyn, and the practical possibilities would seem to be great enough to warrant the adoption of crystallization for high-test strikes.

286. Froth "Fermentation." When low-grade massecuites are boiled at high temperatures they often foam and run over the sides of the containers. The foaming may begin immediately or some hours after striking. This phenomenon is termed fermentation or froth fermentation. Only in recent years has there been a fairly definite knowledge of the cause of the foam. It is not due to fermentation, that is, to the activity of bacteria or other microorganism, but to the decomposition of certain salts and organic non-sugars.

Herzfeld states that Kraisy has shown that Lafar's [76] results on the reaction of the invert sugar with amino acids, e.g., glutamic acid, splitting off carbon dioxide, is the cause of foaming. Massecuites (sugar beet) foam suddenly when a temperature of about 65° C. (149° F.) is reached. Kraisy explains the phenomenon by the carbon dioxide remaining in supersaturated solution until the formation of sugar crystals catalyzes its liberation. It is generally stated that lime will stop the foaming by combining with the CO_2 but the efficacy of this has been questioned.[77]

The usual method of stopping foaming in a cane sugar factory is to pour water upon the surface of the massecuite. This reduces the viscosity and facilitates the escape of the gas. Cane massecuites that have not been heated above 156° F. do not usually foam. A crystallizer should always have provision for escape of the gases or preferably should be uncovered except while discharging by air pressure. The volume of the crystallizer should be about one-third larger than the volume of a strike to take care of swelling of the massecuite in the crystallizer. The same conditions as those observed in beet sugar manufacture are probably the cause of foaming in cane massecuites. J. A. Ambler [78] states that the action of amino acids on glucose forms CO_2, melanoidins and other substances, promoting frothing by lowering the surface tension.

This subject is considered at greater length in the chapter on molasses (see p. 252) since it is now recognized that the action in massecuites is the same as that which takes place in blackstrap molasses during storage.

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[76] *Z. Ver. deut. Zuckerind.*, **64**, 543.

[77] Grogan, *Intern. Sugar J.*, July, 1932, p. 265.

[78] Before the American Chemical Society, Swampscott, Mass.; *Facts About Sugar*, Sept. 29, 1928 (abs.).

CHAPTER 13

PURGING, PACKING, AND WAREHOUSING THE SUGAR

CENTRIFUGING

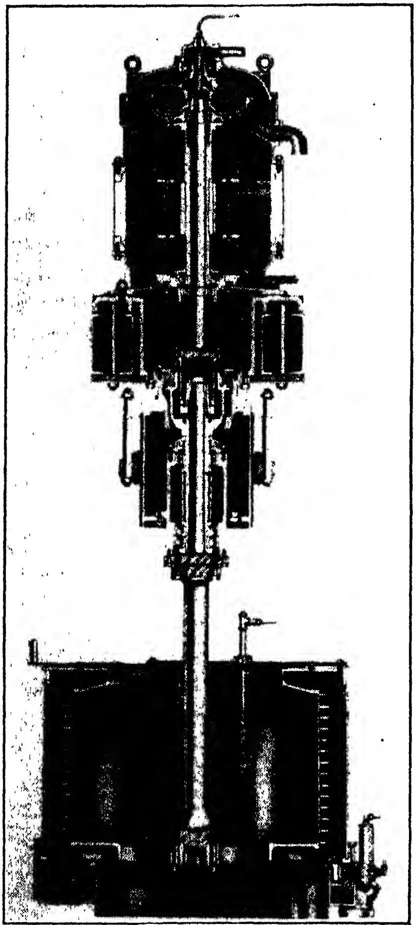
287. Centrifugal Work. The massecuites as delivered from the vacuum pans or from the crystallizers go first to a mixer, which is in general a trough-like container in which revolving arms prevent the crystals from settling. The sugar crystals in the massecuite are separated from the surrounding molasses by centrifugal force in a machine called a centrifugal (Fig. 80), which is essentially a perforated drum so arranged that it may be revolved at high velocity on a vertical axis known as the spindle. The basket revolves within an iron casing called the curb, which serves to catch the molasses and lead it to the conduit. The basket is lined first with a brass wire backing screen of ten meshes to the inch inside of which is a perforated sheet of brass or bronze known as the lining. The use of two backing screens or of a brass grid in conjunction with the backing screen permits of more rapid drainage of molasses.

The older type of lining has 400 to 625 round perforations to the square inch, but a newer type has slotted apertures 0.012 in. by 5/32 in. tapered from the inside outward to more than twice the inside width in order to give a freer flow of the molasses. The slotted linings permit the use of much heavier metal than the old round-hole type, but a tendency for the slots to widen with use, allowing the escape of fine crystals to the molasses, has led many users to prefer the round-hole type. The older centrifugal baskets were 30 in. in diameter and occasionally 36, but all modern factories use 40-in. centrifugals 24 in. deep or more, which have a speed of at least 1000 revolutions per minute. As will be noted later, modern practice has developed machines of much higher speeds.

Each battery of centrifugals has its own mixer, and the charge of massecuite is fed to each machine through its own special spout on the side or bottom of the mixer. The charge generally is added while the centrifugal is in motion. The massecuite rises in the basket because of the centrifugal force and distributes itself over the perforated lining through which the molasses passes while the crystals are retained. The spinning or purging is continued until the sugar is practically free of molasses after which the sugar may or may not be further freed of the molasses film by spraying it with water. In raw sugar manufacture the sugar is not generally washed, but in the production of white sugars and high polarization yellow sugar washing is invariably practiced.

Centrifugals are frequently arranged with double gutters so that the higher-purity wash and the lower-purity "green" sirup or molasses may be separated.

The operator accomplishes this in the older-style machines by throwing a lever which actuates a tilting bucket just before starting the water spray on the sugar, so that the molasses or mother sirup goes to one gutter and the high-test wash water is directed to another gutter. This arrangement is not entirely satisfactory on account of the lag of the heavy molasses which stays in the curbing so that part mixes with the wash. Modern high-speed machines have a more effective method of separation as will be described later. Double purging, described in Sec. 290, is also employed to effect the classification.



Western States Machine Co.

FIG. 80. Motor-Driven High-Speed Centrifugal.

Each sugar drier or purger manipulates two and sometimes three or more centrifugals. A 40-in. by 24-in. centrifugal, driven by belts and working with one operative to two machines, should purge from 5000 to 6000 lb. of 96° polarization sugar per hour, the quantity varying with the skill of the operative, the quality of the massecuite, and the facility with which the centrifugal may be started and stopped. This capacity may be greatly increased by the use of mechanical dischargers, and self-discharging baskets will give double the capacity cited above.

The discharger is a plow arrangement that is lowered into the basket and directed against the wall of sugar. The basket is revolved slowly against the plow, which cuts down the sugar and pushes it out of the machine by the bottom valve. There are many makes of these, all of the same general design, and their use is common in cane, refinery, and beet house work.

288. Self-Discharging Baskets. The self-discharging basket has a steep conical section at the bottom and may or may not have a discharge valve. The commoner type is without discharge valve and has a deflector on the spindle or shaft to direct the massecuite toward the wall of the basket. In the other type, the valve may be raised while the centrifugal is running. When the sugar has been purged and the centrifugal is stopped the sugar usually falls out without assistance. In the other form, it is necessary to lift the valve before the centrifugal is stopped. These machines are used only with freely purging sugar of strong grain such as high-grade massecuites and refinery affination magmas. They are not used for refined sugars because of the danger of contaminating the purged sugar with drippings of massecuite. The high-speed centrifugals described later have largely displaced the self-discharging type of basket.

289. Purging White Sugar. From the nature of the product white sugars require special care in purging. The sugar is washed in the centrifugal with large quantities of water (preferably heated to 130° F. or higher), 2 to 6 gal. being required per charge in a 40-in. machine. Automatic washing devices for insuring exact measurement and distribution of the water are customary in refining and white sugar practice, and dischargers or plows, as described above, are universal. A small quantity of ultramarine blue was formerly used in the wash water to kill the yellow tinge in off-white sugars, but its use is now contrary to the United States Pure Food regulations.

290. Double Purging. Two sets of centrifugals are necessary for double purging. The sugar is purged in the first set without washing or with very little water. It is then "cut down" into a mingling device and formed into a magma with sirup from the second purging. The magma is elevated to the second mixer and is purged in the second set of centrifugals with thorough washing.

Double purging serves a twofold purpose: it separates the dark, low-purity molasses of the first purging from the rich, light-colored sirup of the second. The mingling process mixes the crystals thoroughly with the light-colored sirup. The friction of crystal against crystal promotes the removal of the adhering film of molasses.

Double purging was introduced into Cuba by Spencer to facilitate the handling of crystallizer sugar in the production of one grade of sugar and final molasses. The crystallizer sugar is purged without washing, and the molasses from it is final. The sugar is discharged into a mingling screw conveyor and transferred to the mingler proper. The conveyor screw is lubricated with first molasses or sirup diluted to about 78° Brix, and this molasses or sirup is also used in the mingler to form the magma. The mingler is simply a mixer, generally with a cut-flight conveyor. The magma is pumped to the first machine's mixer for immediate purging or to a storage crystallizer to be used as a footing or seed in boiling massecuites.

291. High-Speed Centrifugals. The speed of a 40-in. centrifugal has for many years been limited to 1100–1200 rpm., but during the past decade machines have been developed with speeds of 1600, 1800, and even 2000 rpm. These high-speed centrifugals (referred to as "high-gravity factor" centrifugals by some British manufacturers) have been made possible through improvements in bearings and high-tensile alloy steels, together with improved form of drives similar in some respects to those used in automobiles. It follows that ordinary centrifugals cannot safely be speeded up beyond the rated speeds fixed by the manufacturers and that only specially designed and specially built machines can be carried to this high rate. A high-speed motor-driven machine of recent American design is shown in Fig. 80.

The Roberts centrifugal as illustrated[1] is driven by a vertical fluid clutch motor with torque control features; the head is of heavy-duty construction with water-cooled air-operated brake; the curb is of split construction with air-operated sirup separator. All phases of the centrifugal cycle are automatically controlled by a combination electric-air control. During discharging and loading, the fluid clutch and air brake are manually controlled and the time cycle is manually started by the control lever on the control box near the centrifugal. Pilot lights indicate when washing takes place and when the sirup separator trips.

The fluid coupling, mounted above the motor and integral with it, is made operative or engaged by feeding oil to its inlet. Oil is continuously discharged from the coupling and returns to a cooling tank. In order to disengage the coupling, all that is necessary is to stop the oil flow to the coupling. This oil flow to the coupling is controlled by the control handle, the electric timer, or the torque control. The torque control can be regulated in the field to obtain any acceleration rate desired (within the power capacity of the machine).

The motor is operating all the time at top speed, and the acceleration rate which is constant from one cycle to the other can be readily changed. The acceleration power is constant from start of cycle to top speed, eliminating the excessive power variations obtained in regular motor-driven centrifugals. No wear takes place in the coupling because the oil is doing all the work and absorbs the heat. One oil pump and oil tank are required per group of centrifugals.

Gear-driven high-speed centrifugals are driven in a group of six machines or less, through spiral bevel gears from a constant-speed horizontal line shaft which is belt connected to a motor (250 hp. for six machines). A water-cooled clutch on each machine attaches it to the shaft as required. Water-cooled brakes and a forced oil circulator system are also features. Very rapid acceleration (45–50 seconds) calls for a high power input which falls to a much lower figure when full speed has been attained. The figures quoted by Gutleben when purging raw sugar magma for affination work[2] in refining with hot mingling were 70 hp. and 8 hp. respectively, but these figures may vary considerably with different conditions. (See below.)

Walter E. Smith[3] describes the development of high-speed machines in Hawaii for purging low-grade massecuites. Belt-driven machines at 1600 rpm. were found to have double the capacity of the same-sized centrifugals operating at 1200 rpm. Direct-driven electric machines with 30-hp. motors were

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[1] Description supplied by the manufacturer, Western States Machine Co.

[2] Gutleben, *Chem. Met. Eng.*, **44**, 14 (1937).

[3] *Intern. Sugar J.*, June, 1937, p. 237.

found satisfactory if acceleration to 1700 rpm. took four to five minutes. Hydraulic drive using water at 400 lb. pressure gave speeds in excess of 1800 rpm. A steam turbine drive operating at 4500 rpm. through a right-angled reducing gear was so arranged as to give 1800–1850 revolutions in the machine. Lower molasses purities are described as resulting with the use of these high-speed machines, mainly because it is possible to work with massecuites of higher density, i.e., molasses of higher viscosities.

One effect of the higher speed, besides increasing the centrifugal force more than twice, is that of heating the massecuite and basket through the friction between the loaded basket and the surrounding air. At the old speed of 1200 rpm. the massecuite cools progressively during purging (increasing the viscosity of the molasses and consequently the difficulty of purging). At 1500 to 1600 rpm. the friction is sufficient to maintain the heat of the massecuite, and at 1700 there is sufficient heat generated to cause a marked rise in temperature.[4] Power determinations for high-speed machines in Hawaii have shown that the increased power per machine is more than offset by increased capacity, so that the power per cubic foot of massecuite purged is less.[5]

292. Separation of Wash and Run-Off in High-Speed Machines. When water is used to wash the sugar in a centrifugal, the separation of the original mother liquor surrounding the crystals (called the run-off or green sirup) from the thinner and higher-test wash is obviously advantageous. The mechanical handling of the problem in the old-type centrifugals by means of hand-operated tilting buckets which diverted the material from one trough to another was never very satisfactory as the lag in the heavy sirup in the curbing resulted in a large amount of mixing. The new high-speed machines have the separating device as a part of the curbing itself (see Fig. 80) automatically controlled and timed. At the proper time the control also actuates a hot water spray within the curbing. This washes the heavy run-off inside the curbing into the proper gutter just before the wash water is applied and the separation device is shifted so as to divert the wash to the other gutter. This automatic procedure insures an efficient separation which results in a sharper reduction in purity of the run-off and in much less in-boiling. This separation is of greatest importance in refining, both at the affination station and for white sugar massecuites, and it has been employed with success for the purging of third massecuites[6] instead of double purging as described above.

293. Hot Mingling. A process for use in conjunction with the high-speed centrifugals described above is a new treatment for massecuites known as hot mingling, originally advocated by Stevens. The heat-treating process is used for raw sugar massecuites, raw sugar thirds, refinery affination magma, and refinery white massecuites. In general, the process maintains a small amount of massecuite at the temperature where it will purge most readily (see Sec.

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[4] C. A. Olcott, *Intern. Sugar J.*, March, 1939, p. 104; *11th Annual Proc. Cane Sugar Tech. Cuba* (1937), pp. 107–111.

[5] Ralph B. Johnson, *Intern. Sugar J.*, June, 1936, p. 220

[6] Stevens, *Facts About Sugar*, May, 1939.

278 and Fig. 76). A similar arrangement of the Stevens system for affination magmas in a refinery is described by Gutleben[7] (Fig. 94, p. 293). The system, wherever applied, allows boiling of heavier massecuites to obtain maximum yields, decreases wash water requirements, increases centrifugal capacity, and maintains uniform massecuite conditions throughout the entire purging of the strike. In the refinery the percentage of raw sugar washings has been shown by Gutleben to be cut about in half by the combined hot mingling and high-speed purging.

294. Molasses Dilution and Heating. The molasses or sirup as it is purged from the centrifugals in the raw house or the refinery must be diluted and heated for reuse in the pans as described in Chapter 11. To eliminate irregularity of molasses dilution as well as the lack of uniformity in heating, the arrangement shown in Fig. 81 has been designed.[8] Irregularity in dilution and heating by hand regulation may result in poor pan performance. A fairly good-sized tank, either rectangular or circular, low enough for gravity flow from the centrifugals, must be provided with a motor-driven circulator, *A*, in a tunnel, *B*. The incoming molasses is delivered into the tank at the tunnel

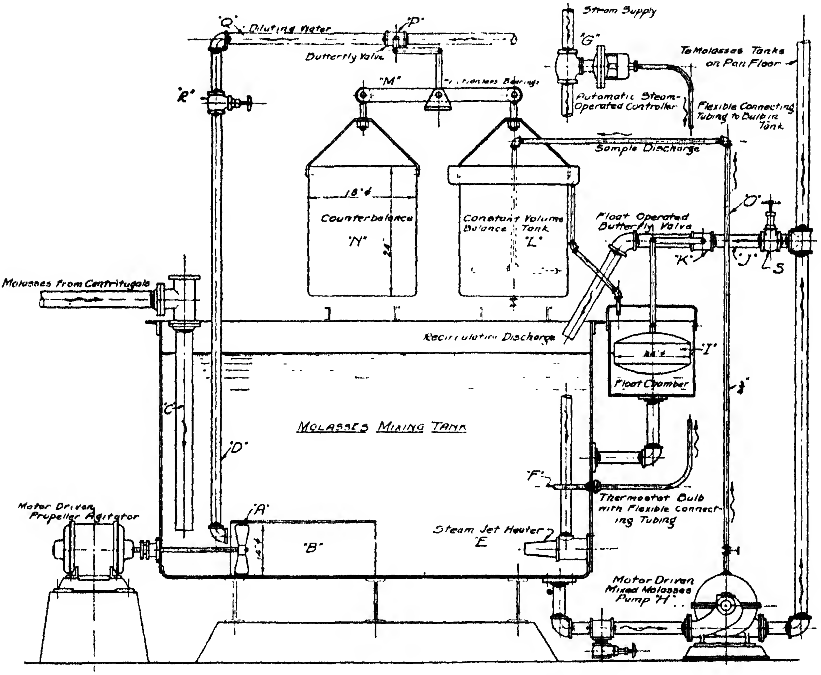

FIG. 81. Automatic Molasses Dilution.

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[7] *Chem. Met. Eng.*, **44** (January, 1937).

[8] A. L. Webre, *Cuban Sugar Tech. Assoc.*, December, **1942**.

inlet through a pipe, *C*. The diluting water also is delivered to the same point through another pipe, *D*, insuring a thorough mixture.

Opposite the discharge of the tunnel is a silent steam heater, *E*. This is controlled by a thermostat, *F*, and diaphragm valve, *G*, so that the temperature of the dilute molasses is fixed at the required point. The pump, *H*, removes the molasses from the tank and sends it to the pan floor, properly diluted and heated. The level of the molasses in this tank is kept constant by means of a float, *I*, and by-pass, *J*, with butterfly valve, *K*, so arranged that when the level is too low, the discharge of the pump is returned to the tank.

The density of the dilute molasses is automatically controlled by means of a small constant-volume tank, *L*, suspended on a balance arm, *M*, with an adjustable counterweight, *N*. A continuous supply of dilute molasses is pumped into the constant volume tank through pipe *O*, and overflows into the float tank, *I*, and back into the main tank. The counterbalance *N* is adjusted so that it has the same weight as the constant-volume tank when the density is at the desired point, say 70° Brix. If the density is greater, the volume tank goes down and by a system of linkage, opens butterfly valve *P* on pipe *Q*, which admits water into the system and thus dilutes the molasses. As soon as the Brix of this molasses is less than the set point of control, the constant-volume tank becomes lighter than the counterbalance and therefore rises and shuts off the diluting water. In practice, it will be found necessary to operate with valve *R* throttled to admit just a little more water than the maximum requirements; also the dilute molasses supply pipe *O* must be large enough to turn over the contents of the constant-volume tank *L* about once in three minutes.

As fast as molasses comes from the centrifugals, it will be diluted, heated, thoroughly mixed, and pumped to the pan tanks upstairs. When molasses ceases to come, the by-pass at *J* and *K* opens and so remains until more molasses arrives from the centrifugals, raising the level in the dilution tank and thus putting the system in operation again.

PACKING

295. Packages. Raw sugars are usually packed in jute bags for shipment, without further drying than they receive in the centrifugals. Their moisture may be reduced by the use of superheated steam in the centrifugal. Raw sugars may also be dried in granulators or driers such as are used in the refineries (see p. 338), provided that they are of high-test and clean crystal. This method of drying is very likely to result in a product that will harden or cake in the packages.

The usual Cuban sugar bag holds from 325 to 330 lb. of centrifugal sugar, measures 29 by 48 or 30 by 50 in., and its tare is from 2.5 to 2.7 lb. Puerto Rico packs in bags holding either 250 or 310 lb., and the Hawaiian Islands use bags of 125 to 140 lb. capacity.

296. Drying the Sugar. Raw sugars are not usually dried. White sugar may be dried in the granulator or drier or with superheated steam in the centrifugal. The crystals lose a part of their gloss in the granulator through friction with one another. The gloss is preserved in drying with superheated steam, and it is such sugars that are made in Java for the home and East Indies markets. The steam should be heated to about 200° C. in a separate boiler.

297. Conveyors. Various types of conveyors are used in transferring sugar from the centrifugals to the packing bins. Sugar should not fall directly from the centrifugal into the package, since under this condition it cannot be of uniform quality, and since it is moist and warm, it will tend to harden. Three types of conveyors are in general use. (1) The ribbon or screw conveyor is a screw or spiral ribbon that revolves in a trough and carries the sugar with it. The cross section of the trough should be a parabola. (2) The endless-belt conveyor, rubber-covered or of fabric, is rapidly increasing in use in both factories and refineries. The belts take much less power and are easier to clean than scroll conveyors. Steel-slat and endless-steel-belt conveyors found favor some years ago but the tendency to rust and abrade makes them less desirable than the rubber-covered belt. (3) The grasshopper conveyor is an efficient conveyor that is used very generally in factories of Scotch design. As is implied by the name, the motion of the conveyor is something like that of a grasshopper. A trough is arranged to move slowly forward, carrying the sugar, and then pull backward very quickly, leaving the sugar. Each stroke advances the sugar a certain distance. All parts of the conveyor are easily accessible for cleaning.

298. Weighing Sugar. The packing bins are generally large enough to permit some cooling of the sugar by keeping it in storage a short time. It is also customary to have the sugar strike on a fan or spreading device as it enters the packing bin. This action throws the sugar out into the air and causes some cooling and evaporation.

In most modern plants the sugar is discharged into the bags from automatic scales connected with these storage bins. These scales have been perfected so that with careful supervision they deliver sufficiently exact weights for control purposes.

299. Caking of Sugars. Caking or hardening of both raw and refined sugars is a frequent difficulty, particularly when sugar has been exposed to a sharp drop in temperature. Investigators have concluded that hardening is not an inherent feature of the particular sugar but that any raw sugar will harden if exposed to certain conditions. Dekker[9] found that the ratio of invert sugar to moisture was a factor in hardening; that where the invert percentage was twice the moisture the sugar hardened much less than when the invert and moisture were 1 : 1. However, with higher invert percentages the

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[9] *Intern. Sugar J.*, July, 1941; p. 224 (abs.).

tendency to harden was greater than with lower invert percentages, if the ratio of invert to moisture remained the same. The generally accepted theory of the cause of caking is that there is a supersaturated film of sirup around the crystals at a given temperature, and with a fall in temperature, or a reduction in the moisture content of the film, crystallization starts up, cementing the original crystals of sugar together with these newly formed, fine crystals.[10]

In accord with this theory (and also with Cuban experience), are some conclusions from a study of the immediate causes of caking of raw sugars by Wuthrich:[11]

1. *Sugar of High Moisture Content.* Sugar with a high moisture content, say 1 per cent and more, was found invariably to cake when brought into surroundings where part of its moisture evaporated or dried out. On the other hand, it was found that caking did not occur as long as the moisture content remained high.

2. *Sugar of Low Moisture Content* (0.3 to 0.4 Per Cent). Normal sugar with low moisture content will not cake, whether cold or hot bagged, as long as it is not exposed to a very humid atmosphere and later on to a dry atmosphere.

3. *Quality of the Sugars.* The foregoing remarks refer to normal sugars. For if the sugar is abnormally viscous and sticky, it will cake no matter whether it has a low or high moisture content; and no matter whether it is bagged cold or hot.

Further, small-grain sugar is much more susceptible to caking than large-grain sugar. Especially sugar with high moisture content cakes very soon when the grain is small. The smaller the grain, the larger the surface of a given quantity of sugar, and the greater the chance of its being cemented together.

4. *Temperature at Bagging.* Hot bagging of sugar of high moisture content will increase the chance of caking considerably, since it helps the water to evaporate. Hot bagging of sugar of low water content does not influence the caking of normal sugar, but it does do so when the sugar is abnormally viscous and sticky.

5. *Weather and Climatic Conditions.* From the above the conclusion may be drawn that weather conditions and change in these conditions, in both the country of origin and destination, and also a difference in the climatic condition of these countries, form a great factor in the formation of caked sugar.

6. *Storage Conditions.* If the air in a warehouse becomes too hot or too moist, this will increase the chance of caking.

It is seen that raw sugars which are of good even grain, well dried, and not excessively gummy (good clarification) are not likely to cake. It has been shown elsewhere (Chapter 15) that these are the characteristics of a good keeping sugar (one that will not deteriorate in test). Packing raws cool and storing them in well-constructed, dry warehouses are also conducive to the prevention of caking. Nevertheless, practically all raw sugar will harden when exposed to very cold weather, softening again when the weather moderates.

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[10] H. S. Walker, *Intern. Sugar J.*, **25**, 131 (1923).

[11] *Intern. Sugar J.*, **25**, 192 (1923).

The investigations of Holven,[12] Webster,[13] Behne,[14] and others on the hardening of sugar during storage have all supported these findings. The caking of well-made refined sugars is largely a matter of storage conditions. Sugar of irregular grain, particularly one containing an excess of dust, will tend to cake more, and a sugar coming from the granulators with more than a trace (0.03–0.05 per cent) of moisture will also cake in storage, but high-grade refined sugars are not generally subject to these criticisms. The usual cause of the difficulty is through the absorption of moisture from the surrounding atmosphere with a subsequent drying out or drop in temperature, setting up the crystallization of fine grain in the moisture film. Heated warehouses, described below, have entirely eliminated this difficulty.

Wuthrich [15] gives an illustration of the effect of moisture absorption on high-test dry Java white sugars. Sugar from the same crystallizer was transported for fourteen days in a hot humid atmosphere, the part in open trucks caked badly, whereas that in closed trucks did not cake at all.

300. Classification of Raw Sugars. The basis of the raw sugar market of the western hemisphere and for a large part of the rest of the world is a centrifugal sugar polarizing 96°. By far the greater proportion of the raw sugars produced are now of this class (or better) and the older molasses sugars and seconds of 89° have practically disappeared from the market. "Muscovado" sugars were made by open-air evaporation of the sirup and removal of the molasses by drainage, but it would be difficult to obtain a sample of such sugar today.

The Dutch color standard formerly used for classifying raw sugars has been abandoned in all but a few countries. The Dutch Standard is a series of sugars ranging in color from a very dark brown, numbered 7, to an almost pure white sugar numbered 25. These samples formerly were prepared annually in Holland and supplied to the sugar trade in small square bottles of uniform glass and size. Tropical raw sugar used to fall below No. 16 of this standard, but with the higher tests now prevalent this is not always true.

301. Classification of White Sugars. The classification of the sugars produced by the American refineries is given on p. 340. The plantation product is usually called plantation white or plantation granulated. Java white sugars in order to meet the market specifications of the East Indies must grade above No. 25 Dutch Standard.

WAREHOUSING

302. Warehousing Raw Sugars. The maintenance of proper storage conditions is of great importance. It will be shown in the next chapter that deterioration of raw sugars is a function of the amount of moisture in the

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[12] *Repts. Assoc. Hawaiian Sugar Tech.*, 1929.

[13] *Intern. Sugar J.*, February, 1941, p. 46.

[14] *Intern. Sugar J.*, August, 1940, p. 283.

[15] *Loc. cit.*

molasses film on the crystals so it is essential that the absorption of atmospheric moisture be prevented after the sugar has been made. Several studies have shown that the critical relative humidity is about 65 per cent,[16] i.e., if the relative humidity is near that point the sugar will neither absorb moisture nor dry out in storage.

If the warehouse is too dry, that is, if the relative humidity is below 60 per cent, the raw sugar will cake and harden to an unnecessary degree. Therefore, heated warehouses for raw sugar are not customary nor, in fact, desirable.

To prevent the absorption of moisture the warehouses should be dry, large, and practically airtight. It is now generally recognized that the safest practice is to keep warehouses closed at all times, particularly in the tropics and in moist climates, in order that as little moist air as possible may come in contact with the sugar.

Webster[17] in an extended study of storage of raws at different temperatures and different humidities summarizes the findings as follows:

> Satisfactory conditions may be achieved in practice by (1) sealed storage conditions, (2) by bagging cool sugar of a low D.I. [that is, safety factor, Sec. 342]. When such a sugar is stored in a closed room, the atmosphere will gradually come to equilibrium with the moisture in the sugar.

Damp-proofing of walls and floors to prevent the seepage of moisture is recommended by many, but this cannot be practiced where galvanized iron buildings are used as in Louisiana.

Concrete walls and floors are advocated for warehouses in the Philippines,[18] but good results are reported from Java by the use of tar (or asphalt) and gravel floors and wooden walls painted with tar. Movable wooden racks are used on the tarred floor. Large stacks piled closely and continuously reduce the surface presented for the absorption of moisture. This point is of the utmost importance and experiments on small stacks of sugar are generally misleading because of the relatively large surface presented to the surrounding air.

Stacking raw sugars on wooden racks which raise the bottom row of sacks 2 or 3 in. off the floor has been found to be excellent practice in Cuba, Louisiana, and elsewhere. Such racks may be made of three pieces 2 in. by 4 in. by 5 ft. (placed flat), across which are nailed 1 in. by 2 in. boards 3 ft. long, spaced 2 in. apart. These racks readily support Cuban bags piled thirty high.

Frequently the storage space for raw sugars is a part of the factory building, and under such conditions it has been found that the absorption of moisture (with consequent deterioration) is quite rapid. Condensation and drip from the roof to the sugar may also cause damage under these conditions. Storage in large stacks, already referred to, and protection of the top, sides,

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[16] Behne, *Intern. Sugar J.*, August, 1940, p. 283.

[17] *Intern. Sugar J.*, February, 1941, p. 46.

[18] Shoemaker, *Intern. Sugar J.*, January, 1932, p. 29.

and bottom of the stacks will prove of great value. Tarpaulins may be used but are expensive, and it has been found that sugar in Cuba can be well protected by heavy waterproof paper of the type containing a layer of asphaltum between two sheets of paper. This paper can be reused for several years if carefully handled.

For calculating warehouse capacities it should be remembered that a 325-lb. bag of sugar occupies approximately 6.5 cu. ft. of space and covers a floor-space area of 7 sq. ft. when lying flat.

303. Bulk Storage of Raw Sugars. Bulk storage of raw sugars in large wooden bins has long been common practice for short periods (two or three days) in refineries where dumping or cutting in is practiced in the daytime only. During the past ten or fifteen years bulk storage bins of capacities up to twenty thousand tons have been installed in refineries on the Eastern seaboard to replace warehouses for raw sugar in bags. These bins are massive structures of wood and steel with elaborate systems of belt conveyors for ingoing and outgoing sugar. The sugar is weighed in the bags as it comes from the ship, after which it is dumped from the bags to conveyors and the handling is then entirely mechanical. Raws stored in this way do not harden even in freezing weather. A surface crust forms but the mass of the sugar remains free.

A concrete storage bin somewhat similar to those used for bulk storage of refined (see Sec. 305) is described with full engineering details by Ballou.[19] No heating system is required because of the sticky nature of most raw sugars, but moisture control is practiced when dry Fiji sugars are stored.

304. Warehousing Refined Sugar. The storage of refined sugar has many similarities to the storage of raw sugar but it differs in one essential since there is no problem presented if the warehouse is very dry, e.g., by artificial heating. Because of the nature of the product, refined sugar warehouses are generally better built than those used for raw sugar; masonry walls and tight wood or concrete floors are essential.

Heated or Air-Conditioned Warehouses. In the past decade it has been found advantageous to have warehouses for refined sugar heated by means of circulating warm air, and in one or two instances elaborately air-conditioned systems have been installed. An electrically heated warehouse in Chicago 120 ft. by 300 ft. with hygrostatic control at 65 per cent relative humidity contains four electrical heaters and large ventilating fans[20] near the ceiling, which consume about 2000 kwhr. Other warehouses with full automatic control of temperature and humidity conditions have been installed, one very large one being at Cairo, Illinois.

The experience of the writer with heated warehouses has shown that close control of the conditions of humidity is not essential. All that is necessary is (1) to keep the warehouse somewhat warmer (3°–5°) than the outdoor air

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[19] *Sugar,* August, 1941, p. 27.

[20] Waby, *Intern. Sugar J.,* July, 1934, p. 285 (abs.).

when temperatures outdoors are moderate, (2) to prevent the warehouse temperature from falling below 60° F. when the weather is cold, and (3) to see that the circulation of air is ample around all stacks.

A heating system, installed at Gramercy in 1929 and successfully operated under these simple regulations is in a warehouse 400 ft. long and 100 ft. wide with a height of 17 ft. to the roof trusses and 23 ft. to the roof in the center.[21] The warehouse is divided into two 200-ft. lengths by a fire wall, and each section of the warehouse is served by a warm-air trunk line along each of the two long walls. Two Weir steel furnaces, burning natural gas, are arranged in a single brick setting as shown in Fig. 82. The warm-air trunk lines have 12-in. outlets provided with dampers at intervals of 15 ft., and the outlets are placed so as to direct a stream of warm air against the brick wall to prevent the possibility of "sweating." The warmed air recirculates without return ducts to the fan serving the furnaces.

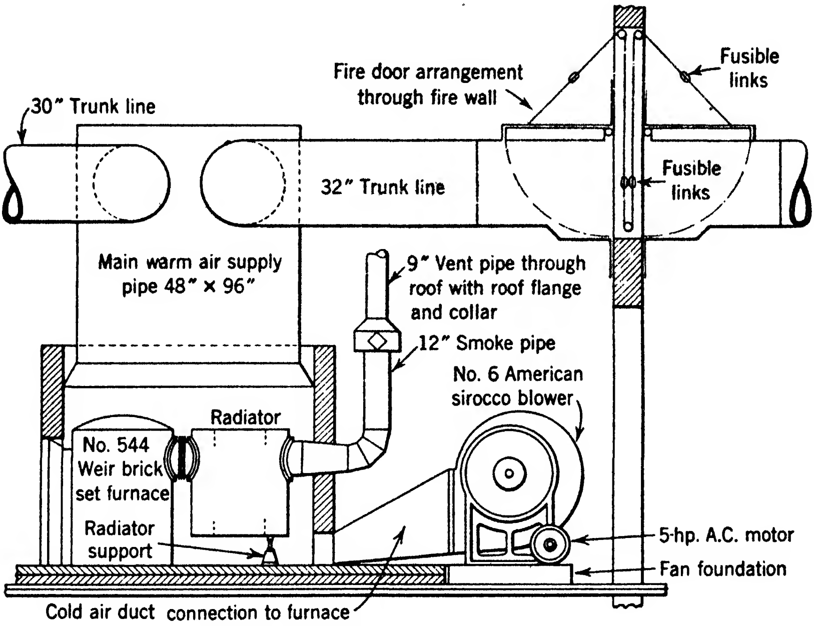

FIG. 82. Warehouse Heating System.

Ventilation on clear dry days is beneficial. During the summer months in Louisiana the heating system is used only at night when fogs are prevalent and on rainy days. Under these conditions relative humidity is always below 60 per cent, that is, the sugar is in a drying atmosphere, and granulated sugar has been stored for long periods without developing even a "warehouse set."

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[21] See *Facts About Sugar,* August, 1932.

305. Bulk Storage of Refined Sugar. Storage of granulated sugar in bulk in concrete silos was first instituted in the beet industry in 1930,[22] but their use has been extended to at least one cane sugar refinery. The largest installation described has a capacity of over 35,000,000 lb. of sugar and consists of five cylindrical concrete bins 40 ft. in diameter and 105 ft. high. The walls are of hollow tile through which heated air can be circulated to regulate the temperature. In another installation warm air is blown directly into the bins as the sugar enters, or whenever humidity conditions require it. All sugar handling is mechanical, and the sugar is packed in the desired packages at the time it is ordered out for shipment.

306. Storage of Soft Sugars. The soft brown and yellow refined sugars which contain 2 to 5 per cent moisture (see p. 342) cannot be stored in such a heated warehouse but must be kept in a cool and relatively moist atmosphere so that they will not lose moisture and become hard. Sugars of this type become hard and stiff in the packages when chilled because of the increased viscosity of the sirup on the soft crystals. They will recondition themselves when returned to normal temperatures in a moist place. Hardening may also occur because of drying out in a heated atmosphere, and this hardening of soft sugars is more difficult to correct.

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[22] *Facts About Sugar,* February, 1934.

CHAPTER 14

BLACKSTRAP MOLASSES AND EDIBLE SIRUPS

307. Definitions. *Blackstrap.* Blackstrap molasses is the by-product (or end product) of either raw sugar manufacture or refining. It is the heavy, viscous liquid separated from the final low-grade massecuite from which no further sugar can be crystallized by the usual methods. It is generally described as inedible because it is not used for human consumption, but it may be eaten without harmful results.

The Association of American Feed Control Officials[1] defines cane molasses for feeding as "a by-product of the manufacture of cane sugar from cane and shall contain 48 percent or more total sugars expressed as invert sugar. Its solution in an equal weight of water shall test not less than 39.75 degrees Brix." (*Note.* The figure for total sugars is generally expressed as the sum of the sucrose [Clerget] and the glucose [or reducing sugars]. Expressed as invert sugar the total sugars would be [Sucrose ÷ 0.95] + reducing sugars. The latter is obviously the correct method since sucrose and invert sugar should not be added unless calculated to equivalents.)

Molasses and Sirup. The distinction between molasses and sirup is not exact in the trade, but technically the terms are accurately defined. The International Society of Sugar Cane Technologists Committee on Uniform Terminology defines sirup as the "concentrated juice from the evaporators," and molasses as "the mother liquor separated from the crystals by mechanical means. It is termed 'first,' 'second,' etc., to 'final,' according to the massecuite from which it is obtained." Blackstrap is therefore final molasses under this definition and has always been designated by this term in raw sugar factories.

In the refineries the term molasses is not used at all, and sirup means the same as molasses does in the raw factory. The blackstrap of the refinery is called barrel sirup by the refiners themselves.

Edible Sirup. Edible sirups and edible molasses are generally classed as sirups commercially, although the definitions given above for cane house work show the difference between the two products. A high-grade cane sirup is one boiled directly from cane juice with no removal of sugar by crystallization. First and second molasses are also made for direct consumption or for blending with other sirups, and are known as "boil-back" molasses in Louisiana and in the wholesale trade. "Refiners' sirups" of various grades are made from

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[1] *Official Publication,* 1940, p. 27.

selected sirups (according to the refiners' definition given above) generally filtered and blended with inverted sirup, if necessary, to prevent crystallization.

308. Molasses as Fuel. In the western hemisphere molasses is rarely if ever used as fuel, but in Australia and Java its use for this purpose is quite common. The fuel value pound for pound is about equal to that of green bagasse (see Sec. 79), so it follows that fuel must be very high priced and molasses quite cheap to warrant burning the molasses.

309. Recovery of Sucrose from Blackstrap Molasses. By the definition already given the sucrose in blackstrap is non-crystallizable by ordinary means because of the presence of reducing sugars and non-sugar impurities (see p. 223). There have been several processes suggested for the recovery of sucrose from molasses, which are all based on the destruction or removal of the reducing sugars. After the reducing sugars have been removed, the sucrose may then be recovered by means similar to those used in the beet sugar industry (the Steffens process). Commercial recovery has never been carried out on any appreciable scale. Two of the methods of sucrose recovery which seem most promising are described below.

Olivarius Process. The reducing sugars in this process are removed by biochemical means. The steps consist of (1) removal of reducing sugars by special fermentation with yeast, (2) elimination of impurities by precipitation with alcohol and lime, (3) recovery of the alcohol for reuse by distillation, and (4) recovery of the sugar by boiling or precipitation as saccharate with lime, barium, or strontium.[2] The process has not been tried on a full commercial scale, although small-scale trials have been made in Hawaii and the Philippines.[3]

Barium Process. This process is a development of DeGuide's patents which have been successfully applied to the beet sugar industry. Patents covering the cane sugar industry were obtained by Spencer in 1923 and later by Meade and Traxler[4] and A. L. Holven.[5] The process consists of adding sufficient barium hydroxide to diluted molasses to destroy the reducing sugars present and at the same time to precipitate the sucrose as barium saccharate. The glucose decomposition products remain in solution, and the saccharate is filtered out, washed with dilute barium hydroxide, carbonated to precipitate the barium and release the sucrose, and then the sucrose may be recovered by boiling. The final traces of barium are completely removed from the sugar solution before boiling by adding a small amount of sodium sulfate. The waste water from the carbonation process must be sulfited for complete recovery of the barium, as CO_2 will not precipitate all the barium. The barium

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[2] U.S. patent, 1,730,473, *Intern. Sugar J.*, April, 1930, p. 218.

[3] L. E. Phillips, *Proc. Hawaiian Sugar Planters Assoc.*, 1932.

[4] U.S. patents 1,884,798–1,884,901.

[5] *Intern. Sugar J.*, April, 1934, p. 164.

precipitates are then mixed with silica (in the form of kieselguhr, which may be used as an aid to filtration) and burned in a rotary kiln similar to a cement kiln to yield tribarium silicate, which breaks up in hot water into monobarium silicate and barium hydroxide. The hydroxide is used to treat more molasses and the monobarium silicate is mixed with the next batch of precipitated barium sulfite and put through the kilns to yield tribarium silicate. The barium recovery process has not been developed to be commercially satisfactory for cane molasses.

COMPOSITION OF BLACKSTRAP MOLASSES

310. Variable Composition of Molasses. Since final molasses contains the greater part of the concentrated non-sugars of the cane juice, together with an unextractable portion of the sucrose and reducing sugars, it follows that the composition, qualitatively, must in general be the same as that of the juice from which the molasses is derived. It also follows that the molasses must quantitatively be quite variable in composition since it has been shown that the composition of cane juice varies widely (Sec. 24) because of such factors as the variety of cane, the extent of milling, climatic and soil conditions, and the treatment of the juice. The juice treatment and subsequent methods of manufacture may have an even greater influence in the composition of the final molasses than the character of the cane from which it is derived. Certain changes take place in the composition during the manufacturing process so that qualitatively and quantitatively there are differences in the character of the non-sugars in the molasses as compared with those of the corresponding juice. The principal changes are those occasioned by the action of lime, or other alkalies, upon reducing sugars, particularly levulose, at high temperatures.

311. Typical Analysis. A typical analysis of cane molasses is hard to formulate, but certain general figures are of interest. The density of blackstrap as purged from the centrifugal ranges from 85° to 92° Brix, containing total solids by drying 77 to 84 per cent (approximately 45° to 47° Baumé). The sucrose varies between 25 and 40 per cent, and the reducing sugars from 30 to 12 per cent with total sugars (the sum of sucrose and reducing sugars) about 50 per cent as a rule. In Louisiana because of less mature cane the reducing sugars are generally higher and the sucrose lower than in Cuba and other tropical countries. King and Rotor gave the sucrose content of twenty-one samples of Philippine molasses as 30.48 to 37.17, and reducing sugars 33.35 to 19.59 per cent which high values they ascribed to immature cane.[6]

The table on p. 248, condensed from many analyses made by Browne about 1906, shows the approximate composition of Louisiana blackstrap. Present-day molasses from variety canes would show a higher nitrogen content.

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[6] *Sugar News*, 1932, No. 3.

	Per Cent		Per Cent
Water	20.00		20.00
Ash	8.00	Silica, SiO_2	0.50
		Potash, K_2O	3.50
		Lime, CaO	1.50
		Magnesia, MgO	0.10
		Phosphoric acid, P_2O_5	0.20
		Sulfuric acid, SO_3	1.60
		Chlorine, Cl	0.40
		Soda, iron, etc., Na_2O, Fe_2O_3, etc.	0.20
Sugars	62.00	Sucrose	32.00
		Dextrose	14.00
		Levulose	16.00
Nitrogenous bodies (Total N = 0.5 per cent)	3.00	Albuminoids	0.30
		Amids (as asparagin)	0.30
		Amido acids (as aspartic)	1.70
		Nitric acid	0.15
		Ammonia	0.02
		Xanthin bodies	0.30
		Other nitrogenous bodies	0.23
Soluble gums	2.00	(Xylan, araban, pectin, etc.)	2.00
Free acids	2.00	Melassinic, glutinic, saccharinic acids, etc.*	5.00
Combined acids	3.00		
Total	100.00		100.00

* Recent work has shown that aconitic acid is the most important acid in molasses (see p. 24).

Molasses also contains a small quantity of caramelization product, the amount of these depending upon the temperature of evaporation and boiling.[7] Caramel is always formed by overheating cane sugar and is a mixture of several dark-colored bodies of uncertain composition.

312. Ash. The amount and composition of the mineral salts or ash in molasses will vary with the character and amount of ash in the original juice. It has been shown by Fort and McHaig (see p. 21) that the ash in Louisiana cane juices varies widely owing to differences in varieties of cane, soil conditions, and other factors. It follows that the difference between the ash in molasses from different countries will therefore be quantitatively quite great although the qualitative differences will not be important. The percentage of ash runs from 7 to 11 per cent in most blackstrap, but Hawaiian molasses sometimes contains 15 per cent or more.

Analyses of the ash of molasses from different countries are given on p. 249. It must be borne in mind that these are not necessarily typical and the pro-

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[7] Caramelan, Caramelen, Caramelin, see von Lippmann, *Chemie der Zuckerarten,* third edition, p. 1210.

portions of the various mineral constituents in molasses now being produced may be quite different from those shown because of the changes in cane varieties in recent years.

	LOUISIANA (Browne, 1906)	HAWAII (1930)	ARGENTINA (Cross, 1906)	JAVA (Geerligs, 1905)
Percentage of Ash in Molasses	8.00	12.60	9.68	9.23
SiO_2 (% Ash)	6.25	3.50	4.13	1.63
K_2O	43.75	37.50	39.98	43.44
CaO	18.75	13.80	8.47	9.21
MgO	1.25	9.70	1.96	1.84
P_2O_5	2.50	2.00	1.03	1.80
SO_3	20.00	12.40	10.74	15.60
Cl	5.00	15.90	8.06	6.93
Na_2O, Al_2O_3, Fe_2O_3	2.50	1.70	6.30	2.21
CO_2 and undetermined	....	3.50	19.33	17.34
	100.00	100.00	100.00	100.00

Figures given by Behne[8] show the variations that may occur in the ash of molasses from the same country. Analyses of the molasses from four mills in Queensland, Australia, gave extremes as follows:

SiO_2	Minimum	1.86%	Maximum	6.60%
K_2O	"	37.48	"	41.78
CaO	"	10.27	"	16.58
MgO	"	5.45	"	11.37
P_2O_5	"	1.53	"	8.50
SO_3	"	3.69	"	9.59

It will be noted that in general these analyses are high in potash, with lime and sulfates the next largest constituents, but King and Rotor[9] found in Philippine molasses that "sodium and silica predominate, potash rather low, lime about double the potash and sulfates low," which is certainly at variance with the analyses listed above from Java, Louisiana, Hawaii, and the Argentine. This illustrates how difficult it is to formulate a typical analysis of the ash of molasses.

313. Organic Non-Sugars. The organic non-sugars of blackstrap will show considerable variation from those of the juice because of changes in composition during the manufacturing process. As will be shown later there are also changes in composition during storage. Geerligs[10] gives the following as typical analyses of the organic non-sugars of Java blackstrap:

[8] *Proc. Queensland Soc. Sugar Cane Tech.,* 1930.

[9] *Loc. cit.*

[10] *Cane Sugar and Its Manufacture,* second edition, London, 1924, p. 311

	I	II
Gums	2.39%	1.51%
Nitrogenous bodies	1.00	1.00
Organic acid	3.81	4.16
Undetermined balance	5.51	4.49
Total organic non-sugars	12.71	11.16

Geerligs admits that the methods of analysis used to arrive at these figures are not exact, but quotes from extensive work by Hazewinkel and others to prove that the relatively large percentage of undetermined is not due to analytical errors but to the presence of some organic body or bodies. He concludes that the undetermined matter is largely made up of decomposition products of reducing sugars.

The character of the gummy constituents has been studied by Hazewinkel and Steuerwald in Java who found that these constituents in the molasses consist of pectins, gums dissolved from fiber and "slime" in varying proportions.[11] A recent study by Browne and Phillips,[12] using more refined methods, found the methoxyl and uronic acid content in the molasses to be about 0.5 and 2.00 per cent respectively.

The nitrogenous bodies in the analysis of Louisiana molasses shown above total 3.00 per cent, but Geerligs[13] gives 1.00 per cent as the figure for Java molasses whereas the Argentine analyses show close to 4.00 per cent. More recent analyses of molasses from Cuba, Louisiana, and Florida show much higher "crude protein," (total N × 6), figures as high as 9.00 per cent being reported from analyses by large companies using molasses for cattle feed.

The organic acids in molasses were found by Nelson[14] to be formic, 0.1 per cent; acetic, 0.2 per cent; aconitic, 0.8 per cent; and lactic, 0.05 per cent. Traces of malic and citric acids were also found. The recent work of McCalip and Seibert[15] shows that aconitic acid is the most important acid constituent of cane products, including molasses. They found 1.80 and 2.52 per cent of aconitic acid (based on solids) in two samples of Louisiana final molasses.

Browne[16] says glucic acid, although formed in the alkaline clarification of sugar cane juices, is speedily broken down into formic acid and other decomposition products that are found in the by-products of sugar manufacture, such as sirup and molasses. It absorbs atmospheric oxygen so vigorously that considerable heat is evolved. Its presence may, therefore, be an explanation of the so-called hot room or froth fermentation of molasses. (See p. 230.) Such acid bodies of molasses, as melassinic, glucinic, and saccharinic, are not present in the juice, but are formed by the action of lime upon the reducing sugars in the clarification.

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[11] Geerligs, *loc. cit.*

[12] *Intern. Sugar J.*, November, 1939, p. 430.

[13] *Loc. cit.*

[14] *J. Am. Chem. Soc.*, 1929, p. 2808.

[15] *Ind. Eng. Chem.*, May, 1941, p. 637.

[16] *Intern. Sugar J.*, January, 1938, p. 30.

314. Vitamins in Molasses. Vitamins in cane sirups and molasses have been reported by various observers. The references to the occurrence of vitamin B_1 (thiamin chloride) in cane molasses are discussed in the "Story of Vitamin B_1," p. 47, published by Merck & Company, Rahway, N. J., 1940. There appears to be a wide variation in the amount present, as one investigator reports more vitamin B_1 in some cane molasses than in yeast, whereas other samples showed little or none. Vitamin H has also been found in cane molasses.[17]

315. Non-Fermentable Reducing Substances in Molasses. It has long been recognized that there are certain copper-reducing substances in cane molasses which are not fermentable, or difficultly fermentable. Various figures have been given, ranging as high as 10 per cent, and the material has been referred to by early investigators as "glutose," which was considered a single substance, a non-fermentable reducing hexose. More recent studies suggest that the non-fermentable reducing matter is a mixture of various substances, very little of which may be sugars. It has been generally agreed that these substances are the result of the action of lime on reducing sugars during manufacture. Geerligs has given an exhaustive review of the literature of the subject[18] and concludes that it "is a question of great interest for distillers and for those dealing in molasses, but it is naturally difficult to state the percentage of a substance the identity of which is still open to discussion."

Zerban[19] has a discussion of the methods of determination, the conditions under which the reducing substances are found and the nature of the substances. He found amounts of unfermentable reducing substances (calculated as invert sugar) in blackstrap molasses ranging from 3.50 to 4.66 per cent and in high-test invert Cuban molasses from 2.38 to 4.45 per cent, but later work showed over 7 per cent in Cuban blackstrap and 8 per cent in refinery blackstrap.[20] Zerban proved that similar non-fermentable substances can be produced from pure invert sugar solutions by mild acid treatment for several weeks, high-temperature (55° C.) treatment producing a larger quantity than room temperature. He concluded that the substances so produced consist of at least one monosaccharide, probably a keto-hexose, and at least one hexose disaccharide. The subject is still being studied.

316. Changes and Decomposition During Storage. An extended investigation in the changes in molasses during storage has been made by Dr. C. A. Browne, of the U.S. Bureau of Chemistry and Soils.[21] Two samples of Cuban blackstrap have been analyzed from year to year since 1914 and the following changes noted:

1. Loss in polarization (1914 to 1935) from +24.86 to +6.4 in No. 1 molasses, and from +25.52 to +9.60 in the No. 2 molasses.

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[17] *Sugar J.*, March, 1941, p. 17.

[18] *Intern. Sugar J.*, September, 1938, p. 345.

[19] *J. A.O.A.C.*, **23**, No. 3, 562 (1940).

[20] *J. A.O.A.C.*, August, 1941.

[21] *Ind. Eng. Chem.*, **21**, 600 (1929).

2. Loss in sucrose: 31.30 to 12.61 per cent in No. 1; 34.79 to 6.61 per cent in No. 2.

3. Increase in invert sugars: 19.10 to 23.57 per cent in No. 1; 25.09 to 34.13 per cent in No. 2.

4. Loss in total sugars (as invert sugar): 52.04 to 36.84 per cent in No. 1; 61.71 to 41.08 per cent in No. 2.

5. Increase in the percentage of organic non-sugars: Exact figures are not given, but assuming the ash to be constant, the organic non-sugars increased from 16 to 28 per cent and from 10 to 30 per cent respectively.

6. Loss in total solids: about 3 per cent in each sample.

7. Great increase in color.

The decomposition is known to be chemical and not biological because the samples in 1914 and on various subsequent occasions showed no yeasts, molds, bacteria, or other organisms. Dr. Browne is of the opinion that the deterioration is a slow retarded type of the hot-room or froth fermentation (see Sec. 286) which is not the result of the activity of microorganisms, and is therefore not a fermentation at all but a spontaneous chemical change. The formation of volatile acids, largely acetic but some formic, was also shown to occur in these two molasses.

The chief cause of the spontaneous decomposition of the molasses in storage is attributed by Browne to the reaction of unstable organic substances (originally produced by the action of lime upon the reducing sugars of the cane during clarification) with further quantities of reducing sugars in the molasses, which results in the formation of dark-colored colloidal impurities of high carbon content. The reaction between the amino acids and reducing sugars of the cane juice (see p. 230) may also play a minor part in the early stages of the decomposition.

The more rapid decomposition sometimes noted in tanks of molasses is also similar to the "hot-room fermentation." A marked evolution of carbon dioxide gas which appears as froth on the surface and is occluded in gas pockets in the body of the material may occur in molasses, particularly if it has been heated beyond 104° F. (40° C.) and may seriously interfere with the measurement of the molasses in a tank. Molasses which has lain dormant in Cuba, for instance, may develop this evolution of gas when shipped and stored in a tank in the United States. In one instance in the experience of the writer 1,500,000 gal. of 88° Brix Cuban molasses delivered from a steamer to a depth of 20 ft. in a large storage tank increased steadily in depth until the level was close to 24 ft. in twenty days, an increase of 20 per cent in volume due to evolved gases. It then subsided to the original level in thirty-five days and continued to settle below the original gaging until it was down about 8 in., representing 3 per cent reduction from the original volume, which was probably due to air entrained in the molasses while pumping from the steamer to the tank. There was evidence that some of this molasses was heated above 110° F. during discharge from the steamer, but there are other instances where molasses has similarly increased in volume in tanks with no indication of overheating at any time.

Cases have been reported where tanks of molasses have undergone such marked decomposition that the contents have changed to a dark porous mass resembling carbonized sugar. Geerligs[22] notes several cases of massecuites in Java acting in this way and comments on the occurrence of this phenomenon in molasses stored in earthen tanks in Egypt.[23] Geerligs is in full agreement with Browne that the decomposition is the so-called hot-room fermentation and is caused by the spontaneous decomposition of the products formed by the action of lime at high temperatures on reducing sugars.

From the practical viewpoint the spontaneous decomposition is mysterious because the immediate cause does not seem to be apparent. In one instance in recent experience a tank car of molasses shipped from a large storage tank foamed so badly on arrival at destination ten days later that it could not be stored in a tank twice the size, yet thirty other tank cars from the same storage tank showed no foaming whatever, although kept for many days in midsummer heat awaiting shipment and in transit.

Dr. P. Honig[24] said that in storing molasses in Java the loss in fermentable sugars at 30°–35° C. was found to be 2 to 3 per cent per year. A rise of 10° C. will quadruple the decomposition, and therefore no heating of molasses is permitted after it leaves the centrifugals.

PUMPING AND STORAGE

317. Pumping of Blackstrap and Invert Sirups. The handling and pumping of a liquid of such high density and viscosity present problems about which little has been written until recently. A careful study of the pumping of viscous liquids such as molasses and invert sirups has been published by A. M. Shaw, J. G. Real, and V. A. Pardo,[25] and much of the information which follows is quoted from that article. The data relate to Cuban molasses but are undoubtedly of general application.

318. Viscosity. The viscosity of the molasses to be pumped is of the utmost importance, since pipe friction has been found to increase about in direct proportion to viscosity. Viscosity is generally measured in terms of the seconds it takes a fixed volume of the liquid to pass through a small tube of fixed dimensions. There are several different types of instruments, but the commonest is the Saybolt universal viscosimeter. Viscosities are expressed in seconds, and the abbreviation SSU. means seconds Saybolt universal.

Shaw[26] says:

> By measuring the time it takes for the fixed volume to flow through the tube, we are really measuring the resistance to flow of the liquid, liquids hav-

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[22] *Intern. Sugar J.*, January, 1940, p. 25.

[23] See also Habif, *Intern. Sugar J.*, July, 1938, p. 276 (abs.).

[24] Discussion of Dr. Browne's paper.

[25] *13th Annual Proc. Assoc. Cane Sugar Tech. Cuba* (1939).

[26] *Loc. cit.*

ing a higher resistance to flow taking longer to pass through the tube. It is obvious then, that this quality is of extreme importance in pumping molasses through any kind of a pipe line.

It has been found in Shaw's investigation that Cuban molasses (and probably molasses in general) is noticeably increasing in viscosity in recent years. The reason for this increase in viscosity is not known, but he suggested that it may be due to more efficient operation of mills, i.e., greater extraction of non-sugars, although it would appear likely that the new variety canes also have great influence. The organic non-sugars "are difficult to deform, that is, they offer high internal resistance to movement. This resistance lies between the individual particles, and the measure of this resistance is the viscosity."[27]

319. Brix and Viscosity. Shaw makes the statement that Brix (or specific gravity) "bears no relation to viscosity." This statement is misleading unless it is understood to mean that there is no relationship between the Brix of two different molasses and their viscosities. The viscosity of any given molasses, or any sugar liquor, will be reduced sharply by dilution, i.e., by changing the Brix. What is true is that Brix is not a measure of viscosity. As a proof that there is no correlation between the Brix of different molasses samples and their viscosity, Shaw gives figures for a Cuban final molasses of 87.5° Brix with a viscosity of 186,000 SSU., and another final molasses of 89.3° Brix and 50,000 SSU. A final molasses of 90.6° Brix showed 128,000 SSU. as compared with one of 90.8° Brix with a viscosity of 303,000 SSU.; all these viscosities were at 100° F. This point needs to be emphasized, as many people erroneously believe that if the Brix of a molasses is known the viscosity will also be known, approximately at least, whereas the figures show that two molasses of practically the same Brix may differ from each other in viscosity by several hundred per cent.

320. Variations in Viscosity of Different Molasses. Shaw's studies included the determination of viscosities on molasses of different grades. His published curves showed such great variations that it was necessary to plot these on a logarithmic scale. The wide variation between the viscosity of different molasses of the same grade from different factories and of molasses of different grades both from the same factories and from different factories is well illustrated by the following figures taken from these curves for the maximum and minimum viscosities of the different grades at 100° F.

VISCOSITY AT 100° F.

Grade	Maximum	Minimum
A (first) molasses	23,000 SSU.	1,300 SSU.
B (second) molasses	60,000	6,400
C (final) molasses	250,000	16,500

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[27] Shaw, *loc. cit.*

The surprising fact brought out by these results is that the maximum viscosity for first molasses is higher than the minimum viscosity for final or *C* molasses, i.e., the first molasses in one factory is actually more viscous at a given temperature than the final molasses from another factory. Similarly the figures show that a *B*, or second, molasses in one factory may be nearly four times as viscous, i.e., four times as difficult to pump, as the final molasses of the factory having the minimum viscosity.

321. The Effect of Temperature on Viscosity. The effect of temperature on viscosity of molasses is well known but the extent of the effect is much greater than is generally supposed. Again taking the figures from Shaw's curve for Cuban molasses of maximum and minimum viscosities we find the following:

	Viscosity SSU.				
Grade	70° F.	80° F.	90° F.	100° F.	130°F.
Final molasses, maximum viscosity	5,000,000	1,300,000	500,000	250,000	65,000
Final molasses, minimum viscosity	150,000	60,000	28,000	16,500	5,500

It will be seen that the viscosity of final, or *C*, molasses may be ten to twenty times as great at 70° F. as it is at 100° F., and this relationship holds quite closely for the other grades. Shaw's curves also show that the viscosity of a molasses approximately doubles for each 10° drop in temperature from 100° to 70° F.

322. Effect of Viscosity on Pumping. It has been stated previously that pipe friction increases in proportion with viscosity. In order that pumping pressures and pump capacities may be calculated it is therefore necessary to know the viscosity of the molasses at the minimum temperature at which it is to be pumped. Shaw gives a graph, which is too extended for reproduction here, by means of which the friction in a pipeline may be calculated, given the size of the pipeline, the gallons per minute to be pumped, the viscosity, and the specific gravity. As an illustration of the results obtained, a molasses of 60,000 SSU. (at the temperature of pumping, say 100° F.) pumped through 1000 ft. of 10-in. line at the rate of 400 gal. per minute will develop a pressure of 200 lb. per sq. in. As has already been stated, a molasses of twice the viscosity pumped under these conditions would develop a pressure of 400 lb. per sq. in.; or the same molasses at a temperature of about 90° F. (at which point the viscosity would be approximately twice as great) would also result in a pressure of about 400 lb. per sq. in. It is obvious from these illustrations that it is not practical to consider molasses as a definite material so far as pumping is concerned. The viscosity and the conditions which affect viscosity, such as temperature, must be known in order to reach any conclusions as to pump pressures and capacities of pumps.

Another point of importance to be remembered is that the pipe friction in the suction line plus the static suction lift must not approach atmospheric pressure. Wherever possible, the pump should be placed so that there is a static head on the suction pipe.

323. Types ot Pumps. Direct-acting steam pumps having large valves and operating at slow piston speeds are still used to a great extent for the pumping of molasses. Vertical triplex pumps and other types of power pumps have also been employed, but within recent years especially designed rotary pumps of the gear type or rotating plunger type have proved efficient for this purpose. The rotary gear pump (Fig. 83) consists of two herringbone (or helical) gears or rotors within a close-clearance casing. When the liquid comes into the pump, it must fill the space between the teeth of the gears to be carried along and around the curvature of the casing to the discharge, and the speed of rotation of the gears, i.e., of the pump, must depend upon the viscosity of the molasses. For high viscosities the pump must operate slowly to allow the molasses to fill the spaces between the teeth, but for low viscosities the operation may be faster.

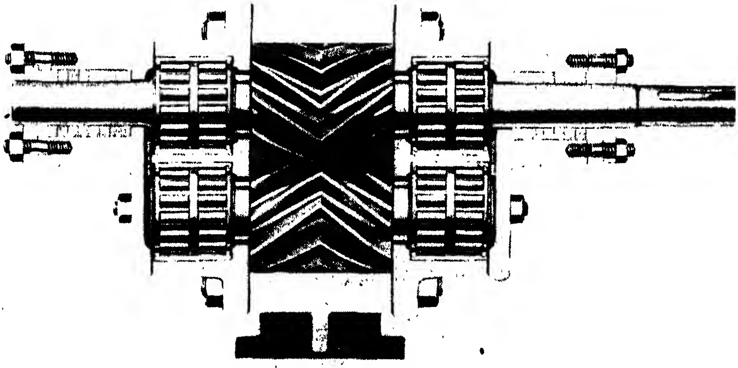

FIG. 83. Worthington Spiral Gear Molasses Pump.

The operation of the rotary plunger pump may be understood from Fig. 84 which shows one side of the pump casing. The two pump chambers are divided vertically by a center wall with suction and discharge openings converging into common pipe connections. The cams in the two sides of the pump are mounted 180° apart on the common driveshaft, each cam operating a plunger having a hollow arm which slides freely in a slide pin. Holes drilled through the plunger carry the liquid being pumped to the cam under pressure. The bearings are lubricated by the liquid being pumped. These rotary pumps of the gear and plunger type cost less than the power-driven or direct-acting steam pumps of similar capacity and occupy much less space. There are no valves or springs in these rotary pumps so that the internal friction is reduced and maintenance costs are small.

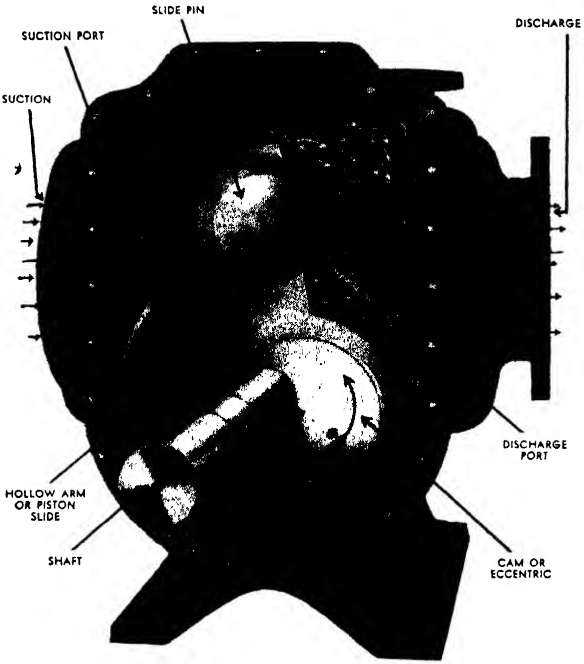

FIG. 84. Kinney Rotary Molasses Pump.

324. Capacity and Horsepower Required. It follows from the previous discussion that the capacity of any molasses pump is dependent on the viscosity, the size and length of pipeline, static head, etc., since all these factors determine the pressure against which the pump will have to work. The graph given in Fig. 85 shows the capacities and horsepower required for a rotary pump at different pump speeds when pumping a molasses of 50,000 SSU. viscosity at various pressures.[28]

The next graph (Fig. 86) shows the increase in horsepower required as the viscosity increases, capacity remaining the same.

325. Storage of Molasses. Tanks for the storage of molasses should have a large margin of structural safety. There is evidence to show that most of the reported "explosions" of molasses tanks have proved to be collapses caused by faulty structure at the time the tank was erected or by weakening of the plates by the corrosive action of the acids in the molasses.[29] The need for frequent inspection of the interior of molasses tanks

[28] Shaw, *loc. cit.*
[29] Browne, *loc. cit.*

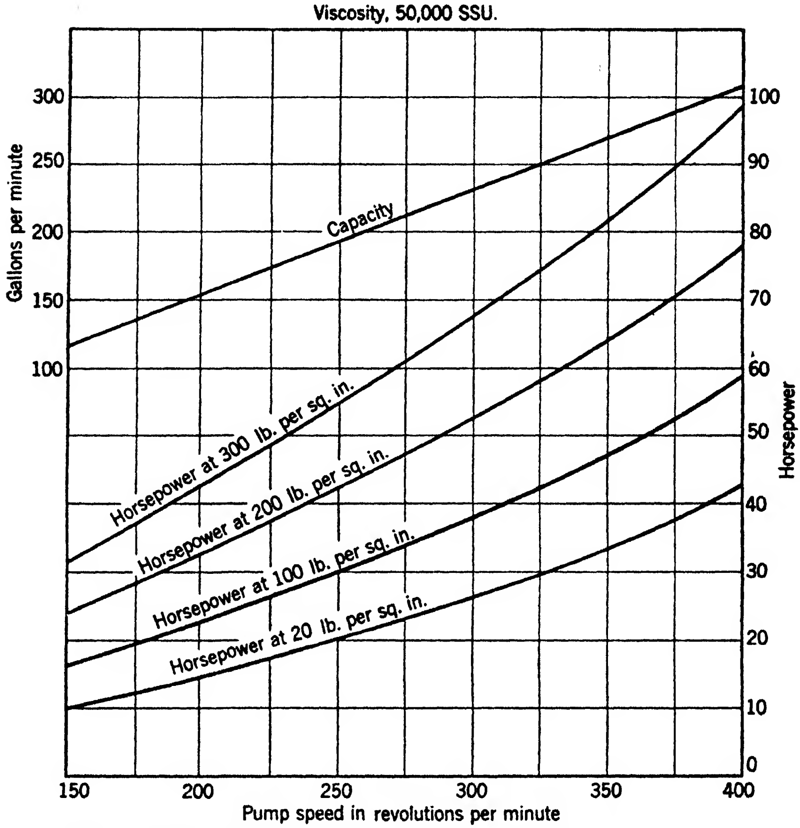

FIG. 85. Relationship between Pump Speed, Capacity, and Horsepower.

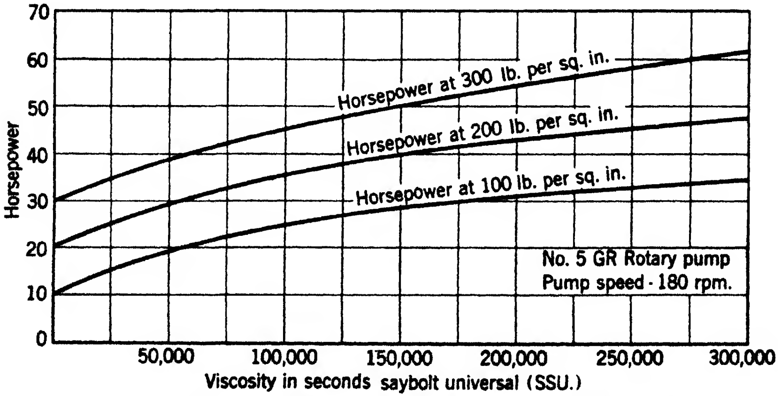

FIG. 86. Relationship between Horsepower and Viscosity.

is evident. Proper grounding to prevent damage by lightning should be provided, and the tank should be properly vented to permit the escape of the gases which may accumulate above the molasses from decomposition.

COMMERCIAL USES OF BLACKSTRAP MOLASSES

326. Alcohol Production. By far the greater portion of the blackstrap produced in the western hemisphere is distilled into beverage alcohol or industrial alcohol. According to the Alcohol Tax Unit of the U.S. Department of Internal Revenue the utilization of molasses by distilleries and industrial alcohol plants accounts for about two-thirds of the total United States consumption of blackstrap, the average gallonage so used for 1935–1939 inclusive being about 200,000,000.

The fermentation of molasses results from the action of yeast which first inverts the sucrose by the action of invertase secreted by the yeast, and then the yeast converts the invert sugar to ethyl alcohol and carbon dioxide according to the following reactions:

$$(1)\quad \underset{\text{(sucrose)}}{C_{12}H_{22}O_{11}} + H_2O + \text{invertase} = \underset{\text{(levulose)}}{C_6H_{12}O_6} + \underset{\text{(dextrose)}}{C_6H_{12}O_6}$$

$$(2)\quad \underset{\text{(invert sugar)}}{C_6H_{12}O_6} = \underset{\text{(ethyl alcohol)}}{2C_2H_5OH} + \underset{\text{(carbon dioxide)}}{2CO_2}$$

The theoretical yield from 1 lb. of invert sugar (0.95 lb. sucrose) is 0.511 lb. of absolute alcohol and 0.489 lb. of carbon dioxide. In actual practice such results are never reached, and the efficiency with crude methods may be as low as 50 per cent. Modern methods with properly selected yeasts will average 90 per cent efficiency or higher.

It is not within the scope of this chapter to discuss the methods used in distilling molasses for the production of beverage or industrial alcohol, but the following yield figures may prove of interest. Owen[30] gives details of the yields to be expected from molasses of 55 per cent total sugars at 12 lb. per gal. (84.0° Brix) at varying fermentation efficiencies from 83 to 95 per cent. The "proof gallons" (50 per cent ethyl alcohol by volume equals 42.4 per cent by weight) range from 0.799 gal. for 83 per cent efficiency to 0.914 gal. for 95 per cent efficiency, the average figure being about 0.85 gal. for 88 per cent efficiency. N. J. King[31] gives a figure of 1 gal. of 95 per cent alcohol for each 15.4 lb. of invert sugar fermented. Magué gives a yield of 6.5 gal. of absolute alcohol for each 100 lb. of fermentable sugar with 90 of the theoretical. The figures all calculate quite closely to the same result, viz., a yield in absolute alcohol of 41 to 42 per cent of the invert sugar input at an efficiency of about 90 per cent.

The by-products of distilling are distillery slops and CO_2. The use, disposal, and economic value of the slops have been discussed at length by

[30] *Facts About Sugar,* July, 1940, p. 38.

[31] *Repts. Queensland Soc. Sugar Cane Tech.,* 1939.

Owen.[32] Solid CO_2, "dry ice," is now regularly produced by many distilling plants.

327. Cattle Feeds. Blackstrap as a constituent of livestock and dairy feed has been used for a great many years. A large part of the molasses which is not used for alcohol production goes into feedstuffs although the amount consumed in this way varies from year to year inversely as the supply of grain. The gallonage ranges from seventy millions to one hundred millions per year in the United States and the amount is evidently on the increase.

The earliest use of molasses for stock feed was probably in the tropics where it is added to the drinking water of the oxen employed in hauling the cane and cultivating the fields. It is being fed directly in this way in considerable quantities to cattle in the United States, but most of it is used to mix with other feedstuffs as a source of carbohydrate and to give palatability to the mixed feed. Molasses feeding increases the consumption of water; the vitamin content, mineral salts, and protein in the molasses are also of value. In commercial ready-mixed feeds it serves as a binder. Many ready-mixed concentrated feeds carry a high percentage of molasses, either partly or wholly dried, and such concentrated feeds serve as a convenient source of molasses for cattle or sheep on the range. On the farm, molasses is generally diluted and sprayed over whatever dry feed may be available.

Formulas for balanced rations vary with the kind of animal to be fed, the work done, the available feedstuffs in the locality, and the price of these feedstuffs. A number of studies on the use of molasses in feeds have been made at various state experiment stations, and favorable results have almost invariably been reported.

The most recent use is a so-called molasses silage which is the general term applied to silage made from any uncured hay crop, which is harvested at the time of highest feeding value (prebloom or early bloom stage), after which it is chopped and sprayed with 60 to 75 lb. of molasses, either diluted or direct, per ton of hay, and stored in the silo in the usual way. The crop is cut while still green regardless of weather conditions, since the moisture (65–75 per cent) is essential. Tests on the feeding value of molasses silage have been made by numerous government agencies with favorable results. In general it has been found to be the equal of good corn silage and more valuable than dry hay.

328. Organic Chemicals. The production of organic chemicals by biochemical methods has been discussed at length by Owen in a series of articles[33] which are now available in book form.[34] The chemicals comprise (1) acetone and butanol in combination as an organic solvent, (2) acetic acid, (3) citric acid, (4) lactic acid, and (5) glycerin.

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[32] *Facts About Sugar,* June, 1938, p. 105.

[33] *Facts About Sugar,* May, 1937, to September, 1938.

[34] *Blackstrap Molasses as Raw Material in the Biochemical Industries,* New York, 1940.

The first of these chemicals (acetone 25 per cent, butanol 70 per cent, ethyl alcohol 5 per cent, in combination) is now produced in considerable quantities from molasses although the greatest source of this solvent in the United States is corn. The principal use is as a commercial solvent for automobile lacquers, but there are many other uses for butyl alcohol derivatives. Among these the most interesting is butadiene, the basis for synthetic rubber, now produced almost entirely from butane gas, the petroleum derivative. The other chemicals named are not of great commercial importance as uses for molasses, but citric acid is produced commercially from refined sugar in considerable quantities.

329. Compressed Yeast. The use of blackstrap in the direct manufacture of a low-grade yeast for cattle food (or human consumption) has advanced farther in Europe than in the United States. The use of molasses as one of the constituents for the manufacture of yeast for the baking industry is on the increase and may be still further developed.[35]

330. Solidified Molasses. This product was formerly made in large quantities in Java for shipment to British India for distillation purposes. Export figures from Java show[36] that the amount decreased from 77,000 tons in 1927 to 11,000 tons in 1936. The manufacture of solidified molasses requires no special apparatus. Molasses is first "blown-up" with steam to about 70° Brix and skimmed, and it is then simply evaporated to dryness in an ordinary vacuum pan operating with a very high vacuum. Serious frothing is liable to occur. The dried molasses, a molten mass, is run into closely woven baskets while it is very hot and solidifies on cooling. The tops of the baskets are covered with burlap for shipment.

The usual commercial specifications for this product are as follows. (1) The solid molasses shall break with a clean fracture. (2) It shall sink in water. (3) It shall be too hard to indent with the finger nail. From what has been shown (Sec. 316) regarding the decomposition of molasses due to heat, it is obvious that excessive decomposition will take place in this process. Geerligs, in the article noted above, reports instances in which vacuum pans have burst due to decomposition in this process.

J. J. Hazewinkel[37] examined solidified molasses to determine whether loss of dry substance occurs during the manufacture. He analyzed the molasses before and after drying and calculated the results to a basis of the ash content of the original material, assuming it to be a constant. His conclusions were as follows. (1) There is considerable loss of dry substance. (2) The extent of the decomposition is dependent upon the temperature. (3) The reducing sugars in part pass over to organic non-sugar. (4) It is possible that to some extent loss of dry substance is due to decomposition of both reducing sugar and organic non-sugar. (5) About equal parts of dextrose

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[35] Owen, *Facts About Sugar*, July, 1937, p. 263.

[36] Geerligs, *Intern. Sugar J.*, January, 1940, p. 27.

[37] *Archief*, 1912, pp. 20, 181.

and levulose are decomposed. In view of the proportions of dextrose and levulose it is probable that polymerization of dextrose to levulose takes place during the long heating. Solidified molasses is used to some extent in feeds in the United States. Patents for its production have been recorded.[38]

EDIBLE SIRUPS

331. Cane Sirup. This is the product made directly by concentrating cane juice as it comes from the cane without removing any sugar. This sirup is generally about 35° Baumé (73°–75° Brix) and is packed in gallon or smaller-sized cans which go direct to the trade. Keller [39] has reviewed at length the processes used in Louisiana and divided them into three general methods.

a. Boil-and-Skim Method. This is the simplest and most primitive process and generally produces a sirup of superior flavor. Cane is crushed in a single three-roller mill as a rule. At most, the milling equipment in larger plants consists of knives, crusher, and two three-roller mills. Heavy crushing is said to affect the product adversely. The strained juice is run to clarifiers and evaporators which in the smaller plants are direct fired and in the larger plants steam heated. Heat alone is the clarifying agent, the albuminoids being coagulated and rising to the surface where the scum is removed by sweeps or scoops made of centrifugal screen. The brushing and skimming continue constantly during the evaporation to sirup

b. Chemical Treatment Plus Boil and Skim. This process is similar to the one above except that the juice is partly neutralized by the addition of lime; then it is brought to a boil and allowed to settle. Brushing and skimming are thus eliminated in the thin juice, but they must be carried on during the evaporation to sirup. The product of this method is darker in color and inferior in flavor to the sirup produced by the first method and does not find as ready a market.

c. Sulfitation Method. This process is employed in the larger plants grinding 200 tons or more of cane per day. For the reasons already stated intensive milling is not practiced, and as a rule the milling plant consists of crusher with six or more rollers on which a little maceration water is used. The strained juice is saturated with sulfur dioxide in the usual way (see p. 109) to an acidity of 3.5 to 4.0 cc. after which this acidity is reduced by lime to 0.8 to 1.0 cc. The juice is then boiled and allowed to settle and the clear juice decanted. Muds are resettled and finally filter-pressed (or in the smaller plants discarded after several settlings). The clear juice is partly evaporated to about 50° Brix in either multiple-effect or open evaporators and again allowed to settle, after which the clear semi-sirup is further evaporated in open evaporators to 70°–72° Brix. The fancy grades are stored in large tanks

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[38] *Intern. Sugar J.*, April, 1938, p. 159.

[39] *Year Book of Louisiana Sugar Cane Industry*, 1939, pp. 190–195.

for several weeks before canning. A large part of this sulfited sirup is sold in bulk for blending with molasses or glucose for table use and cooking.

332. Edible Molasses. Many Louisiana sugar houses which make cane sirup on a large scale by the sulfitation process produce a direct consumption sugar by sulfitation from part of their cane crop and sell the molasses from this sugar. This molasses is about 80° Brix, 45°–50° apparent purity, clear, and light brown in color. It is known to the trade as boil-back molasses, and generally it is sold in bulk for blending.

The old-fashioned New Orleans molasses which is no longer on the market was made by the open-kettle process. The clarified juice was boiled to grain in open kettles, allowed to stand in cooling tanks, and then purged in centrifugals. The molasses was quite dark but had a characteristic flavor which was highly prized for many purposes.

INVERTED SIRUPS AND MOLASSES

333. Process. The production of invert molasses or high-test molasses from surplus cane has attained considerable magnitude both in Cuba and Puerto Rico. In Cuba alone as high as three hundred million gallons have been produced annually in recent years. Strictly speaking, the product is an inverted sirup and not a molasses as the concentrated cane juice from the evaporators is the basis of the product. The milling, clarification, and evaporation are the same as for sugar manufacture except that a minimum of lime is used (in some cases none at all) to give a sirup (meladura) of about 6.0–6.3 pH. The inversion is effected in either of two ways, by acid or by invertase (yeast), the latter method now being the more general. Inversion by sulfuric acid is carried out at a temperature of 195°–200° F., the sulfuric acid being in the proportion of 1–2½ gal. per 100 gal. of sirup. Heating is continued until a rapid analysis shows a polarization from 0° to 10°, after which milk of lime is added until a pH of 6.0 is obtained. It is then evaporated to about 85° Brix, the result being a deep red, viscous molasses containing about 78 per cent total sugars of which about two-thirds is invert sugar and one-third sucrose.[40, 41] Continuous processes of acid inversion have been reported upon.[42]

In the newer Guerrero process the invertase is obtained from special strains of yeast which are very rich in invertase. The yeast is grown in last mill juice and concentrated to a cream in yeast separators. The usual proportion is four volumes of 12 per cent cream to 100 volumes of 53° Brix sirup, and 65 per cent of the sucrose originally present is inverted in ten hours at 60° C.[43] The crystallizers are commonly used as inverters.

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[40] Lopez Ferrer, *9th Annual Proc. Assoc. Cane Sugar Tech. Cuba* (1935), p. 234.

[41] *Committee Report, 12th Annual Proc. Assoc. Cane Sugar Tech. Cuba* (1938), p. 232.

[42] Mascaró, *12th Annual Proc. Assoc. Cane Sugar Tech. Cuba* (1938), p. 199.

[43] *12th Annual Proc. Assoc. Cane Sugar Tech. Cuba* (1938), p. 232.

Much more usual practice is to invert fully all the sucrose in about 60 per cent of the sirup and then to draw this inverted sirup and a portion of uninverted sirup into the pans for concentration. The publications on the use of yeast invertase in high-test production have been numerous and the reader is referred to recent issues of the Annual Proceedings of the Association of Cane Sugar Technologists of Cuba.[44, 45, 46, 47] Other references are given in Sec. 667 on the analysis of high test and in Sec. 829 on the chemical control of the process.

The advantages of the invertase (yeast) method are that it eliminates the loss of sugars usual in the acid inversion (about 4 per cent) and gives no increased ash as does the neutralization of the sulfuric acid with lime. The lower temperatures (140°–150° F.) are also advantageous in preventing destruction of levulose.

334. Uses and Composition. The high-test molasses made by these methods is used largely for distilling, the higher sugar content making it more valuable for this purpose than blackstrap molasses. Its use in cattle feeds has been experimented with but because of the higher cost it could only displace blackstrap in emergency conditions.

A typical molasses made by this process analyzes as follows:

Brix	85.0
Sucrose	27.0
Reducing sugars	50.0
Ash	2.25
Water	15.50

It will be noted that the dry substance or "true solids" (100 – moisture) is very close to the Brix as determined by "double dilution" (see p. 526). This is because the solids present are largely carbohydrates with relatively little ash.[48] With blackstrap the Brix is always much higher than the total solids (5 per cent or more) because of the high ash and non-sugar content (see p. 526).

The clarity, purity, and flavor of some high-test molasses might make it of value for blending and in the manufacture of some foods and beverages, but the tariff entering the United States would be much higher if it were used for human consumption instead of for the uses to which blackstrap is put (see Sec. 7).

335. Inverted Sugar Sirups. Inverted sirups have also been made in Cuba and to some extent in the United States by dissolving washed raw sugar or other high-test sugar products in water to the desired density and inverting

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[44] Gonzalez Maiz, *13th Annual Proc. Assoc. Cane Sugar Tech. Cuba* (1938), p. 181.

[45] Fowler, *13th Annual Proc. Assoc. Cane Sugar Tech. Cuba* (1938), p. 227.

[46] Lopez Ferrer, *15th Annual Proc. Assoc. Cane Sugar Tech. Cuba* (1938), p. 199.

[47] Lima-Romero, *15th Annual Proc. Assoc. Cane Sugar Tech. Cuba* (1938), p. 197.

[48] Zerban, Mull, and Martin, *J.A.O.A.C.*, August, 1942, p. 763.

by either of the above means. Hydrochloric and phosphoric acids have been substituted for sulfuric acid.[49]

These inverted sugar sirups are generally press-filtered to make them perfectly clear. Many refiners produce inverted sirups of high purity, particularly in large cities where delivery by truck may be practiced. Their use in ice cream, candy, baking, and other food maufacture is definitely on the increase.

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[49] Aurioles, *9th Annual Proc. Assoc. Cane Sugar Tech. Cuba* (1935), p. 240.

CHAPTER 15

KEEPING AND REFINING QUALITIES OF RAW SUGAR

336. Introductory. Recognition of the importance of the physical and chemical characteristics of raw sugars other than the polarization has been increasing during the past twenty-five years. Formerly a sugar approximating 96 test was generally the only aim, but now the best-controlled factories strive for a sugar which will not deteriorate in storage and which will work well in the refineries. The prevention of loss of polarization and weight during storage and transportation is of great economic value both to the manufacturer and refiner; and although the working qualities of the sugar in the refinery interest the refiner more directly, the manufacturer is also concerned in order to avoid discrimination against his product.

Within the past decade the refining quality of sugar throughout the world has been adversely affected by the prevalence of variety canes which give refractory juices in the clarification station. More intensive extraction through heavier grinding and elaboration of maceration methods has also contributed toward making poorer raws, so that in spite of long study and careful control the sugars today offer problems to the refiner which he did not encounter in those melted ten or fifteen years ago.

KEEPING QUALITY

337. Control Laboratories. The first attempt to regulate the character of the raw product systematically was undertaken in 1914 by Dr. Spencer at the Central Control Laboratory of the Cuban-American Sugar Company at Cardenas under the immediate direction of the writer. Details of the methods used at Cardenas have been described by Tillery.[1] Later the Hawaiian Sugar Planters Association instituted a similar control and other sugar companies in Cuba and Puerto Rico have since followed the same general plan.

338. Desirable Characteristics. In order that a sugar will not deteriorate in storage it is generally agreed that it must have the following characteristics: (1) comparative freedom from insoluble matter, i.e., boiled from a well-clarified juice; (2) a hard, uniform and fair-sized grain free from "rolled grain" or conglomerates; (3) a moisture content in relation to polarization to conform with certain "factors of safety"; (4) an "unwashed" sugar, i.e., the crystals must be surrounded by their original film of molasses; (5) manufactured under sanitary conditions to reduce contamination by fungi, yeasts, and bacteria to a minimum.

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[1] *The Planter,* Dec. 3, 1921.

339. Cleanliness of the Sugar. The direct bearing of cleanliness on keeping quality might be hard to establish, but sugars boiled from poorly clarified juices would seem more likely to deteriorate. Particles of bagacillo and other organic matter hold moisture and serve as breeding places for microorganisms while colloidal matter probably affects the formation of sharp hard grains in the pan boiling.

As a rapid test, dissolve a weighed amount of the sugar in hot water, shake and compare the suspended matter against standard tubes. A convenient method of reporting is on a scale of ten, No. 10 containing no suspended matter, No. 9, 20 mg. per 100 grams of raw sugar and so on up to 180 mg. per 100 grams for a No. 1 sugar. A weekly check may be made by determining the insoluble matter by weight. This is done by dissolving 20 grams of the raw in 200 ml. of water, bringing to a boil, filtering through a tared alundum crucible, washing with hot water to remove all sugar, drying at 105° C., and weighing. The result in milligrams multiplied by 5 gives insoluble matter per 100 grams raw. Any sugar containing less than 40 mg. insoluble is extremely clean (No. 8 on cleanliness scale), while above 100 mg. is poor and marks the clarification of the factory as deficient.

Insoluble matter determined in this way gives only the larger particles and does not indicate the dispersoids and extremely fine particles which cause turbidity in the solutions and which are probably of greater importance from the refining viewpoint than the material which can readily be filtered out. Harman [2] reports that in Australia the amount of insoluble that can be filtered off with kieselguhr is found to be a more valuable figure than the dye-test, surface tension, non-dialyzable matter, etc., as it shows a better correlation with the filterability test than any of these so-called tests for colloids. This method would seem to avoid the objections to the simple filtration method cited above.

340. Character and Size of Grain. The size and general character of the grain have several important functions in connection with deterioration. A uniform, fair-size grain will purge more freely in the centrifugals, making it easier to attain the proper moisture-sucrose ratio for safety. The larger grain presents less surface and will therefore absorb less moisture during adverse storage conditions as conclusively proved by Owen,[3] whereas rolled grain, conglomerates, or grain clusters will occlude dilute molasses that may serve as an incubator for bacterial and mold growth.

The size, regularity, and formation of the grain can be examined by means of the projectoscope, an improved form of which is shown in Fig. 87. It consists [4] of a Bausch and Lomb Micro-Tessar Lens, 72 mm. focus, *C*, mounted in a rack and pinion combination, *D*, with suitable condenser, *F*. The light source is a 6-volt 108-watt Mazda lamp in housing, *G*, the whole being

[2] *Proc. 5th Congr. Intern. Soc. Sugar Cane Tech.* (Brisbane, 1935), p. 765.

[3] *The Planter,* **70,** 68 (1923).

[4] Meade, *Ind. Eng. Chem.,* **13,** No. 8 (1921).

mounted on an "optical bed," *H*, with a 45° mirror, *A*, at the top to direct the projected image horizontally. A screen (preferably a plaster of Paris plaque), is placed 73 cm. away to give a magnification of 10 diameters.

A small amount of the sugar to be examined is placed in a Petri dish, *E*. covered with sugar-saturated alcohol, the grains being separated by rubbing gently with the ball of the finger. The image of the sugar on the screen may be compared with a series of grill-like squares drawn on the screen itself, giving the average size of the grains on a scale of ten. A No. 3 sugar is 0.4 mm. square; No. 4 is 0.6 mm.; a No. 5 is 0.8 mm. and so on. Most sugars fall between a No. 3 and No. 7, so squares representing these five sizes only need be drawn on the screen, multiplied by ten to allow for the magnification.

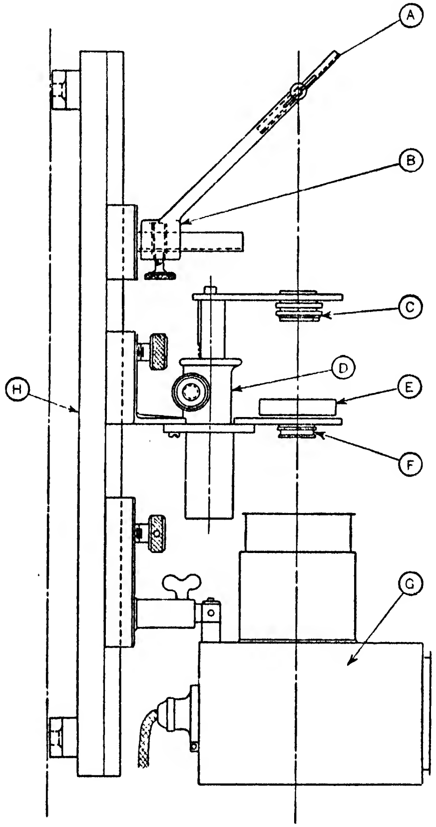

Fig. 87. Projectoscope for Examining Sugar Crystals.

A much cheaper form of projectoscope may be made in any laboratory by means of a simple tripod lens of the type commonly used by pan boilers. One arrangement of this sort is shown in Fig. 88, but other assemblies which the ingenuity of the chemist may suggest will serve equally well.

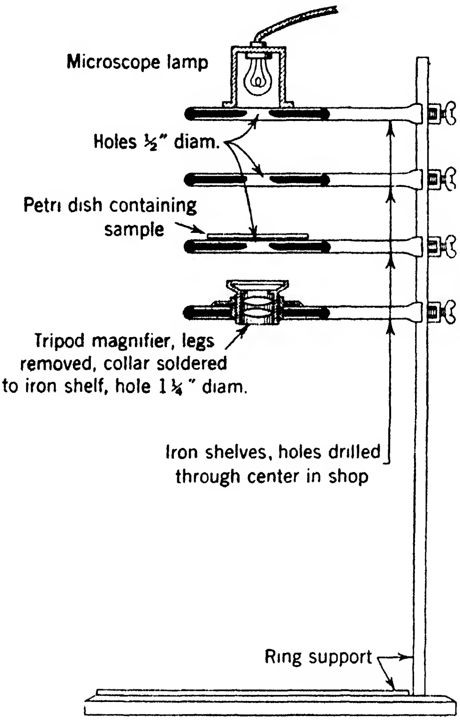

FIG. 88. Small Projectoscope.

341. Hardness of Grain. Hardness of the sugar crystal is difficult of determination; in fact some persons maintain that all crystals must be of the same hardness.[5] The work of Paine and Balch [6] shows that colloids and ash constituents of pan sirups were distributed throughout the volume of individual sugar crystals. Such crystals might easily be more friable ("softer") than a pure crystal. Harman [7] shows that much more impurity may be included in the crystal (after affination) of a raw sugar made from a turbid juice, even though grain characteristics, regularity in size and shape, are good. At any rate it is well recognized that many sugars feel soft when the crystals are rolled between the thumb and first finger, while others have a sharp, hard feeling and resist crushing to a greater extent.

342. Moisture Content and the Safety Factor. The importance of moisture content as an influence in the deterioration of sugars is now undis-

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[5] Norris, *The Planter,* **60,** Nos. 4 and 5.

[6] *Facts About Sugar,* June 12, 1926.

[7] *Loc. cit.*

puted, and it is agreed that the moisture percentage must be closely controlled. (The Spencer Oven, Sec. 524, is particularly well adapted to moisture control of sugars.) The relationship between the percentage of water and the non-sucrose has been shown to be the determining factor, since the microorganisms which cause deterioration cannot develop in solutions of high density. The Colonial Sugar Company of Australia were the first to state the limits of this relationship in a widely known "Safety Factor" which prescribes that the moisture must not exceed one-third of the non-sucrose; or expressed numerically

$$\frac{\text{Moisture}}{100 - \text{Polarization}} = 0.333 \text{ or less}$$

Many investigators have studied this point and they agree that this factor of safety is too high for many sugars and that to cover all cases the relationship should not exceed one-fourth, that is, for positive safety

$$\frac{\text{Moisture}}{100 - \text{Polarization}} = 0.250 \text{ or less}$$

These factors are calculated on the chart (Fig. 89) for polarizations ranging from 94.0 to 98.0. The use of the chart is simple. On the vertical scale at

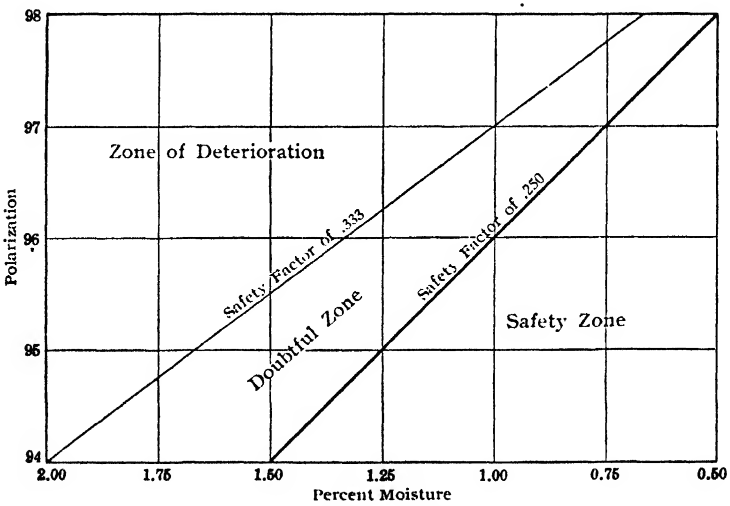

FIG. 89. Safety Factor for Raw Sugars.

the left-hand, note the polarization of the sugar, and on the horizontal scale at the bottom, the moisture. Follow the horizontal line out from the polarization until it crosses the vertical line from the moisture. If the lines cross to the right of the heavy line marked "Safety Factor of .250" the sugar will be almost sure to keep in storage; if it falls between the two lines it will probably keep provided all considerations such as warehouse conditions, character of grain, degree of contamination with microorganisms, etc., are favorable;[8, 9] whereas if the lines cross to the left of the lighter line marked "Safety Factor of .333" it will almost certainly lose in polarization if stored for any length of time. It is obvious that the greater the failure to conform to the safety factor the more rapid and severe the deterioration is likely to be.

As an example of the use of the chart: A sugar polarizes 96.2 and has a moisture of 0.90 per cent. It is within the "safety zone." If the moisture of a sugar of the same polarization were 1.05 per cent it would fall in the doubtful zone whereas a moisture of 1.35 per cent at 96.20 would definitely mark the sugar as likely to deteriorate.

Browne[10] has shown that a mixture of a "safe" sugar and one having too high a moisture ratio may show a "safe" average but the crystals too high in moisture will deteriorate and become foci of infection for other crystals.

343. Dilution Indicator. A modification of the safety factor is now used in Australia. It is called the dilution indicator (D.I.) and is derived from the equation

$$\frac{100 \times \text{Per cent moisture in sugar}}{100 - \text{Pol} - \text{Per cent moisture}}$$

or

$$\text{Dilution indicator (D.I.)} = \frac{100 \text{ moisture}}{\text{Solids non-sucrose (pol)}}$$

This is considered as giving a truer index of conditions within the molasses film on the crystal as there is a greater relative increase in the D.I. with an increase in moisture than there is with the safety factor formula.[11] Factors of safety of 0.25 and 0.33 correspond to dilution indicators of 33.3 and 50 respectively. It should be noted that in the older formula the moisture is included in both the divisor and the dividend, since the factor of safety is the moisture divided by the moisture plus the solids non-sugar. In the newer dilution indicator the moisture is eliminated from the divisor, and therefore a change in moisture makes a much greater change in the result. A further advantage is that the dilution indicator is expressed in whole numbers whereas the factor of safety is a decimal fraction. In the western hemisphere the factor of safety has become so well established that, in spite of the advantages of the D.I. figure, the adoption of the newer indicator is doubtful.

344. Washed Sugars. The safety factors outlined in the previous section depend upon the density of the molasses surrounding the crystals and are valid only for sugars carrying the original film of molasses. "The practice of

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[8] Browne, *J. Ind. Eng. Chem.*, p. 190, 1918.

[9] N. Kopeloff and L. Kopeloff, *Louisiana Bull. 176.*

[10] *Loc. cit.*

[11] Bell and Staunton, *Intern. Sugar J.*, April, 1935, p. 142.

washing raw sugar deprives their films of the protective action of the original molasses."[12]

Avice in an extended study of high-test raws in Mauritius[13] also found that sugars in which the molasses layer has been diluted by washing may invert even though the factor of safety is below 0.25. Owen and others have also noted that the factors are not dependable for sugars which have already undergone deterioration. So it is evident that the polarization and moisture determinations should be made as the sugar leaves the centrifugals in order that positive assurance of keeping quality may be had.

345. Bacterial, Yeast, and Mold Contamination. Extensive studies by many investigators, including Kammerling, Grieg-Smith, Owen, Browne, the Kopeloffs, Van der Bühl, and others have demonstrated beyond question that the loss of sucrose in raw sugars during storage is due to the action of certain types of microorganisms in the film of molasses surrounding the crystals. The ordinary soil bacteria, certain types of yeasts (torulae), and various mold fungi, as well as combinations of all three, have been shown to be the actuating influence in different cases. Owen[14] sums up the recognition of the various forms of infection as follows:

> Bacterial deterioration occurs only in dilute molasses films and gives abnormal Clerget values. If a sugar which was dried to a proper moisture ratio when manufactured evidences this type of deterioration excessive exposure to moisture during storage is indicated. Increase of polarization and loss in reducing sugars suggests the action of torulae, in which case there may be no real sugar loss. Losses in polarization without moisture absorption are almost invariably due to mold fungi if the "safety factor" is within the 0.333 zone. In such cases a careful study of sanitary conditions in and around the centrifugal and packing departments may discover and prevent the recurrence of this costly infection.

When it was first discovered that deterioration in storage was due to the action of microorganisms it was thought that bacteriologic methods would become as essential to sugar house control as the chemical routine. It is now thoroughly demonstrated that good clarification, good pan boiling, proper moisture control, and reasonably sanitary manufacturing conditions (particularly at the centrifugals) will produce a sugar that will not go off in test during ordinary storage periods, so that bacteriologic methods need not be made use of except in the rarest cases and then only by experts called in for the purpose.

Bell and Staunton[15] reviewed the conditions causing raw sugar deterioration in Australia and emphasized the necessity of maintaining temperatures of materials in process above 160° F. in order to avoid development of bacteria, molds, and fungi. Cleanliness and the use of uncontaminated water at the centrifugals were also stressed.

[12] Owen, *Facts About Sugar,* March 25, 1925.

[13] *Intern. Sugar J.,* July, 1936, p. 275.

[14] *Facts About Sugar,* June 30, 1925.

[15] *Intern. Sugar J.,* April, 1935, p. 142.

346. Absorption of Moisture during Storage. Reference has already been made (Sec. 302) to the necessity of storing sugar under such conditions that it will not absorb moisture. There have been many studies on this subject, one of which by A. G. Keller[16] contains a useful review of the literature on deterioration in storage. Keller's studies showed no absorption of moisture at 50 relative humidity, appreciable absorption at 75 relative humidity for certain classes of sugar and heavy absorption at 100 per cent. The amount of moisture absorbed increases with increase of temperature of the higher humidities, e.g., there is over twice the absorption at 30° C. that there is at 25° C. with 100 per cent humidity. Most investigators of absorption have worked to closer limits than Keller and have variously reported critical humidities from 60 to 70 per cent. Behne[17] suggests that 66 per cent relative humidity is the safe upper limit for Queensland (Australia) conditions.

A more elaborate study by Webster[18] along the lines of that done by Keller studied moisture absorption at 50, 62, and 78 per cent humidity respectively, each at 30°, 35°, and 40° C. The influence of ash, colloids, and reducing sugars together with polarization was also investigated. In general, all the sugars exhibited similar behavior, the moisture content increasing with both humidity and temperature, but the changes were not of the same order. Some showed relatively small increases, whereas in others the moisture increases were rapid. All the sugars in the latter class had high reducing sugar content, and it is clearly shown that reducing sugar has a very large influence on the hygroscopic properties of sugars. The organic non-sucrose content of the raw sugar governs equilibrium moisture content in contact with air of a given relative humidity, with reducing sugar by far the most important individual constituent.

The moisture content of the sugars in each group plotted against relative humidity for the three temperatures gave a striking illustration of the rapidity with which moisture increases with humidity and temperature when the reducing sugar content is high. With low values of this constituent the change is but small.

Scott[19] found that raw sugar in the vicinity of New Orleans could be kept without change of weight or test for nine months in a warehouse by maintaining the relative humidity always below 75 per cent and by raising the temperature when the humidity rose above this figure. (Figures are not given as to how much below 75 per cent the relative humidity fell when the heat was applied.) On the other hand, the same sugar lost 0.8 per cent in weight and decreased 4° in polarization in a warehouse without heat, i.e., without humidity control. The humidity of the outdoor atmosphere was generally above 80 per cent and frequently above 90 per cent during the storage period. The safety factor of the sugar in these tests was very low (less than 0.20) which

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[16] *Sugar J.*, **2, 25–30 (1940).**

[17] *Intern. Sugar J.*, **August, 1940, p. 284.**

[18] *Intern. Sugar J.*, **February, 1941, p. 46.**

[19] *Proc. Louisiana Sugar Cane Tech.*, **June, 1939.**

may be the reason why the maximum safe humidity was found to be at 75 per cent rather than at 65 to 70 per cent as other observers have found. Scott recommends humidity control of warehouses to insure proper storage but as Keller[20] says:

> Where sugar is stored in a well constructed warehouse which can be tightly closed . . . the moisture content [of the air] soon reaches an equilibrium with the sugar present and no further gain or loss of moisture by the sugar occurs. This practice of keeping warehouses closed at all times is much more satisfactory than the old practice of opening them for ventilation on dry days.

This statement is in accord with general experience in Cuba where well-made sugars have been kept in closed warehouses for two years with no loss in test. It might be added that large, compact stacks are also an essential part of this plan.

347. Changes in Storage Other than Loss in Test. Practically all investigators have studied the storage problem from the viewpoint of polarization losses and have disregarded changes which take place even though the loss of sugar may be negligible. These changes are of profound importance to the refiners, possibly of greater importance than the loss of polarization, since the latter is compensated for in the price paid for the sugar. A. P. Fowler and F. W. Kopfler in a commendable study of the changes in raw sugar during storage[21] showed that such changes may occur not only in polarization (and Clerget), moisture, and reducing sugars but also in color, dye number, filterability, pH, and insoluble content. They found that there is an increase in color in all raw sugars during storage (100 per cent or more in twelve months), but that the percentage increase is not in relation to the original color, i.e., a light-colored sugar may darken much more than a dark sugar, or vice versa. There is a direct relationship between the nitrogen content of the sugar and the color increase during storage, but the nitrogen does not seem to affect the original color. Slightly alkaline defecation possibly gives a sugar which will show less increase in color in storage than will a sugar from an acid juice. (This is contrary to expectation.)

The pH of fresh sugars ranges from 6.3 to 6.6 which decreases to 5.1–5.7 during twelve months' storage, and there is no relationship between the pH of the defecated juice and the pH of the sugar.

The lower the temperature at which the sugar is stored, the more nearly will it adhere to its original characteristics. Samples kept at 0° C. showed little change in polarization, color, dye number, reducing sugars, and pH during seven months, whereas the same sugars at 53° C. increased 400 to 500 per cent in color, doubled in dye number, and dropped below 5 pH, with only moderate changes in polarization and reducing sugars. The series at room temperatures (Cuba) showed intermediate changes in color, dye number, and pH, but no loss in test or change in reducing sugars. The sugars in this set of tests were well made and of a safety factor below 0.25.

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[20] *Loc. cit.*

[21] *9th Annual Proc. Assoc. Cane Sugar Tech. Cuba* (1935).

These investigators also found that although several sugars examined did not lose in polarization they showed an increase in reducing sugars and a drop in Clerget sucrose, indicating a destruction of levulose accompanied by an inversion of sucrose. Such instances result in no apparent deterioration as judged by the polarization but mean a large loss to the purchaser or refiner of the raw. They also found that in the sugars in which this destruction of levulose occurred (probably by the action of torulae) there is an abnormal increase in the color and dye number during storage, both of which changes would make the sugars more difficult to refine. A finding directly at variance to this is reported by F. W. Hayes[22] for Natal (South Africa) sugars. Torulae-infected raws in which the Clerget is lower than the direct polarization after storage were lighter in color than when produced.

The apparent relationship between nitrogen content and increase in color during storage as found by Fowler and Kopfler is a point on which further study should be made. The difference noted above in color changes in the Cuban and Natal sugars may be explainable by differences in nitrogen content. The whole subject of changes in storage other than loss in test is one on which extended work could profitably be done. Recent experience in refineries of the United States indicates that large unknown losses occur when old sugars (not necessarily deteriorated in polarization) are melted.

REFINING QUALITY

348. General Considerations. The characteristics desired by the refiner are: (1) a sugar that will "wash" well, i.e., give a maximum yield of washed sugar of high purity with the use of a minimum quantity of wash water in the centrifugals, thereby assuring a relatively small amount of "raw sugar washings" or "affination sirup"; (2) a sugar that will filter rapidly in pressure filters or defecate well in the Williamson process; and (3) a sugar of such color that it will be decolorized readily during char filtration.

In recent years many sugars have tended to give hazy char filtered liquors throughout the entire running of the filter. This haze due to some fault in clarification at the factory is most objectionable and difficult to deal with. This does not refer to the so-called "smoky liquor" of first runnings (see Sec. 386), but to a persistent haze in all liquors.

R. W. Harman has developed a series of tests for the Colonial Sugar Company of Australia which will be cited later. He says, "The fundamental cause of poor quality sugar originates in the cane and unusually poor quality sugar is generally attributable to some abnormal conditions of the cane or its growth whether due to cane variety, soil, cultivation, climate or damage."[23] However, manufacturing methods play a great part in the refining quality of the resulting sugars, and Harman places good clarification as the prime requisite

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[22] *Proc. 11th Congr. South African Sugar Tech. Assoc.* (1937).

[23] *Proc. 5th Congr. Intern. Soc. Sugar Cane Tech.* (Brisbane, 1935), p. 768.

for good sugars, followed in importance by proper sugar boiling and, of lesser effect, good purging.

Besides these primary characteristics the refiner also prefers a sugar of low ash-glucose ratio because such sugars in general give higher yields of granulated sugar; also a low sulfate content in the ash is preferable to avoid scaling evaporator tubes and to insure freedom from turbidity in final sirups. It is obvious that, other conditions being equal, the higher the polarization the easier a sugar is to refine, but this alone is not a criterion of refining quality.

Though practically all refiners would concur in the above statement of desirable characteristics, agreement as to methods for determining these characteristics would be difficult to obtain. Wayne[24] says, "It is our belief that if the sugar conforms to certain specifications for color, grain and filterability that most of the other considerations are automatically taken care of at the same time." Horne[25] outlines certain laboratory tests with a view of paralleling house conditions, such as washing in a small centrifugal, filtration through bag filter cloth, and decolorization by means of powdered boneblack. Other refiners feel equally sure that only actual practice in the refinery will demonstrate the refining quality.

To the producer of raw sugar the primary concern is keeping quality, after which he is anxious to please the refiner only in so far as it is commensurate with highest yield and economic factors. However, it may be said with certainty that those factors which favorably influence the refining quality of a sugar, good clarification and good pan boiling in particular, will result in better yields in the factory. The factory owner is therefore contributing to his own success when he strives for a good refining sugar. To obtain these results the manufacturer need not concern himself with the more elaborate tests given below. If he makes a clean sugar, well boiled and well purged, with an even, hard grain of medium size, from a brilliant juice, his product will almost certainly be acceptable.

349. Affination Quality. *Size and Character of Grain.* The same considerations which make size and uniformity of grain desirable for good keeping quality hold for a sugar which will work well in the refinery wash-plant or affination centrifugals. A hard, uniform, medium-size to large crystal purges freely, presents less surface for the wash water to act upon and in turn carries less molasses on its surface to be removed by the washing process. Conglomerates and fine grains form a mat in the centrifugals, do not drain freely and require more wash water which dissolves the crystal more easily because of the greater surface presented, giving an excess of affination sirup. The methods of pan boiling used in making the raw (see pp. 204–214) also influence the affination quality greatly. If the nucleus of the crystal (seed grain) is dark colored, this color will remain in the washed sugar, and the resulting melted liquor will be dark colored also.

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[24] *The Planter,* August 15, 1925.

[25] *J. Ind. Eng. Chem.,* October, 1918.

The size and character of the grain as determined by the projectoscope for keeping quality will also serve for the affination quality.

Laboratory Affination Test (Horne). Mix 100 grams of raw sugar with 45 ml. water, shake to saturation, and strain through a small piece of cotton cloth. Pour this sirup, which should be 92 ml., into the laboratory centrifugal basket with 200 grams of the raw sugar, mix, and spin until dry. The weight of washed sugar left in the basket, divided by 2, gives the percentage yield of washed sugar.

Harman[26] gives a more elaborate test, which consists of a laboratory affination and a filtration test of the affined sugar and the raw washings obtained. The laboratory affination is carried out in a 12-in.-diameter centrifugal under conditions similar to those in actual refining practice, and with all details standardized and closely controlled. The chief details follow.

A mixing sirup is first prepared by purging successive portions of the sugar under test, first with water and then using the sirup spun off for each subsequent purging, until a sirup of about 95° purity and 70° Brix is obtained. This mixing sirup is adjusted to 7.5 pH; 7.23 lb. is heated to 140° F.; 10 lb. of the raw sugar is added and stirred to a magma. After three minutes, this magma is added to a 12-in.-diameter centrifugal (one minute charging) and purged in four minutes, the cycle being: purging 60 seconds, washing 15 seconds, drying 130 seconds, and braking 35 seconds, while the 12-in.-diameter centrifugal accelerates to and runs at 2000 rpm. according to a definite schedule. For the washing, 203 ml. water at 180° F. is applied through a vortex spray. The quantity of mixing sirup is equivalent to 120 gal. per ton of raw sugar, the wash water to 10 gal. per ton, and the yield of affined sugar is 90 per cent. Filtration rates are then determined on sugar and raw washings, using standard conditions for the sugar (0.4 per cent supercel 60° Brix sirup for sugar), but for the washings a 54° Brix solution is used at 7.5 pH with 1.5 per cent supercel on solids.

Investigations with this test showed that conglomerate and aggregate grain is the most difficult to refine, and that mixtures of different raws gave lower filtration rates after affination than the average of the separate rates. This corroborates refining experience that a small quantity of poor sugar may exert a very adverse effect on a melt.

A further investigation of the cause of turbid liquors off char, or smoky liquors (see Sec. 386), as they are sometimes called, indicated that the cause lay fundamentally with the raw sugar, but the trouble may be aggravated by the condition of the char. Moreover, the type and efficiency of the clarification in the refinery has an important influence on this, one of the refiner's most troublesome difficulties, carbonation diminishing the trouble very much more than supercel filtration.

350. Filterability. *Importance to the Refiner.* Most refiners now add an inert filter aid such as kieselguhr to the washed sugar liquor and filter through

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[26] *Loc. cit.*

pressure filters; but several have the Williamson clarification system which uses phosphoric acid and lime but no filtration. In general a raw sugar which works well in any one of these systems will work well in the other, and the term filterability is here used to include the defecation as well as the removal of the precipitate, no matter what the material or means used. The wide variation in the filtering quality of different sugars and its effect on costs has long been recognized, since a poor filtering sugar not only slows up the entire refining process but also requires more defecating material or filter aid, both of which are costly.

Colloids and Filterability. Investigations as to the cause and the direct and indirect measurement of this quality have been numerous. The work of Walter E. Smith [27] and others in Hawaii and of Badollet and Paine [28] on Cuban raws have shown the close relationship between filterability and colloids and the finer dispersoids. Smith showed a direct relationship between filtration rate (by the Elliott test) and "non-settling matter" which is the suspended matter not removable by filtration through fine linen, or by centrifuging. This "non-settling matter" Smith claimed to be largely cane wax which the clarification had failed to remove. He further showed that the raw sugar solutions after filtration through the Elliott apparatus, i.e., after the colloidal material had been removed, filtered as rapidly on refiltration as a refined sugar solution of the same density. R. H. King [29] failed to corroborate Smith's findings, particularly as to the importance of cane wax, his conclusions being that the cause of the retardation of the filtration rate is the presence of gelatinous colloidal suspensions, both inorganic and organic, which form a pasty mass on the filtering medium and stop the flow of the filtrate. These gelatinous suspensions in the raw sugar are mainly due to the poor clarification of the juice.

Badollet and Paine correlated the actual filtering qualities of a number of sugars in a large refinery, their colloid content by ultra-filtration, and their "dye test value" (a rapid method of estimation of colloids as described below) showing that the filtering qualities were inversely as the colloid content and the dye test value, i.e., colloids or colloidal material are the cause of poor filtering quality.

Fowler and Kopfler [30] found that sugars with low dye numbers almost invariably showed a high filtration rate and, generally speaking, vice versa; but a few sugars with high dye numbers were found to filter well. These writers conclude that a low dye number indicates a good filtering sugar, but a high dye number is only a caution signal to be corroborated with a filtration test.

A correlation between the gums (alcogel) and filtration rate by the Elliott method is shown by du Toit.[31] The filtration rate dropped sharply as the

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[27] *The Planter,* October 11, 1924.

[28] *The Planter,* December 25, 1926.

[29] *The Planter,* **79,** 221, 242, 287 (1927).

[30] *Loc. cit.*

[31] *Proc. 11th Congr. South African Sugar Tech. Assoc.* (1937).

alcohol precipitable matter rose above 0.30, being reduced by nearly two-thirds for sugar over 0.50 per cent of alcogel.

351. Filtration Tests and Apparatus. The first device for determining filterability was developed by R. D. Elliott about 1923 and came into rapid use. The method uses vacuum at room temperatures and is simple and convenient, but unless conditions are strictly standardized the results are not reproducible by different observers or even, in certain cases, by the same observer. A more elaborate test, simulating refinery conditions, filters the liquors under pressure at 180° F. and will be described later. Much confusion had arisen by the careless use of both methods, and in 1932 the writer suggested to Dr. F. W. Zerban of the New York Sugar Trade Laboratory that he undertake a systematic study of filtration tests. The description of the apparatus and tests here given are based on his findings.[32]

The Elliott test was found more convenient and simpler and was recommended for routine tests, but the pressure apparatus described later is recommended for investigation work because it more nearly approaches refinery filtration practice both in procedure and in results.

Elliott Filtration Apparatus. R. D. Elliott devised an apparatus which is described by A. A. Blowski[33] as follows:

> Each filtering unit consists of a leaf 4 inches in diameter, equipped with a coarse-mesh screen on each side, over which standard cotton filter-cloth is fastened. For each filter leaf there is provided a trough to hold the unfiltered solution, a bottle to receive the filtrate, and a short piece of vacuum tubing to connect the leaf to the filtrate bottle. Each filtrate bottle is connected to a vacuum manifold which in turn is connected to a vacuum pump.
>
> A 1500-gram sample of the raw sugar to be tested is dissolved in 1500 grams of water at room temperature (20° C.). Thirty grams of kieselguhr, or 2 per cent on the weight of sugar taken, is added to the solution and mixed thoroughly. After passing through an 18-mesh screen the prepared solution is transferred to the unfiltered liquor trough in which the covered filter leaf has been placed. After allowing the solution to stand for two minutes without further agitation, the vacuum is applied in such a way that it increases to 26 inches in two minutes. With the aid of a mercury seal the vacuum is maintained at 26 inches for the duration of the test. At the end of 30 minutes the test is complete, the vacuum is broken and the filtrate weighed.
>
> The weight of filtrate in grams, divided by 2658 and multiplied by 100, gives the filtration efficiency of the sugar. The standard weight of 2658 grams of filtrate, representing 100 per cent efficiency, was adopted simply because it happened to be the quantity of filtrate obtained from the best filtering sugar available at the time the test was developed. It was found that a standard sugar is not necessary, provided that a uniform cloth is available and all other conditions of the test are maintained constant.
>
> It should be mentioned that the temperature must be kept uniform, as a difference of two or three degrees centigrade makes an appreciable difference in the result. The test is conducted at room temperature because of the difficulty of maintaining uniform high temperatures, particularly when using

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[32] *Facts About Sugar,* January, 1934.

[33] *Facts About Sugar,* Aug. 8, 1925.

vacuum. This is a radical departure from operating conditions, but it adds greatly to the simplicity of the test.

The Zerban investigation suggested the following modifications which were agreed upon by the great majority of those who had supplied data for the investigations: Use "Filter Cel Laboratory Standard" prepared by Johns-Manville Corporation, Manville, N. J., and a fresh piece of standard filter cloth furnished by California Cotton Mills, Oakland, Calif., specified as 2-yd., 40-in. dull, 70 by 84 weave (trade name M-451 Calcot); the standard temperature is 27.5° C. The filtration rate of 100 per cent is taken as 3000 grams, so that to calculate the percentage rate divide the grams of 50° Brix filtrate by 30.

The results found by the methods outlined by Zerban are to be reported as "revised Elliott filtration rate." It will be noted that the need for a standard sugar which was suggested in the original Elliott test has been dispensed with in the revised method.

This filtration test is entirely arbitrary and it is therefore necessary to adhere strictly to standard conditions. The arrangement of the filter cloth on the filtering unit must be done with great care and exactness, and all connections must be absolutely airtight. Even with the precautions outlined above there are still maximum differences of 6 per cent between duplicate determinations by skilled operators. Any carelessness in the use of this test will give very misleading results.

352. Filtration Test Under Pressure. This is a much more accurate test devised by the technical staff of the Johns Manville Corporation.[34] It simulates refinery conditions in one very important particular, viz., the filtration is done at 80° C. whereas the Elliott is at room temperature. For any important research, this test is to be recommended as against the simpler Elliott procedure.

The filter unit illustrated in Fig. 90 consists of four complete bomb presses immersed in a common thermostatically controlled oil bath and connected to a common air pressure supply. Each of the bombs is provided with both mechanical and air agitation and with a separate reserve liquor tank. Pressure can be maintained either automatically by means of a time-pressure regulator or manually by means of a hand-operated pressure-reducing valve. Each bomb is provided with a precoat chamber for applying a uniform filter aid precoat under pressure at the start of a run. Parts coming in contact with the liquid being filtered are of brass, bronze, copper, or Monel construction.

Individual bombs are of cast-brass constructions, 5 in. in inside diameter and 11 in. deep, with diameter narrowing to 3 in. at the bottom to reduce dead space around the filter leaf. Air agitation is provided by a $\frac{5}{16}$-in.-diameter copper tube perforated in the bottom portion except for the part directly under the filter leaf. Filter leaf generally used is of brass and is 1.5 in. in

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[34] Cummins and Waymouth, *Ind. Eng. Chem.*, April, 1942, p. 393.

diameter. When no precoat is employed, a threaded filter plate cap is used, ⅛ or ½ in. deep, depending on anticipated filter cake thickness.

The maximum working capacity of the bomb is 0.687 gal. (2600 ml.) which allows the filtrate volume of 0.555 gal. (2100 ml.). A suitable quantity of raw sugar or washed raw sugar is accurately weighed out in a pan, and the calculated amount of filtered water is measured or weighed out and added. Raw sugar tests have generally been run at 60° Brix and washed raw commonly at 65° Brix. In runs with lime addition, somewhat less than the total amount of water required is initially added in order to allow for water later added as milk of lime suspension. The mixture is heated with agitation to approximately 158° F. (70° C.), the milk of lime suspension is added to bring the liquor to the desired pH, and water is added if necessary to adjust the liquor to the density desired. The liquor is then split up into as many separate batches as required, a previously weighed portion of filter aid is then added, and the batches are heated to the desired filtering temperature, most commonly 176° F. (80° C.). The batches of liquor are transferred to the bombs by being poured through a 30-mesh screen to remove bag fibers, and agitation is started.

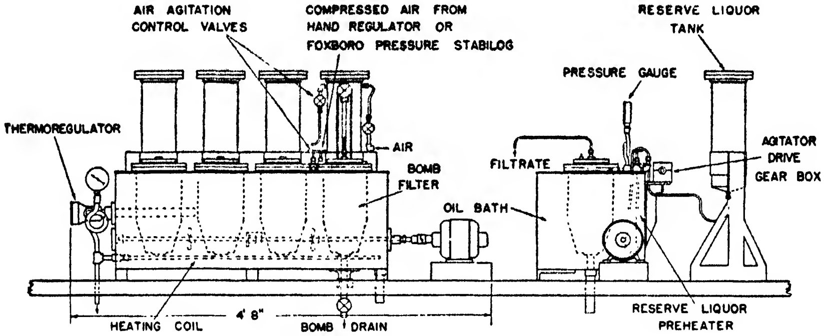

Fig. 90. Assembly for Filtration Tests.

Tests are generally run on a definite pressure-increase schedule which is controlled either automatically or by hand. Filtrate volumes are read at definite time intervals, and filtrate fractions are removed at suitable times for clarity measurement. Ordinarily clarity fractions are collected during equal time intervals for the entire test although in a short 30-minute test a single 15- to 30-minute filtrate fraction may suffice. After centrifuging and cooling where necessary, the filtrate samples are ready for clarity measurement. The same standard filter-cel and standard cloth are used as in the revised earlier test, a fresh dry cloth being cut for each test. To produce comparable results this test must be run under the same exacting standard condition at all times. For more complete details reference should be made to the original article.

353. Dye Test. This is a simple and rapid method for the approximate determination of the quantity of colloidal material in sugars and sugar solutions. The description here given is abstracted from the articles by Badollet and Paine of the Carbohydrate Division of the U.S. Bureau of Chemistry who devised the method.[35] This test was given great prominence when its use was first suggested, but later doubt as to the value of the findings was indicated by many observers. It has some value in testing raw sugars and other sugar products, but the results should be judged with caution.

The flocculation of colloids may be accomplished by the addition of free ions with an electric charge the opposite of that of the colloidal particles, or by adding a quantity of oppositely charged colloid sufficient to produce mutual electric neutralization. In general, the colloidal particles in sugar products are negatively charged. When a negatively charged colloid is mixed with a positively charged colloid in a certain proportion, a point of electrical neutrality is reached at which the charges exactly neutralize each other, frequently causing mutual colloidal flocculation and producing a precipitate that settles out of solution. If at this so-called isoelectric point the liquid containing the suspended flocs is examined with an ultramicroscope fitted with a cataphoresis apparatus, no movement of the colloidal particles or aggregates toward either electrode is observed. (Cataphoresis is the migration or progression of colloidal particles in a solution under the influence of an electric current.)

The cataphoresis apparatus consists of a U-tube, the center portion of which is capillary, and which has a platinum electrode in each arm (Fig. 91). The apparatus is fastened rigidly under the microscope, through which the motion of the colloidal particles in the capillary is observed. When the colloidal solution has been mixed with a portion of the solution containing the oppositely charged colloid, it is poured into the cataphoresis cell, and an electric potential is impressed on the electrodes. If the colloids present in the solution are not at a point of electrical equilibrium, they will progress toward the positive electrode if their charge is negative, and toward the negative electrode if their charge is positive. If they are at the isoelectric point, the point where the electrical charges are exactly neutralized, they will show no progression toward either electrode.

The electric charges of the colloids in sugar liquor may be neutralized by any of several dyes, night blue being the one used most extensively, because it produces the most rapid flocculation and a precipitate that settles most rapidly. In making the test 1 gram of the solid dye is dissolved in distilled water, and made to the mark in a liter flask. The solution to be tested should be first adjusted to some standard pH value. This standard is set at 5.2 for raw cane juice and defecated juice, and at 6.0 for other raw house

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[35] *Intern. Sugar J.*, **28**, 23–28; 97–103, 137–140 (1926); *The Planter*, **79**, No. 7 (1927).

and refinery products. The pH of the solution to be tested should always be recorded, since the dye number varies markedly with pH.

The dye number is calculated as follows. The weight of dry solids used in the test is determined, either by weighing beforehand, as in the case of raw and washed sugars, or by measurement and the use of the specific gravity or the refractive index. Then the number of milliliters of dye solution necessary for neutralization of the colloids in the sugar solution, multiplied by 100, and divided by the weight of dry solids used in the test, will be the dye number of the substance in question.

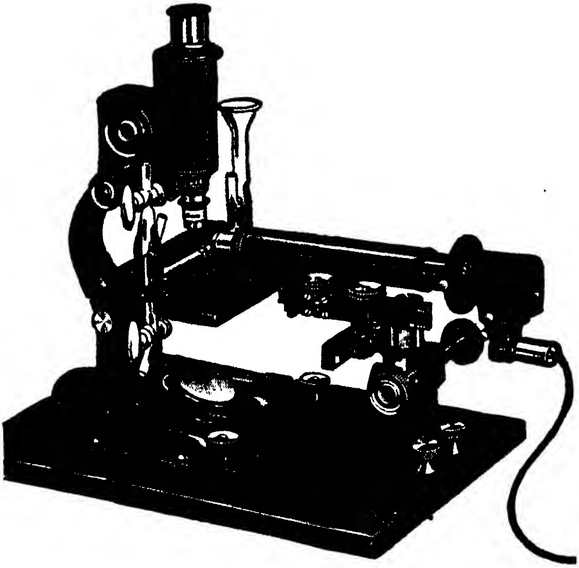

FIG. 91. Dye Test Apparatus.

354. Technic of the Dye Test. *Procedure No. 1 for Raw and Washed Raw Sugars.* Five grams of the sugar is dissolved in 25 ml. of distilled water, and the solution is filtered through a 100-mesh screen, which is then washed with small quantities of water. The filtrate and washings should be combined and diluted to a volume of 100 ml. in a 600-ml. beaker. A raw sugar solution prepared with distilled water will generally have a pH value of about 6.0, and adjustment of pH is not usually required. If, however, the pH value is found to vary from 6.0 by more than 0.2 or 0.3, the pH of the solution should be adjusted to the value 6.0 in order that all raw sugars examined may be on a comparable basis so far as influence of pH is concerned. The pH of the raw sugar liquors may be adjusted by the addition of small amounts of 0.05 normal sodium hydroxide or hydrochloric acid and tested separately with an indicator solution in any convenient comparator.

Ten milliliters of the standard dye solution is added to the 100 ml. of sugar solution, which is then tested in the ultramicroscope. If the colloid particles move toward the positive electrode, they still carry a negative charge, and the stopcocks are opened and the solution is drained into the original beaker. More dye solution is then added, and after mixing well, the sugar solution is retested in the cataphoresis cell. This process is repeated until the colloid flocs fail to move progressively toward either anode or cathode when current is applied at the electrodes.

For example, suppose that the sugar solution required 20 ml. of dye solution for adjustment to the neutral or isoelectric point (zero progressive movement of the colloid flocs). Then,

$$\text{Dye value of the sugar tested} = \frac{20 \times 100}{5} = 400$$

As a check on the electric neutralization of the colloid particles, add a small excess of the dye after the neutral point is reached. This will reverse the electric charge in the colloid particles, causing them to become positively charged and to move toward the cathode. The volume of dye solution used in the calculation of the dye value should, of course, be that which *just* causes electric neutralization.

Procedure No. 1 is sufficiently accurate with raw sugars of a moisture content as high as 2 per cent, but for sugar of a higher moisture content it is preferable either to dissolve the sugar to a 10° Brix solution and use Procedure No. 2, or to weigh out 5 grams of the sugar, as in Procedure No. 1 and correct for its moisture content.

It is advisable to wash a portion of each sample of raw sugar examined to 99° purity and to determine the dye value of the washed sugar as well as that of the original sugar. The purpose of this scheme is to ascertain the relation between the quantity of colloids eliminated by washing and the quantity remaining in the washed crystals. This step is important in view of affination of the raw sugar before filtration and subsequent treatment of the melt. Raw sugars of good refining quality when washed to 99° purity give a dye value of approximately 100 or less, whereas some raw sugars of poor refining quality when washed give dye values well over 100, sometimes as high as 200.

Procedure No. 2 for Raw Cane Juice, Defecated Juice, Filter Press Juice, Massecuites, Sugar, Sirups, and Molasses. The dye value of the raw cane juice, defecated juice, and filter press juice may be determined at the original density. But for massecuites, sugars, sirups, and molasses, considerable time may be saved by diluting an aliquot of the double-diluted laboratory sample to 100 ml. for the dye test. Use distilled water. Weigh out a definite quantity of diluted liquor using a sample of such size that 15–25 ml. of the dye solution is required for neutralization and base the dye value calculation on the percentage of solids in the weighed sample. The percentage of solids in the weighed sample can be calculated with sufficient accuracy from the refractive index or specific gravity by hydrometer. The pH may be adjusted by the addition of 0.05 normal hydrochloric acid or sodium hydroxide if required; 5.2 is a convenient pH for raw and defecated juice, but 6.0 is best for other sugar house products.

After the pH is adjusted (if required) 10 ml. of dye solution is added and mixed thoroughly with the 100 ml. of liquor, after which the test is carried out as in Procedure No. 1.

355. Miscellaneous Notes on the Technic of the Dye Test. 1. The voltage across the cell should be around 200 volts. This can easily be procured by a battery of five 45-volt radio B batteries. A 50-watt lamp should be connected in series with the cell. The switch controlling the voltage across the electrodes should be of the reversible type in order to eliminate polarization.

2. Adjust the microscope carefully, so that it is focused at a point in the vertical central plane of the capillary at a distance of 0.293 times the radius of the capillary below the upper wall of the capillary. This focusing may be best accomplished by the following method. First see to it that the microscope is in the central plane of the capillary, and then focus on the upper wall of the capillary by using a suspension of powdered granulated sugar in sugar-saturated alcohol, and focusing rapidly on the highest visible particle before the suspension has had the time to settle. Then allow the suspension to settle, and after recording the reading of the vernier on the milled head, the microscope is focused on the lowest particle in the capillary. One-half the number of turns on the milled head (expressed to 100ths) necessary to change the focus of the microscope from the upper to the lower wall is multiplied by 0.293, and after refocusing on the upper wall of the capillary, the microscope is turned down the calculated fraction of a turn.[36]

3. Keep the stopcocks of the cataphoresis cell well greased.

4. In filling the cell, be sure that the liquid overflows at the top stopcock so that no air will be retained in the closed.chamber.

5. In making a test, be sure that the stopcocks are well closed.

6. When the colloidal particles are observed through the microscope (after the instrument is correctly adjusted), they should not show any *progressive* movement (neglecting Brownian movement) toward either electrode when the current is not applied. If the particles continue to move when the current is off, look for a leak in the stopcocks or for air bubbles in the closed chamber or for cracks in the glass. The colloid particles must show no progressive movement whatever before making a test.

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[36] In some cases, where the microscope is equipped with an objective of 8 mm. or less focusing distance, it will be impossible to focus on the lower wall of the capillary, and it will then be necessary to determine the radius of the capillary in terms of turns of the fine adjustment of the microscope by means of an objective of greater focus (for instance 16 mm.), and then focusing on the upper wall with the higher-powered objective and lowering it the calculated amount.

7. Always mix the dye solution and sample thoroughly before making a test in the cataphoresis cell. Try to standardize the test as much as possible, regarding original amount of dye added, number of milliliters added in successive fractions, time of stirring and standing, etc.

8. The cell should be cleaned every few days. Alcohol or dilute hydrochloric can be used satisfactorily, but it is necessary to remove all traces of the cleaning substance before another test is made.

356. Color of Raw Sugars. The amount and nature of the color of a raw are of obvious importance to the refiner. The factors which influence color of raw sugars are the nature of soil supplying the factory with cane, the ripeness of the cane, the clarification of the juice (particularly as to the avoidance of excess lime salts), the methods of pan boiling (see p. 206), and the prevention of overheating and caramelization throughout the process. The effect of storage on the color of raw sugar has already been referred to (p. 274), and it frequently happens that old sugars have twice or three times the color they had when they were fresh.

T. B. Wayne[37] made an extensive study of adsorption of coloring matters in sugar products by boneblack, using spectrophotometric color analysis. He found that the reddish color due to the caramelizing action of lime and heat on invert sugar is not necessarily the most difficult type of color to remove, as has so long been supposed by refiners. This red coloring matter is less easily removed when small proportions of char are used, as in most laboratory tests, but when the ratio of char to liquor is increased the adsorption curve of the reddish sugars continues in a straight line to almost complete removal, whereas for greenish-gray sugars the initial color removal is greater for small char ratios but falls off when the ratio of char to liquor approaches that of actual refinery conditions (60 to 100 per cent). In other words the residual color after intensive char filtration will be greater with greenish-gray sugar (color due to colloidal iron) than with reddish sugars (color due to incipient caramelization). The resulting refined sugars from the liquors in the first instance will have an ugly dead gray appearance.

E. W. Rice[38] found wide differences in the character of the coloring matter in various raws as evidenced by the rate of adsorption (Q values, see Sec. 592) by bone char. The amount of original color was by no means a measure of the amount of color in successive portions of filtrate or of the nature of that color. In a second article Rice[39] suggests that the refiner should purchase sugars of light color, if no other tests are applied, and this is undoubtedly true, as a broad generalization. However, sugars of light color made with some variety canes work much worse in the refinery than other sugars of greater color made from the older native canes.

A complete discussion of color determination methods is given in Chapter 24, and the methods for raw sugars in Chapter 29, "Analysis of Sugars."

[37] *Ind. Eng. Chem.*, **18**, No. 8 (1926).

[38] ***Facts About Sugar*, 1930, No. 12.**

[39] ***Facts About Sugar*, May, 1931.**

CHAPTER 16

SUGAR REFINING

357. Introductory. The process of cane sugar refining is simple in theory, but very complex in actual practice. The working up of the lower-grade materials, the handling of the sweet waters and the control of the bone char filtration involve an enormous amount of detail, much of which cannot be fully considered in this chapter for reasons of space.

A flow diagram of the refining process employing boneblack is given in Fig. 92. No attempt has been made to show the methods of producing soft sugars or specialty sugars as this would have further complicated the diagram.

The narrow margin, or difference between the price of raw and refined sugars, has tended to cause the concentration of the industry into large units. Shipping facilities and the multiplicity of grades of refined sugar have also helped in bringing about this concentration. The greater portion of the refined sugar used in the United States is now produced in about twenty large refineries situated in or near the principal seaports.

Since 1930 refining by vegetable carbons and by chlorine has developed to a considerable extent so that the production is no longer entirely by means of boneblack. In general, refineries using these methods are smaller and are located in the tropics.

Until recent years a traditional conservatism on the part of refiners prevented the publication of investigations relative to the industry. This dearth of literature on refining has led to the false impression among those engaged in other branches of sugar work that the process was cut and dried and devoid of scientific interest, but during the last twenty years many articles by refinery men, particularly on the subject of char filtration, have appeared in the scientific journals.

There is now in progress an exhaustive study of the action of bone char which is sponsored by the combined refining interests working in collaboration with the Bureau of Standards. A 900-page *Bibliography of Solid Adsorbents,* by Victor R. Dietz, published in June, 1944, is one of the valuable results of this project.

A reference book called *Elements of Sugar Refining,* by Bardorf and Ball has also been published, and a well-written set of articles by an anonymous correspondent which deal with refining up through the bone char filter station appeared in successive issues of the *International Sugar Journal,* July,

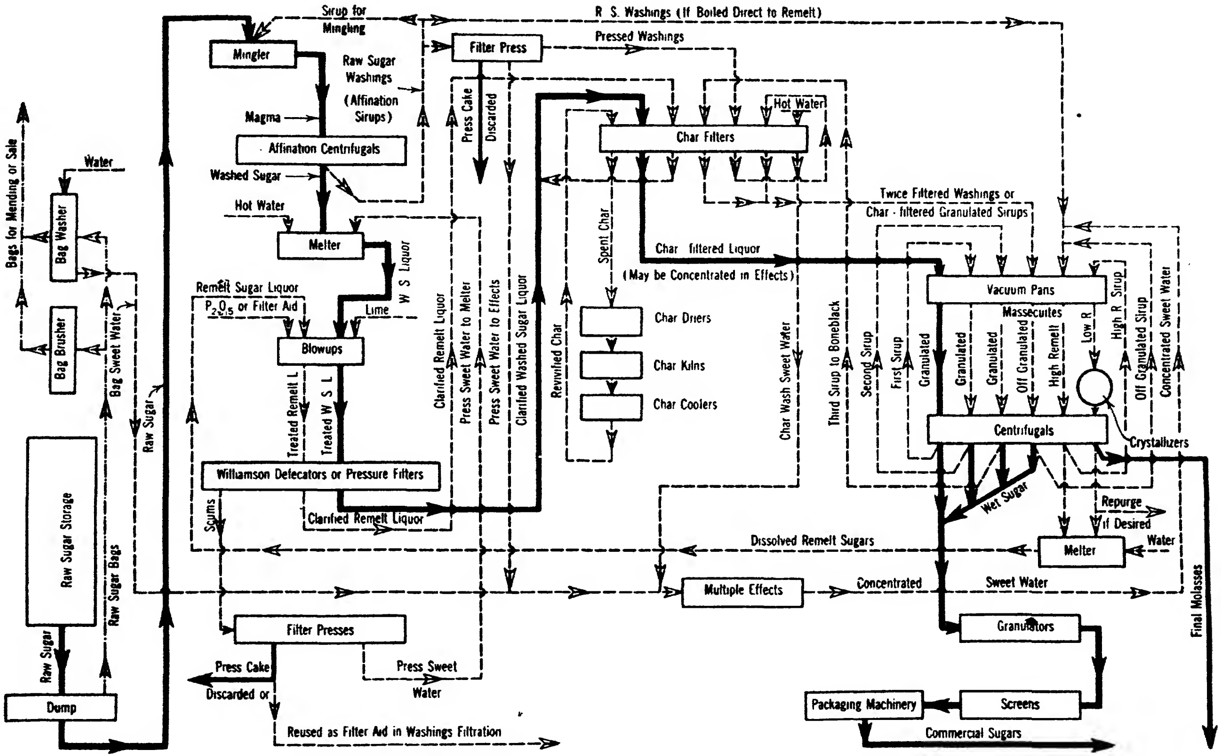

FIG. 92. Flow Diagram of Boneblack Refining Process.

1932, to March, 1933. The most recent book on refining by Oliver Lyle[1] deals with the subject from the English viewpoint but contains many new and interesting ideas for American refiners. The explanations of technical subjects for the non-technical worker are clear and easily understood.

358. Definitions. Refineries in continental United States may be divided into two classes: (1) those which produce (in addition to the ordinary commercial grade of granulated) the many different grades of sugar which are known as specialties (soft yellow and brown sugars, large-grain sugars for candy-makers, etc.), and (2) those which produce only the ordinary grade of granulated. The first class includes practically all the boneblack refineries, and the second class the refineries employing other methods of decolorization. The boneblack refineries have a wide variety of packages as well as grades so that the packing operation in these plants requires an elaborate department.

The term liquor is used in refining to refer to a heavy sugar solution from which no sugar has been removed by crystallization, that is, since its last treatment or char filtration. Sirup refers to a heavy solution from which sugar has just been crystallized and corresponds to the molasses of the raw sugar factories. It is to be noted that the term sirup in the refinery has an opposite meaning from the definition in a raw sugar factory, i.e., in the refinery a sirup is something from which crystals have been removed, whereas in the raw house it is a heavy sugar solution from which nothing has been removed by crystallization. A sweet water is a wash water which contains sufficient sugar to warrant recovery. A magma is strictly speaking an artificial mixture of crystals and sirup, but in some refineries the term magma is used synonymously with massecuite, as the term is known in the raw house. However, massecuite is also widely used in refined sugar terminology. Dissolving is described as "melting" the sugar, and the amount of raw sugar handled in twenty-four hours is termed the melt.

359. Raw Materials. Centrifugal sugars of 96° test or above form practically all the melt of the refineries. The present tendency, however, is for the raw sugar factories to produce sugars of higher test than 96° with a view to obtaining better storage qualities. The lower-grade sugars, cane seconds or molasses sugars, muscovadoes, Philippine mattes, concrete sugars, and palm sugars have practically disappeared from the market and most refineries have operated for many years on centrifugal sugars testing above 96°.

The character of the raw sugars received by the American refiners is indicated by the following statistics of the New York Sugar Trade Laboratory which represent all the sugars arriving at the port of New York. The average test of all samples received during the years 1908 and 1940, inclusive, are shown in the chart, Fig. 93. A comparison of the distribution of the various grades for 1914, 1927, and 1940 is as follows:[2]

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[1] *Technology for Sugar Refining Workers,* Chapman & Hall, 1941

[2] See also Zerban, *Sugar,* December, 1941.

Sugar Testing	Per Cent of the Total Number of Samples 1914	1927	1940
Above 98°	0.924	0.497	13.82
97–98	9.453	15.082	54.20
96–97	38.602	65.313	27.40
95–96	31.037	15.602	3.47
94–95	8.041	2.643	0.51
93–94	3.005	0.205	0.24
92–93	1.009	0.229	0.36

The tabulation and chart show the steady increase in the polarization of raws. In 1914, less than 50 per cent of the samples were above 96°; in 1927 81 per cent were above this grade, but in 1939 practically all samples exceeded 96° and two-thirds of the sugars were above 97°. In twenty-five years the average polarization has risen more than 2° which means that the refiner is handling only about half the amount of impurities that he did formerly.

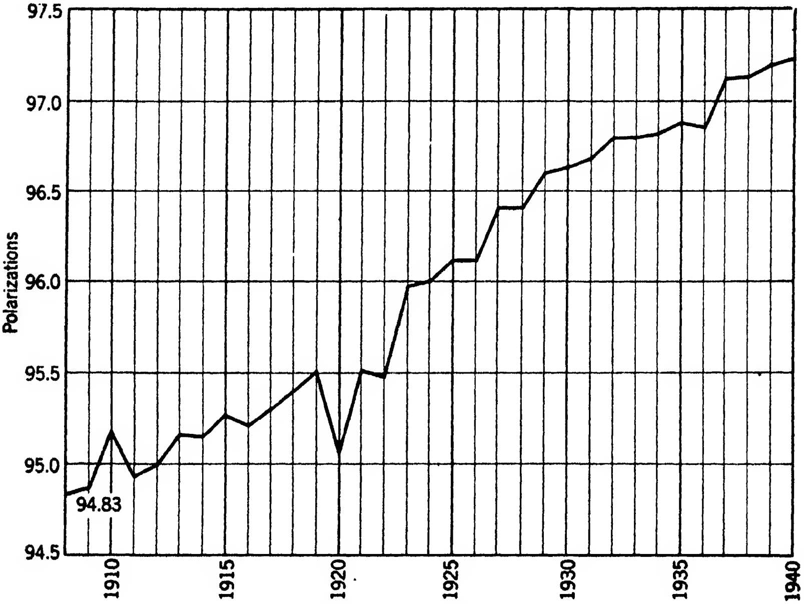

Fig. 93. Average Raw Sugar Polarizations.

360. Kinds of Packages. The raw sugars arrive at the refinery in various kinds of packages. Jute bags containing 300 to 330 lb. each are received from the West Indies. Hawaiian and Central American sugars are packed in smaller jute bags. Java sugars are packed in palm-leaf baskets. Formerly barrels and hogsheads were used for muscovadoes and concrete sugars.

361. Dumping or "Cutting In." All the sugar is weighed and sampled before it is dumped. The weighing is done in drafts of several bags or separately, depending on the method of handling. Where weighing is done by the government for duty purposes special electrically operated scales are used but large platform scales of the overhead suspension type are usual for public weighers or for refinery control weights. Sampling is done with a trier from each individual bag, though some refineries sample for control purposes after dumping.

Most of the larger refineries have installed storage bins for raw sugars which are placed above the minglers. The packages of sugar are hoisted directly from the ship, and after weighing and sampling are emptied into the bins. Sufficient sugar is dumped during the day to supply the refinery for the twenty-four hour period or longer, and the sugar is fed continuously by belt conveyors from the bins to the minglers. Larger bins for bulk storage are also in use (see Sec. 303). Raw sugars have also been shipped in bulk from Cuba and from Hawaii, requiring special machinery for handling.

Where storage bins are not available the sugar is dumped into bucket elevators and is carried directly to the minglers. Because of the prevalence of hard raw sugar during the winter months some auxiliary apparatus must be at hand for breaking up the sugar before going to the elevators. Slow-moving cast-steel spiral conveyors which intermesh have been found preferable to any fast-moving devices as less grain is fractured in the breaking operation. The empty jute bags are either brushed mechanically or washed with hot water for the recovery of the adhering sugar, then dried and sold. Many refineries now repair their own bags for return and reuse. The wash or sweet water is mixed with other similar solutions and is evaporated to a sirup and thus enters the manufacture. This sweet water should never be used for melting as it contains many objectionable impurities which should not be put into high-grade liquors.

362. Affination. Washing the Raw Crystals. The first step in the process of refining is termed affination, or more commonly washing, and consists of removing the adhering film of molasses from the surface of the raw sugar crystal. The molasses film has a purity of 65 or lower, depending on the class of massecuite from which it was boiled in the raw factory, whereas the crystal itself is nearly pure sucrose. The separation is accomplished by mingling the raw with a heavy sirup (65°–70° Brix) and then purging the mixture in centrifugals, washing with water after the sirup has been spun off.

The raw sugar enters the minglers, which are heavy scroll conveyors fitted with strong mixing flights, and is then mixed with water or remelt sirup at the beginning of the run to form a heavy cold magma or mash which drops into a mixer of the same style as those used in raw sugar work. With the mingling and the mixing the added water dissolves the molasses film (and a small part of the sucrose of the crystal) and forms a heavy sirup. After the sirup has been spun off, the sugar in the centrifugal basket is sprayed with water (6–10 qt. per 40-in. machine), and the washed sugar, which should be 99 purity or above, is sent to the melter as described below. After sufficient sirup has accumulated, it is used for the mingling process instead of water, as noted later.

Until the last two or three years the centrifugals used in the affination process were generally of the self-discharging type (see Sec. 288), and the capacity of these machines for this purpose was about 10,000 lb. of sugar per hour on a three-minute cycle. The old-style flat-bottom machines handle 6000–7000 lb. of raw per hour per machine. With the invention of the high-speed centrifugal (Sec. 291) these have come into use in several refineries in connection with the hot mingling process to be described later, and the capacity of one of these machines with hot mingling is approximately 13,500 lb. per hour. Obviously, the capacity of any type of centrifugal is influenced by the character of the raw sugar. The high-speed centrifugals are also fitted with effective means for making a sharper separation of wash sirup and greens (see Sec. 292), which is of value as will be noted later.

The sirup purged from the mash is returned to the minglers for mixing with the incoming raws instead of water. It is evident that with the successive minglings and purgings the raw sugar washings (also called affination sirup, wash sirup, and raw greens) will exceed the needs of the mingling process. That portion of the raw sugar washings not used for the mingling is sent to process for treatment as described below. In order to reduce sugar losses caused by material being on hand too long, it is good practice once every twenty-four hours to pump all the washings on hand to the process and to start the mash with fresh water or remelt sirup. The portion of the washings used for mingling (called sirup for washings) is diluted in some refineries to 65° Brix or is warmed to 120°–125° F.,[8] or both, since the object is to reduce the viscosity and facilitate the removal of the dense molasses film on the raw crystals.

The raw sugar washings vary in purity from 74° to 80° and constitute from 12 to 14 per cent of the weight of the raws melted with the ordinary cold mingling process. The amount of raw sugar washings will be greater as the test of the melt is lower. The character of the grain (Sec. 349) will also affect the amount, a small, soft-grain sugar giving more washings than a well-boiled, large-grain raw.

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[8] The Fahrenheit scale is used in most American refineries and is therefore used throughout this chapter.

Harman[4] found that conglomerates or mounted grain presented the most difficulty in affining a raw sugar, and this is to be expected from the nature of the process.

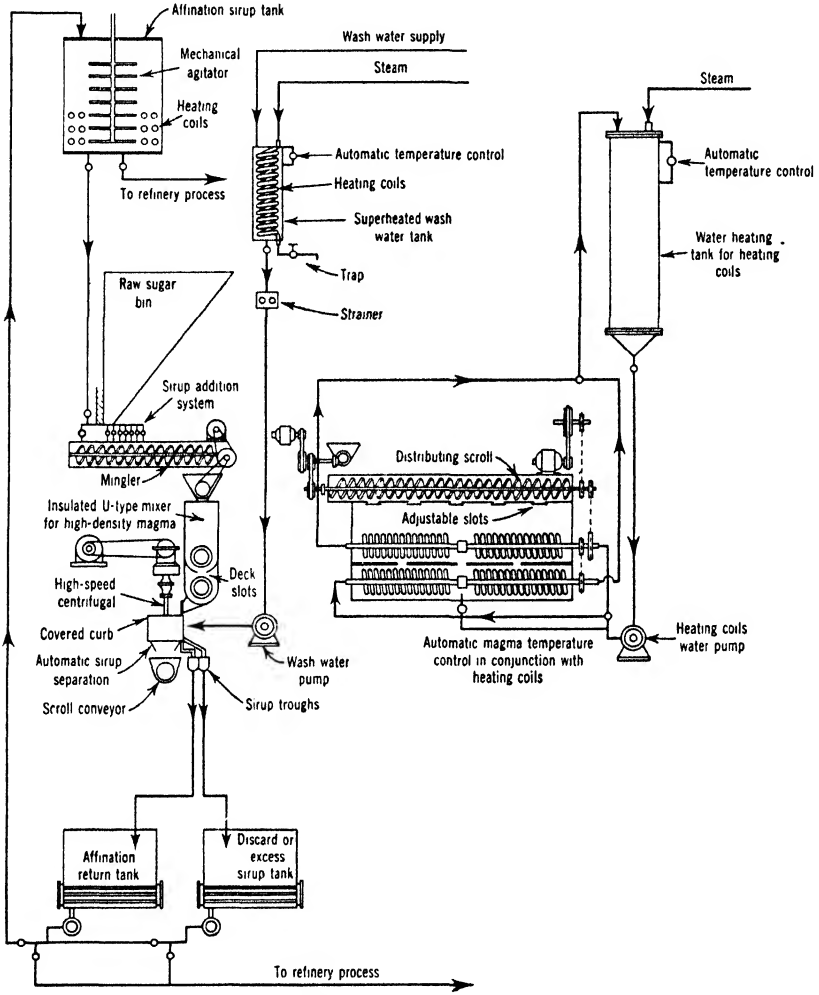

FIG. 94. Hot-Mingling Affination Process.

Hot Mingling. This process has been developed in conjunction with the recently invented high-speed centrifugal.[5] The primary purpose of the affination process is to reduce the viscosity of the molasses film surrounding the raw sugar crystal so that it may be readily removed by centrifuging.

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[4] *5th Congr. Intern. Soc. Sugar Cane Tech.* (Brisbane, 1935), p. 767.

[5] G. E. Stevens, *Intern. Sugar J.*, November, 1938, p. 440.

The more effectively this is done, the less water will be needed for washing the crystal after purging, and in consequence less of the crystal will be dissolved. Refiners have heated the sirup for washings or used a more dilute sirup, but the method used in this new development is to heat the magma to a temperature of about 165° F. just before it goes into the centrifugals. The heating is effected by means of rotating coils in the mixer (Fig. 94) through which water at 210° F. is circulated. The fluidity of the magma is naturally greatly increased by this heating, and it has been found possible to operate with a magma having as high as 95 per cent dry substance, as against 88–92 per cent with the old cold magma process. The greater effective centrifugal force of the high-speed centrifugals operating on the hot fluid magma purges the crystals freer of sirup and requires less wash water. The high-speed machines have also been found to permit the use of very hot water (180° F.) without dissolving an appreciable quantity of grain.[6]

The hot mingling, high-speed centrifugal combination therefore gives less washings to process and a correspondingly higher yield of washed sugar.

363. Dissolving or Melting the Sugars. The washed sugar is dissolved in about one-half its weight of water in a tank provided with mixing arms called a melter, exhaust steam being applied from a perforated coil to aid solution. High-test sweet waters from pressure filtration or the Williamson system are used in melting, but it is obviously bad practice to contaminate the melted liquor with impurities by using other sweet waters which are much below the purity of the washed sugar itself. Maintaining the test of the washed sugar liquor at 99° purity or above at all times aids the defecation and improves the grade of the char-filtered liquors.

364. Treatment of Affination Sirup. The affination sirups or raw sugar washings go to the process for treatment in one of two ways. The simpler method is to send the washings to the pan floor where they are boiled into high remelt strikes, but the practice in refineries with large char capacity is to dilute the affination sirup to about 54° Brix with dark sweet waters and pump them to the defecators for filtration through pressure filters and the boneblack.

DEFECATION

365. General Principle. The raw washed sugar liquor from the melter contains some insoluble material, such as bagacillo, clay, or sand, and an appreciable amount of fine suspensions and dispersoids as well as gums, pectins, and other true colloids that have escaped the clarification of the raw house or have been formed in the subsequent process of manufacture. The raw liquor is also acid as a rule. Similar considerations in a more exaggerated form hold for the raw sugar washings (affination sirup), which contain the major portion of the impurities of the original raw. Defecation may be defined as the treatment of the raw liquor or raw washings with certain sub-

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[6] Gutleben, *Chem. Met. Eng.*, **44**, 14 (1937), also personal communication.

stances and heat in order to render the solution suitable for filtration or clarification.

A great number of defecants have been suggested or experimented with in refining work. All have essentially the same idea, namely, the formation of a flocculent precipitate during heating to adsorb the colloids and enmesh the suspensoids and suspended matter so that they may be caught and removed by the meshes of a cloth or fabric. Blood or blood albumin was the defecant formerly used but its use was entirely discontinued many years ago.

The defecation, no matter in what form, is carried out in defecators (or "blowups" as they are generally called in refining practice) which are usually circular tanks with conical bottoms to facilitate drainage and cleaning, fitted with steam coils and air connections for agitation or preferably mechanical stirrers.

The raw sugar washings are defecated in separate blowups, in the same manner as the washed sugar liquor, the difference being in the quantity of defecant used. Many refineries also defecate other materials returned from the process—remelt sugars and granulated sirups—which are to be char-filtered.

366. Phosphoric Acid-Lime Defecation. Up to 1915 the defecating materials most commonly employed were milk of lime and phosphoric acid in conjunction with bag filters. This form of defecation is still essential in those refineries which employ the Williamson system. Inert filter aids and pressure filtration superseded the phosphate-lime defecant in the majority of the plants of the United States during the years from 1915 to 1930, but there is now in progress among the refineries a movement toward a return to the use of the phosphate treatment.[7] The amount of P_2O_5 needed for defecation varies from 0.025 to 0.05 per cent of solids in the melt. The P_2O_5 may be in the form of monocalcium phosphate, phosphoric acid paste, or clear phosphoric acid. In the old bag-filter days the monocalcium phosphate was produced in the refinery by the action of hydrochloric acid on fine discard boneblack. This black paste contained about 10 to 12 per cent available P_2O_5, but it contained chlorides which were objectionable as they passed through the char and increased the quantity of residual sirup. Pastes were also made by the use of sulfuric acid but the high sulfate content was objectionable. These methods are now of historical interest only.

Phosphoric Acid. Improved methods of manufacturing *ortho*-phosphoric acid direct from phosphate rock by smelting with coke and silica in a blast furnace or an electric furnace[8] result in an acid of 55 per cent P_2O_5 free from arsenic, lead, sulfates, or other objectionable impurities which were common in acid and pastes made by the older wet method of treating phos-

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[7] See Meade, "The Use of Phosphoric Acid in Cane Sugar Manufacture and Refining," *Proc. 6th Congr. Intern. Soc. Sugar Cane Tech.* (Baton Rouge, 1938), p. 1032.

[8] Curtis, *Trans. Am. Inst. Chem. Eng.*, 1935, p. 278.

phate rock with sulfuric acid. The higher class phosphate pastes on the market are made by mixing this pure acid with lime and these were used by refineries up to a few years ago. The most recent practice is to purchase the heavy phosphoric acid in bulk in rubber-lined tank cars and either use this direct or make a paste with fine discard boneblack. Sufficient phosphoric acid is used in making this paste to change the tricalcium phosphate in the boneblack to monocalcium phosphate. All the salts formed are phosphates, and no objectionable impurities are introduced into the liquors as with the other boneblack pastes made with hydrochloric or sulfuric acid.

A typical analysis of the *ortho*-phosphoric acid now produced is given in Sec. 161.

Lime. The lime which is used in refineries is the hydrated type or, more recently, pulverized quick lime (see Sec. 158). In either case it is made to a milk of fixed density (usually 20° Brix) and is circulated to the blowups and other parts of the plant where lime is used. The amount of lime used for the Williamson system is sufficient to give an effluent liquor of 7.0–7.3 pH, and it is good practice to add about half of the required lime to a blowup, then add the phosphoric acid (or paste mixed with water) followed by the remainder of the lime. The amount of P_2O_5 required varies somewhat with the class of sugar being melted, but the limits are not wide. With raw sugars of 97° test such as are now generally handled the defecants needed for the Williamson process will be per million pounds of melt, about 400 lb. of P_2O_5, and 800 lb. of lime (CaO).

Treatment. For the old bag-filter system it was customary to heat the treated liquor in the blowup to 180° F., but for the Williamson clarifiers it is preferable to work in the blowups at a lower temperature (140°–165° F.). There is considerable difference of opinion and in practice on this point, some refineries holding the treated liquor at as low a temperature as practicable with the idea that more air can be entrained in the colder liquor. Extended experiments have shown that proper aeration can be obtained with liquor at 165° F. and that the tendency to after-precipitation of tricalcium phosphate is less if the defecants are added at this temperature. The density of the washed sugar liquor in the blowups is adjusted to about 62° Brix or even higher if good defecation can be obtained with heavier liquor.

Action of Phosphoric Acid and Lime. The lime neutralizes the organic acids present in the raw sugar and the excess combines with the phosphoric acid to form tricalcium phosphate. The heating and defecants cause the coagulation and precipitation of the greater part of the gums, albumins, and pectins. The whole forms a heavy, flocculent precipitate which entrains with it the finely suspended materials derived from the raw sugar together with bagasse fiber, jute, etc., and occludes much of the colloidal matter, including a very appreciable amount of color. This color removal and elimination of colloids by the phosphoric acid-lime treatment is the reason for the increased interest among refiners in the Williamson system which, until recently, was the only practicable method for employing phosphate-lime defecation. The character of the color removed is also of importance as this color includes most of the colloidal iron compounds (polyphenols of iron) which impart a brown-green color to the washed sugar liquor. This color removal and color-tone improvement are evident by simple inspection of liquor defecated with and without phosphate.

Experience has shown that between 20 and 40 per cent of the total color of the melt liquor is removed by phosphoric acid, which means that the boneblack required for decolorization will be from 40 to 60 per cent less than with untreated liquors. Cummins and Morris[9] in the first published study of the effects of phosphoric acid with modern color and turbidity measurements found that with affination sirup treated with 0.1 per cent P_2O_5 as compared with Hyflo filtered sirup at the same pH (7.2), the color removal was 20 per cent at 495 millimicrons and 28 per cent at 620 millimicrons, and "the same amount of char will decolorize 50% more sirup treated with 0.1% P_2O_5 than it will untreated." Of equal importance with the color removal was the clarity of the phosphate-treated sirups. The turbidities in foot candles were:

	Before	After
Pressure filtered	61.5	38.8
Phosphate treated-pressure filtered	17.5	9.9

The turbidity of the phosphate-treated sirup before char filtration is much less than that of the untreated sirup after char filtration. These published results on affination sirup as to color removal, char efficiency, and clarity conform with many years' practical experience with phosphate-treated washed sugar liquors. The color removal varies widely with the character of the raws being melted, but in general the percentage removal is greatest with dark-colored raws.

367. Defecation with Inert Filter Aids. Diatomaceous Earth. Kieselguhr. This is a fossil deposit made up of countless microscopic skeletons of diatoms and consists of almost pure silica in a highly porous form. It is calcined and processed after mining. This material was first introduced into refining by Wiechmann, who suggested it as a filter aid in bag filtration some forty years ago. It is now used in the majority of the refineries in conjunction with pressure filtration.

The kieselguhr defecating process consists in neutralizing the acidity of the liquor with milk of lime (generally to pH 7.0–7.3) and then adding the required quantity of the kieselguhr while the liquor is being heated to 180° F. The amount of lime used is about 300 lb. per million pounds melt. The kieselguhr is most conveniently mixed to a cream with water and pumped to the blowups for use, as in the case of lime. However, somewhat higher efficiency is obtained by adding the filter aid dry, provided proper agitation disperses the dry material through the liquor promptly. The action of the

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[9] *Facts About Sugar*, February, 1938.

kieselguhr is largely mechanical. The lime and heat flocculate some of the colloids, and the porous mineral coats the fabric of the filter and retains the precipitate and suspended matter. There is some adsorption of colloids but little removal of color by the kieselguhr filtration. A full discussion of "Silica Filter-aid Clarification" by Elsenbast and Morris[10] gives details of methods and equipment now in use.

It was long recognized that the phosphate-lime defecation would not serve with filtration under pressure since the flocculent tricalcium phosphate is forced into the pores of the cloth, forming a gelatinous mass which stops the flow almost immediately.

With the introduction of the Kelly, Sweetland, and other Vallez leaf-type filters the use of mineral filter aids became essential, and the quality of these materials now obtainable is much superior to that formerly on the market. The Johns-Manville Company produces many grades, of which Filter-Cel, Super-Cel, and Hyflo, are those most generally used in the sugar industry. Filter-Cel gives the best clarity and the lowest speed of flow, whereas Hyflo gives the highest filtration rate but does not remove the finer dispersoids as readily as the two slower filtering grades. The Dicalite Company produces corresponding grades of kieselguhr for refining use with the names Superaid, Speedflo, and Speedex.

Practice varies in the different refineries in the use of the various grades, some using filter aids of slow filtration rate for pre-coating the presses, following this with those of higher filtration rate. Some refineries still use the filter aid having a middle rate of flow in order to insure clarity. The quantity of kieselguhr used varies with the character and test of the sugar melted, the grade of filter aid employed, and the filter equipment, but from 4 to 8 lb. per ton of melt for the filtration of washed sugar liquor would cover most conditions.

For the defecation of affination sirups it is necessary to use much more filter aid, as high as 40 or 50 lb. per ton of solids being required. It is customary to reuse the kieselguhr from washed sugar liquor filtration by beating this into a slurry with water and adding it to the sirup to be filtered. Precoating with fresh filter aid is general in this practice. The affination sirups are diluted to 50°–55° Brix and heated to 180° F. before pressure filtration.

368. Regeneration of Kieselguhr. The high cost of inert mineral filter aids has resulted in some large refineries installing Wedge multiple hearth furnaces for the regeneration of kieselguhr. The process is reported as satisfactory, although repeated regeneration causes a steady increase in the specific gravity of the kieselguhr with consequent reduction in filtration efficiency.[11]

369. Inert Filter Aids with Phosphoric Acid. The greatly increased interest in the use of phosphoric acid in refining has resulted in a number of

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[10] *Ind. Eng. Chem.*, April, 1942, p. 412; also Cummins, *ibid.*, p. 403.

[11] Elsenbast and Morris, *Ind. Eng. Chem.*, April, 1942, p. 417.

publications on the use of a small amount of phosphoric acid in conjunction with a filter aid of very high porosity, e.g., Celite 503. The amount of P_2O_5 recommended is about 0.01 per cent on solids in melt and flow rates are reported as high as with untreated liquors and standard or Hyflo Super-Cel.[12, 13]

These experiments give further proof of the recognition of the value of phosphoric acid in refinery defecation. The purpose of this combination is to secure the positive action of the filter aid in pressure filtration together with the removal of color and other advantages which phosphoric acid has been shown to give. (See Sec. 366.)

370. Defecation by Carbonation. This process is not used in the western hemisphere but is standard practice in refineries in England, Australia, and South Africa. In general the same procedure is followed as for carbonation in raw sugar practice (see pp. 115 to 119). A detailed description of the process will be found in the *International Sugar Journal* for September, 1932,[14] and in *Technology for Sugar Refinery Workers*.[15] Proof is given in the latter work (p. 254) that carbonation produces a sugar of much higher quality than do the practices used in refineries in the United States.

371. Paper Pulp as a Filter Aid. This is a patented process employed in conjunction with Vallez filters. The liquor is adjusted to the desired reaction with lime as before, and the pulp is thoroughly beaten up with water and then added to the blowups. About 0.1 lb. of dry pulp is used per square foot of filter area for pre-coating after which the proportion of pulp is about 1 lb. to each 75 gal. of liquor. The advantage of the pulp is that it may be washed and reused many times, replacement of the pulp daily amounting to about 150 lb. per million melt which makes the cost about one-tenth that of the phosphoric-lime defecation and still more favorable than that in comparison with kieselguhr. The disadvantages are that the pulp is purely mechanical in its action and does not remove the very finest dispersoids without double or triple filtration. Small quantities of phosphoric acid and lime have been added with the pulp by some refiners to obviate this difficulty.

The washing is done in a pulp washer similar to those used in the paper industry, a counterflow system being used to economize water. The washer agitates the pulp, passing it over a series of cylinders, which successively remove the water with vacuum between sprayings.

Paper Pulp and Phosphate Defecation of Affination Sirup. H. I. Knowles has described a successful process for filtering phosphate defecated affination sirups with paper pulp on Vallez filters.[16] The process is carried out at low

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[12] Cummins, *Ind. Eng. Chem.*, April, 1942, p. 348.

[13] Frankenhoff, *Ind. Eng. Chem.*, June, 1942, p. 742.

[14] Anon, p. 354.

[15] Oliver Lyle, Chapman & Hall, London, 1941.

[16] *Ind. Eng. Chem.*, April, 1942, p. 422.

pressures (5 to 7 lb. gravity feed). Low labor costs, long cycles, and satisfactory clarity are reported for this method.

CLOTH FILTRATION

372. Bag Filters. The Taylor bag filter was universal in American refineries up to 1915, but it has now been entirely superseded by pressure filtration or Williamson clarification. Phosphoric acid-lime defecation was employed with the bag filter which consists of a cast-iron casing or box, the top of which is perforated at intervals with holes, each having a short nipple fitted into it from which is suspended a filter bag or element. Each bag is made up of an outside sheath of loosely woven cord, about 6½ ft. long and 6 in. in diameter, and an inside bag of cotton-twill fabric which is 6 ft. long and 24 in. across.

The high labor cost, losses in sugar, and generally unsanitary and unsightly condition of the station have completely driven the bag filter from modern refining.

The defecated liquor is run onto the top of the filter, which is surrounded by a shallow curbing, and flows down through the bags, the precipitate being retained. The filtered washed-sugar liquor is of a light-brown color, quite free of suspended matter or turbidity. The filtered raw washings are also clear but of much darker color. After from fifteen to twenty hours' use the bags fill with mud and filtration becomes very slow. The liquor going on to the filter is now shut off, and the liquor remaining in the bags is sucked out by a vacuum pipe, or is allowed to drain out. The filter bags are flushed once or twice with thin alkaline sweet water from the filter presses, and the mud is further washed in the bags, as they hang in the filter casing, by introducing a jet of hot water into each successively. This sticking is repeated from three to five times, after which the bags are taken out of the filter and washed in a series of tubs to remove all the mud.

373. Pressure Filters. These are filters in which the cloth or filter fabric is held on a frame with sufficient backing so that the liquid may be forced through under pressure from the outside to the inside of the filter element. The pressure generally ranges up to 30 to 40 lb. per sq. in. As already explained pressure filtration involves the use of inert filter aids such as kieselguhr or paper pulp, since the tricalcium phosphate precipitate will not permit of any flow under pressures beyond the few pounds of hydrostatic head encountered in the bag filter.

The principle in all pressure filters is the same. The defecated liquor containing the filter aid is forced through the press by centrifugal pump pressure. The cake is retained on the cloth and the clear liquor flows through and emerges from the filter element which is of the leaf type in most of the filters used. The cake is then washed free of sugar in place with hot water and sluiced off the plates by suitable means, or it is sluiced off after partial washing, the cake then being mixed with water and refiltered. The latter practice is safer as it reduces the possibility of sugar losses.

Sweetland Stationary Leaf Filter. This press, originally called the clam-shell type of press, took its name from its two parts opening and closing after the manner of the clam-shell steam-shovel bucket. The body of the filter (Fig. 95) comprises two semi-cylindrical members hinged together, with suitable gaskets, to form a watertight cylinder when closed. The filter leaves are composed of crimped-wire screens, each provided with an outlet nipple for the filtrate. Filter cloth is fastened over these screens which are so arranged that they may be clamped tightly in place in the filter body with the outlet nipples in connection with the delivery fittings outside the filter. Variable spacing is provided for the leaves to suit different filtration conditions, 1½- or 2-in. centers being used in refineries. Each leaf has separate delivery, shutoff cock, glass delivery tube, etc. The opening and closing of the press are easily and quickly accomplished, the largest presses using hydraulic power.

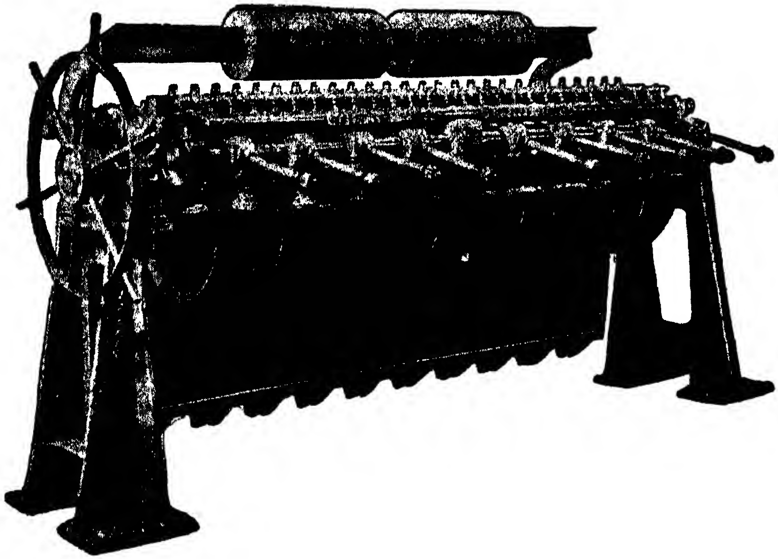

Fig. 95. Sweetland Stationary Leaf Press.

The sluicing device consists of a manifold pipe passing through the entire length of the filter inside the shell just above the leaves (at the back of the figure). This pipe extends out through stuffing boxes at both ends of the filter, nozzles being placed at equal intervals along the pipe so that a nozzle comes directly behind each leaf. The stuffing boxes allow a double movement of the pipe: first, it may be rotated through an arc of approximately

110°, by suitable mechanism attached to one of the projecting ends; second, it may be moved longitudinally through the stuffing boxes during this rotation.

Vallez Rotary Filter. This was the earliest of the rotary-leaf filters to be used extensively As shown in Fig. 96 it consists of a series of disk-shaped filter leaves mounted on a hollow shaft enclosed in a cylindrical cast-iron vessel or shell. These leaves are hollow and are covered with filter cloth. The material to be filtered is pumped into the filter shell under pressure. The leaves and shaft revolve slowly as the liquid is being filtered through them. Each leaf filters as a unit, discharging the filtrate into the shaft, which serves as a common header for carrying the filtrate out of the shell. The Vallez is generally used in sugar refineries with woven-wire filter cloth of Monel metal or bronze. This does away with the frequent changing of cotton cloths, to accomplish which in this filter necessitates the entire removal of the shaft and all filtering disks.

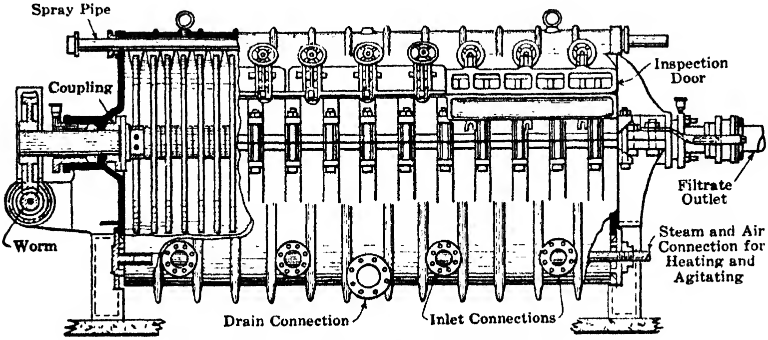

FIG. 96. Vallez Rotary Filter.

When the filtering cycle is complete the cake is sweetened off either partly or entirely, and then the remaining water in the filter is agitated violently with air which loosens the filter mat on the leaves. A final spraying with water completes the cleaning after the cake and water have been discharged from the bottom of the filter.

The rotating leaf type of filter offers several advantages over the fixed type. The cake is more evenly deposited on the leaf; the filter aid is kept in better suspension; breaks in the cake are less frequent and washing off in place may be more efficiently accomplished. Finally, the removal of the cake from the leaves is much more readily done by a spray since all parts of the leaf are brought into closer contact with the sluicing jets.

Plate-and-Frame Presses. It has been found that pressure filtration with filter aids such as kieselguhr can be carried out in plate-and-frame presses of the type so generally used in the raw house (Sec. 142) and refinery for filtration of muds. The operation is as efficient, so far as filtration is concerned, as with the leaf type of filter, but there is the labor of opening the presses, cutting down the cake, etc., which the closed presses do not require. Both washed sugar liquor and defecated washings have been successfully filtered with plate-and-frame equipment.

374. Williamson Clarification System. This is a process patented by George B. Williamson, of Louisiana, for the clarification of defecated raw liquors without filtration. The system is continuous and makes use of the phosphoric acid and lime defecation that is described in Sec. 366. Essentially, it consists of impregnating the defecated liquor with air and then heating to 210° F. in flat vessels so that the air bubbles will rise and carry with them the precipitate of tricalcium phosphate and occluded material, forming a scum which is drawn off by suitable means.

This method of flotation of the floc by means of air bubbles is the basis of the Williamson process so that the form of clarifier used, the method of removing the mud blanket, etc., are merely modifications of the original idea. Defecation is carried out as described on p. 295. The temperature at which the raw liquor comes from the melter (about 160° F.) is suitable, although lower temperatures are advocated by some recent users of the Williamson system.

The method of impregnating the liquor with air varies considerably in different installations. The original aerators were small tanks containing jet nozzles through which compressed air is blown into the treated liquor as it flows from the blowups. A second system which has proved successful is to pump the treated liquor at about 120 lb. pressure through small nozzles (3/8 in.) fitted like an ordinary ejector so that the issuing stream of liquor aspirates air bubbles from the atmosphere. The ejector type of aerator can not be slowed down without affecting its efficiency. The method of varying the amount of liquor aerated is to have a number of ejectors which may be turned on or off to suit the requirements. A home-made type of aerator is shown in Fig. 97, although purchased types have also been found effective. A third method of aeration has been to admit air to the suction side of the centrifugal pump handling the liquor, and still another method is to let the liquor fall on a rapidly rotating disk. Properly aerated liquor will be so full of fine air bubbles as to have a creamy appearance.

The original form of Williamson clarifier consists of a flat tank 6 ft. by 12 ft. by 2 ft. deep containing transverse copper steam pipes at the bottom with 100 sq. ft. heating surface for each clarifier. The liquor entering the clarifier at one end flows slowly through it, the temperature rising until it is 210° F. when the liquor reaches the outlet end. By this time the air bubbles have carried the flocs of precipitate to the surface, forming a tough blanket of scum. This blanket is drawn off by means of a slowly turning roller which pulls the scum over a lip and into the mud trough. The clear defecated liquor is drawn off through pipes from the body of the clarifier about 6 in. below the surface. Each clarifier has a capacity of from 800 to 1000 gal. of liquor per hour, a million-pound melt requiring eight clarifiers for washed sugar.

The clarified liquor contains some flocs of bagacillo and other light material which are removed by passing the liquor through a woven wire metallic cloth screen 120 by 120 mesh. The solids drawn off as scums amount to 4 to 6 per cent of the solids entering the clarifier, but by far the greater proportion of these solids are in the form of sugar which is recovered in the sweet waters as described below. Solids discarded as press cake amount to 0.7 to 0.8 per cent of the melt.

An early modification was to have the clarifier deeper at the entrance end than at the outlet, with the steam pipes lengthwise of the clarifier. Several installations have steam-jacketed bottoms instead of steam pipes.[17] A modification by Jacobs[18] has the clarifier divided lengthwise into six U-shaped compartments which are 30 in. deep at the entrance end and 10 in. deep at the exit end, the steam being in a bottom jacket surrounding the compartments. In this Jacobs modification there is a slow-moving drag the entire length of the clarifier to remove the mud from the back (inlet) end. This drag provides a more positive removal of the mud blanket than does the roller at the front of the older type.

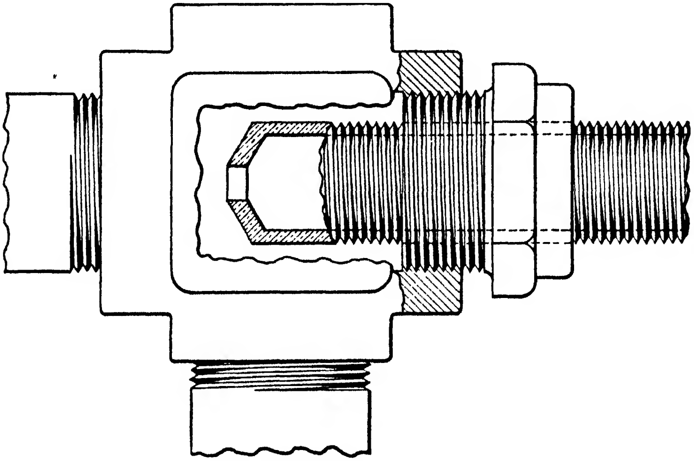

Fig. 97. Aerator Ejector.

A modification of the clarifier devised by Edson for use with the Sucro-Blanc process (see Sec. 404) has the heating surface in the form of tubes running lengthwise of the clarifier and also employs the scum drag used by Jacobs, but it operates to bring the muds to the outlet end. It is obvious that this type might also be used for any refining process.

375. Advantages of the Williamson Process. The primary advantage of the Williamson system is that it permits the use of the phosphoric acid

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[17] See Brown and Bemis, *Ind. Eng. Chem.*, April, 1942, p. 420.

[18] Greven, *Ind. Eng. Chem.*, May, 1942, p. 633.

defecation with the consequent removal of objectionable colloidal and coloring matter. This feature of the phosphate treatment has been discussed in detail on p. 297. There is no other equipment at present except the obsolete bag filter which can be used in conjunction with phosphoric acid and lime although the use of a small amount of P_2O_5 with high porosity filter aids is finding some favor (see p. 298). The Williamson apparatus is simple and requires little expense in upkeep and operation. The cost of defecation is considerably lower than with any other form of treatment. Brown and Bemis[19] say the defecation cost is one-half that of pressure filtration; Greven[20] gives exact figures showing about this same relationship, and these figures corroborate the writer's experience. The recent increased interest in the Williamson method is evidence that these advantages have merit.

376. Disadvantages of the Williamson Process. The system has some disadvantages, but it is of interest that many of the so-called defects are argued from the theoretical viewpoint by those who have not had extensive experience with the method. The objection is raised that heating heavy sugar liquors to 205°–210° F. offers great possibility of inversion or destruction of sucrose. Accurate routine analyses covering many years of actual practice show that with careful control of pH and of temperature the losses from the melter through to the clarified washed sugar liquor are very small—less than 0.05 per cent on solids. Possibly a more accurate control is required than in the pressure filtration methods, but good results are readily obtainable. The automatic control of temperature is employed in some clarifier installations, but the movement of the liquor is so slow and changes so slight that hand control can be satisfactorily practiced.

Another objection is that the clarification is not so positive as filtration and that the effluent liquor will be variable in character. This is true to an extent, but it is not of great importance in practice if the test of the washed sugar is kept close to 99° purity. The process is unquestionably sensitive to raws of poor character, possibly more so than a filtration system. The Williamsons do not always give brilliant clarified liquor, but the turbidity is due to particles of such a size that the char readily removes them, and the suspended matter so removed does not clog the char unless the amount of suspended matter is excessive.

Sand filtration of the Williamson defecated liquor has recently been worked out according to J. M. D. Brown[21] and successfully eliminates this valid objection to the system. The sand-filtered liquor is brilliant, the flow on the char is increased, and the expense is very small. It seems certain that this improvement will become a part of all Williamson systems if it proves as practicable as the first tests indicate.

Another objection is that the Williamsons are not adapted to work on low-purity liquors. With raw washings they will not serve at all because of the

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[19] *Loc. cit.*

[20] *Loc. cit.*

[21] Personal communication.

bulk of the precipitate set up by the defecation, and even with remelt liquors of 94°–96° purity they do not give very good clarification. The use of the two systems, i.e., Williamson clarifiers for washed sugar liquor and pressure filtration for affination and other low test sirups, is no complication in practice.

377. Handling of Mud Waters. *Pressure Filtration.* The cake of the press-filtered washed sugar liquor is generally reused in the defecation of affination sirups. One method is to dewater the sluicings from the presses in Oliver vacuum filters (of the general type of the Oliver-Campbell cachaza filter, Sec. 145), and then puddle the cake to a cream to be added to the raw washings blowups. The final mud waters sluiced from the filtration of these raw washings are then heated, diluted, and filtered in plate-and-frame presses or Oliver vacuum filters. The dark press waters from this filtration go to the multiple effect for evaporation.

Where the affination sirups are boiled away to remelt instead of being defecated and char-filtered the sluicings from the washed sugar liquor filters are treated in the same way as described below for Williamson mud waters.

Williamson Muds. The mud blanket from the Williamson system consists of precipitated tricalcium phosphate, occluded impurities, and some liquor. Some users of the system redefecate the muds in another clarifier, but the general practice is to dilute the muds with hot water, lime to 8.0–8.4 pH, heat in mud blowups, settle, and decant. The settlings are again diluted and then filtered through plate-and-frame presses. The sweet waters from the Williamsons are of high purity—96° or above—and are used for melting washed sugar.

378. Control of the Defecation and Filtering Stations. The amount of defecant or filter aid varies with the character of the raw sugar melted and with the test of the liquor to be treated. The determining factors in the amount to be used are the speed of filtration and the clarity of the filtered liquor. Experience and trial and error are the only guides in this matter. In general the lower the test of the melt the more defecant or filter aid required, but it has been shown (Chapter 15) that filterability is not necessarily a function of the test of the raw, some raws giving less than half the speed of flow that other raws of the same test give.

The effective amount of P_2O_5 used for phosphate defecation does not vary widely with good and bad sugars, i.e., increasing the chemical defecant beyond relatively narrow limits will not improve the results with bad sugars. The amount of filter aid can be increased to a much greater extent to give greater rates of flow with pressure filtration, although here there is an economic limit.

The adjustment of the reaction of the liquors at the blowups is now done by hydrogen-ion (pH) control. The usual pH range for filtered washed sugar liquors is 7.0–7.3 pH but some refineries prefer higher alkalinities than this (7.5–8.0 pH). As will be shown later the changes in pH of the liquor during the char filtration determine what initial pH is chosen for the on-going liquor.

Since blowups are treated by the batch method, manual control of the reaction is quite satisfactory although the automatic addition of lime (Sec. 99) could be employed at this station. Holven[22] reported on automatic pH recorders for refinery alkalinity control and found these practicable but stated that the colorimetric control was sufficiently accurate to maintain close regulation of liming.

A method which has proved successful in the adjustment of the reaction of each blowup is outlined on p. 462. Ordinary unskilled workmen have no difficulty in maintaining the pH within limits of 0.2 to 0.3. It should be noted that with the phosphoric acid-lime defecation if the pH is determined on defecated liquor containing the phosphate precipitate it will show from 0.3 to 0.5 pH higher than after filtration or clarification. In adjusting the reaction at the blowups allowance must be made for this drop in pH because of the removal of the phosphate precipitate. That this drop is due to the presence of the tricalcium phosphate and not to any decomposition during the filtration or clarification can be proved in the laboratory by determining the pH of the defecated liquor before and after removal of the precipitate by filtration through carefully washed paper.

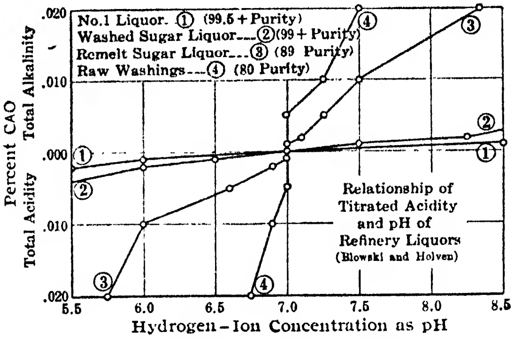

FIG. 98. Titrated Acidity and pH of Refinery Liquors.

Blowski and Holven[23] in a study of the use of pH in the refinery showed that test papers and titrations are no reliable guide to the reactions of refinery liquors. To prove this point they made up several different refinery materials to definite total acidities and alkalinities by adding known quantities of *N*/28 H_2SO_4 and *N*/28 NaOH to the neutral solutions. They determined the pH of these solutions at these known total acidities and alkalinities; some of their results being plotted in Fig. 98. It will be seen that an acidity of 0.003 in washed sugar liquor (raw liquor) gives a pH of 5.5, at which point active inversion would take place, while raw sugar washings at double that acidity would still be 7.0 pH (or effective neutrality so far as inversion is concerned).

Blowski and Holven worked out a method of pH determination using the spot-test method against colored celluloid standards which is simple and effec-

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[22] *Ind. Eng. Chem.*, **21**, 965–970 (1939).

[23] *Ind. Eng. Chem.*, **17**, No. 12 (1925).

tive. (See p. 463.) They also developed a system of regulating the reaction by the addition of lime at various points in the house instead of at the blow-ups only, which they report uses less lime and maintains the pH of all products at close at 7.0.

This study was made in the earlier days of pH control and demonstrates the well-understood principle of buffer action in impure solutions.

379. Perone. Within the past few years it has been demonstrated that a small amount of high-test hydrogen peroxide added to defecated or press-filtered washed sugar liquor acts as an aid to decolorization by bone char. The peroxide is sold under the name Perone and is added directly to the washed sugar liquor before it goes to the char filters, together with a small amount of lime to offset the acidity of the Perone. The results obtained vary widely with the character of the raws melted, but with dark-colored raws 0.03 per cent of Perone on solids will give char-filtered liquors from 20 to 35 per cent lighter in color than liquors without Perone. Continued use is also reported to improve the effectiveness of the boneblack itself. The advantage of Perone is that it can be used when dark-colored raws are being melted and discontinued with good raws. No capital investment or apparatus is involved.

CHAR FILTRATION

380. Purpose and Importance. The liquors which have been clarified by defecation and cloth filtration, or by the Williamson system, contain coloring matter and impurities which must be removed before refined sugar can be produced economically. The material used as a decolorant is animal charcoal or boneblack, usually called black or char (in some few refineries, bone coal) which is the granular residue obtained by the destructive distillation of bones. Boneblack was first employed in sugar refining about 1811, when it was introduced into the liquors in powdered form and then strained out. Its development as a granular filtering medium dates from 1828. Although primarily used as a decolorizer it has the property of removing organic and inorganic impurities, and this property is of the utmost value, since the removal of these impurities reduces the amount of residual sirup (barrel sirup) or waste molasses, and increases the yield of refined sugar accordingly.

The economy and efficiency of the refining process depend upon careful attention to detail in the char house; exact regulation of the densities, temperatures and reactions of onflowing materials; constant attention to the color, speed of flow and brilliancy of the effluent liquors; control of the revivification and the physical condition of the boneblack itself, and finally maintenance of a definite time schedule or cycle for the various steps of the filtering operation. The elaborate control is further complicated in large refineries by the fact that two or three grades of boneblack are maintained, each having its own equipment of filters, kilns, and handling apparatus. Many of the liquors are double and triple filtered, which adds to the complications of char house procedure and control.

381. Char Filters. The filters are cast-iron vertical cylindrical cisterns, conical at the top and bottom, and usually in American practice about 10 ft. in diameter and 20 ft. deep (1200 cu. ft. capacity). The top is closed by a movable door termed the filter head. Two manholes at the sides near the bottom serve for the removal of the exhausted black. The char rests on a perforated plate covered with a coarse-weave cotton blanket, and this in turn with a blanket of finer weave, to prevent the char dust being carried out with the filtered liquor. The inlet pipe is at the side of the filter, close to the top, and the outlet is at the bottom, below the perforated plate. The outlet pipe is carried up in a gooseneck on the outside of the filter to within a few feet of the top. Figure 99 shows the general arrangement of a char filter and its accessory equipment.

382. Char Capacity. Boneblack Ratio. The ratio of boneblack used to sugar melted varies widely in different refineries. The amount used depends on the available equipment, the quality of raws melted and the grades of refined turned out.

The char capacity is usually given as the pounds of boneblack revivified per hundred pounds raw melted, and in the old days when low-test raws were melted and large percentages of soft sugar were produced the average refinery revivified (or "burned") 100 lb. of char per 100 lb. of raw melted. Today, refineries operate on a much lower boneblack ratio because of the high-grade raws and the reduction in the demand for soft sugars. A ratio of 40 to 60 lb. for refineries filtering all affination sirups is now maintained, whereas those refineries which filter less low-grade material operate with a ratio of 30 to 35 lb. or even less.

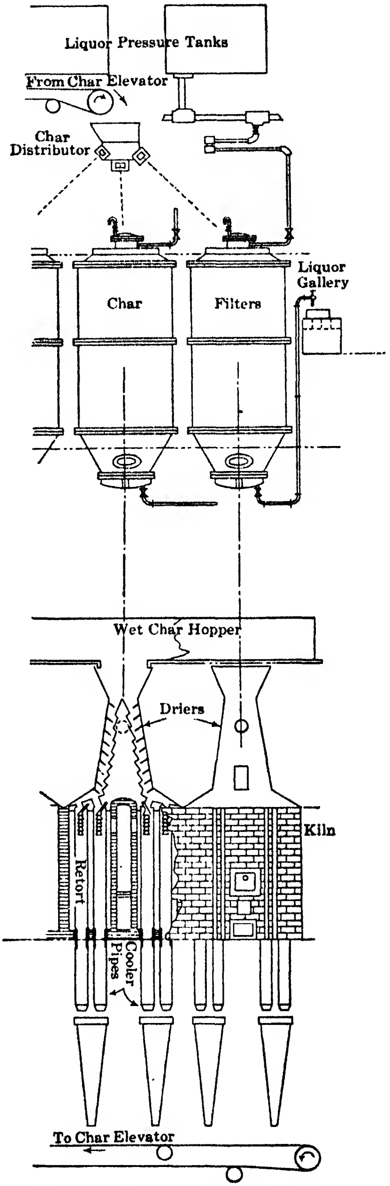

FIG. 99. Char Filters, Driers, and Kilns.

The boneblack ratio is not a true measure of char capacity; first, because it should be on a volume basis and not on weight, and secondly, because it does not take into account the length of cycle (see Sec. 383 below). The volume is the true measure of the amount of black revivified since the weight per cubic foot varies with different service chars, and the heavier the black the less effective it is. The longer the cycle the more filters will be in use at any given time so, of two refineries burning the same amount of black per day and melting the same amount of sugar, the one with the longer cycle will have the greater effective char capacity.

383. Filter Cycle. Schedule of Operations. Every filter goes through the successive steps of the cycle according to a time schedule, which is based on the length of time that it takes to fill a single filter with char (which is in turn dependent on the rate that the kilns are delivering revivified black) together with the number of filters operating in the cycle. Since each filter must be emptied in time to take its filling of burned black, it is evident that the total time of all the operations of the filter cannot exceed the time of filling one filter multiplied by the number of filters.

It is customary in most char houses to separate the boneblack into two or three grades, designated by letters or numbers. Let us assume that a filter house has thirty-six filters, eighteen of which are used for the A grade boneblack and eighteen for the B black. Considering only the first of these, the A filters, and assuming that the number of kilns allotted to that grade of black is sufficient to revivify a filter-full of black every four hours, the cycle of the A filters would quite evidently be seventy-two hours. That is, each of these filters must go through these successive operations in that time: (1) filling with char, (2) covering the char with liquor (settling the filter), (3) running liquor, or the filtration proper, (4) washing the sugar out of the char with hot water, (5) blowing out the residual water with air, (6) discharging the wet char, or dropping the filter. Filter schedules vary through wide limits, fifty-four to sixty hours being the shortest and one hundred to one hundred and twenty hours the longest. All these operations except (3) running liquor, are more or less fixed, no matter what the length of the cycle, so the variable is generally the time allotted to running liquor, i.e., the filtration proper. It is evident that the larger cycle will give relatively much more time for filtering or refiltering liquors.

Two systems of working char filters formerly prevailed: the set, or battery, and the continuous systems, but the set system has now been largely discontinued. In the set system, all the filters filled in one day were worked as a group unit, all the filters in the group doing the same class of work at the same time. In the continuous system, each filter goes through its cycle independently, a step ahead of the filter filled immediately after it. The set system was used by refineries making a large percentage of soft sugars because it lent itself more easily to their pan boiling system and also simplified the use of double and triple filtration. The continuous system requires a much smaller char-filter installation for a given melt, so with the reduction in soft sugar demand it has superseded the set system. The handling of any one filter in the two systems is essentially the same.

Tandem System. One large eastern refinery employs a filtration system in which two filters are operated in tandem, only one grade of liquor being filtered in a given group of filters. The liquor is pumped at about 30 lb. pressure through a primary filter and thence through a riser to a secondary filter. After a number of hours running the primary filter is disconnected and washed off in the usual way while the secondary filter becomes the primary for another filter as secondary. The off-color liquor from the filter that is washing off is run to the pressure tank and refiltered in another tandem. In this system the liquors going on the char are divided into three grades: (1) high test, (2) 93° purity, and (3) below 93° purity. The system insures double filtration for all liquors and a more effective exhaustion of the black.

384. Filling the Filter with Char. The distribution of the boneblack in the filter is of primary importance. If the dust and larger grains of char are not evenly distributed throughout the filter, the liquor will flow through the coarser particles, forming channels and causing many difficulties during the washing-off period. Even distribution may be obtained in many ways and there are several devices designed to secure this result. The char may be delivered into the filter by means of a funnel with a slightly bent stem. The funnel is turned at intervals by hand or continuously by a motor. The oldest effective method of filling the filter with dry char is to deliver it into the filter promiscuously and have a man enter the cistern from time to time and distribute the material with a shovel. After the filter has received sufficient char, this is drawn up into a cone in the middle.

H. I. Knowles[24] in a study of boneblack filtration showed that even with hand leveling ten or twelve times during the filling of a filter the distribution of char leaves much to be desired. The sides of the filter contained char of much coarser grist than the center, and samples taken later during the filtration period by special sample pipes inserted in the filter showed higher color, ash, and organic removal in the center of the filter than at the sides. This was in large part due to more rapid flow and less contact through the coarse char of the sides than in the fine char in the center. Wet filling, i.e., adding the char and liquor to the filter together has generally been adopted to avoid this separation of coarse and fines and will be described later.

385. Covering or Settling the Boneblack with Liquor. Air pockets in the char must be avoided. To prevent these, with dry filling as described above, the liquor is delivered slowly at the base of the cone of char, with the head of the filter off. The liquor runs down the sides of the filter until it reaches the blanket, then flows across the whole width of the filter at the bottom and rises, forcing the air out through the cone of char, which is kept dry throughout the covering. When the liquor begins to flow from the gooseneck, the outlet valve is closed and liquor is run in until the filter is filled.

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[24] *Ind. Eng. Chem.*, **17**, No. 11 (1925).

In spite of all precautions there will always be some entrapped air, and this is permitted to escape by allowing the filter to "boil" as filter men call it. After there is no further evidence of escaping air, the head is put on the filter and the liquor is turned into it under pressure (15–25 ft. head). The covering usually requires about four hours.

Considerable heat is generated when hot liquor is first run onto well-burned char. With the char at a temperature of 140° to 150° and the liquor at 165°, the outflow during the first few hours of running will normally have a temperature of 185° to 190°. If covering is stopped under these conditions, or if filtration is suspended during the early stages, the temperature in the filter may rise to such a point that the liquor will be scorched. The char should be cooled to at most 160° before it enters the filters, and every precaution should be observed to avoid interruptions to the flow of liquor during the covering period and until the temperature of the outflowing liquor is practically that of the liquor entering the filter.

Knowles in the study already referred to showed that the temperatures of the liquor at the side of the filter are much lower than in the center where he found liquor rising to 210° F. during the first twelve hours of running.

Covering filters by bottom-filling has been advocated but has never found much favor in American refineries. In this practice liquor (generally once-filtered washed sugar liquor) is run into the bottom of the filter through the outlet pipe. As the liquor rises in the black the voids are completely filled and there is less opportunity for entrapped air or air pockets to form. The practice might possibly have some advantages over top-filling and covering with dry char but is not so advantageous as the wet-filling method described in the following paragraph since there still remains the "channelling" of liquor due to poor distribution of char.

386. Filling and Covering Together. Wet Filling. The process of intimately mixing the char and the liquor and adding them together to the filter has been tried out from time to time through many years but a workable procedure was not developed until 1925. The method as now used was worked out in the Californian and Hawaiian Refinery and consists of first adding dry char until the filter is filled one-fifth to one-fourth of its volume and then running in the char and liquor together through a set of three eccentric funnels placed one above the other and hung in the mouth of the filter. The eccentric arrangement gives the char and liquor a swirling motion which mixes the two, the flow of char and liquor being so adjusted that the mixture falls from the bottom funnel at about the consistency of a loosely mixed concrete. With a little care this mixture can be so maintained that there is no tendency for the fines and coarse to separate. If the mixture is too free, i.e., too great a proportion of liquor to char, this classification of coarse and fine will occur, whereas with too dense a mixture dry pockets of char will occur in the filter and give trouble later both in the filtration and washing periods. It has been found advantageous in one refinery to measure the onflowing liquor by means of a measuring box similar to that used at the liquor gallery for measuring outflowing liquors (see Fig. 100). The flow of char is very regular and by adjusting the liquor flow by measurement all difficulties are avoided and close attention is not required.

In practice this wet-filling method has reduced "channelling" of liquors to a minimum; has cut down the time of filters in sweet water about 25 per cent and has practically eliminated bad washing after sweet water. About three hours' time is also added to the time of filtration by wet-filling which is otherwise taken up in separate filling and covering. It is probably the greatest improvement in char-filter technic of the past twenty-five years.

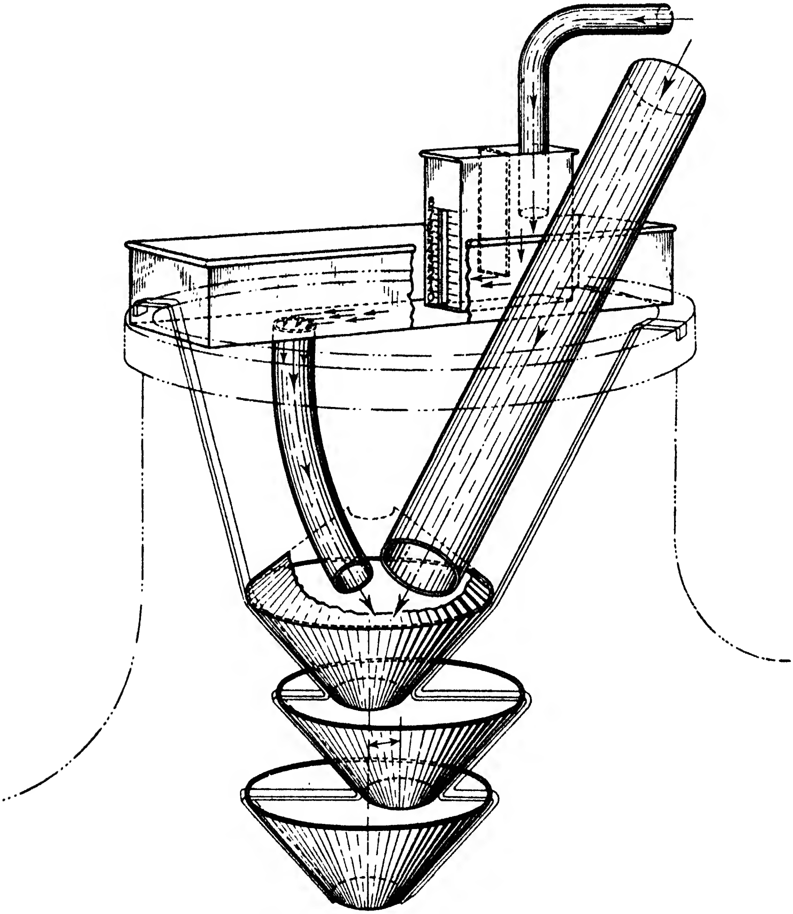

Fig. 100. Funnels and Assembly for Wet Filling.

An objection to the wet system was the prevalence of "blue" liquor or "smoky" liquor in the first runnings from filters so covered, this being much more evident with some kinds of raws than with others. It was easy to prove that this blue coloration was due to colloidal fixation of fine char dust in the liquor, but no effective method of prevention was devised until recently when it was found that filters covered with early runnings of a previous filter, i.e., nearly water-white liquor, will never give cloudy first runnings.

Some refiners find that wetting the portion of dry black first put into the filter will correct the hazy liquor difficulty, but others prefer to wet-fill by first putting liquor into the char cistern and then adding the boneblack to the liquor. J. M. D. Brown in 1941 questioned all refiners in the United States and Canada and found much divergence of opinion as to filling practice and results. The advantages of wet filling as given above are from the writer's personal experience, but they are in accord with the majority opinion brought out by Brown's questionnaire.

That wet-filled filters do not channel and that dry-filled filters do is easily shown by running two parallel filters covered by the two systems. The color of the filtered liquor from the dry-filled filter will break, that is, turn yellow, several hours earlier than the wet-filled one.

387. The Filtration. As soon as the covering is complete and the filter head is in place the filtration proper begins. Practice in refineries differs as to how the filtration schedule is conducted, depending on the char capacity (boneblack ratio), the length of the cycle, the character of the melt, the number of grades of char, the amount of refiltration and the quantity of soft sugars to be made, besides many purely local considerations. Certain general principles are followed, however, in all refineries. The highest-purity liquors are filtered first and followed by liquors of lower purities. Washed sugar liquor (99°) goes on the char at 160°–165° F. and the same Brix that it comes from the cloth filter or Williamsons, viz., 60°–65°. The speed of flow is from 200 to 300 cu. ft. per hour. The density of the lower-purity liquors is usually somewhat less than those of highest purity, and with each successive grade that is filtered the speed of flow is reduced and the temperature increased. Each grade of liquor follows the preceding one without a break in the filtration; the swing pipe at the top of the filter inlet is shifted from the delivery line of one liquor pressure tank to the other, after which the filtration proceeds.

Where two or three grades of black are maintained, the principle is followed that the oldest black receives the materials having the largest amount of impurities, but the liquors for refiltration are filtered through the best grade of black. The reason for this is apparent since the impurities removed by the first filtration are in greater quantity but are the easiest to remove, whereas those removed by refiltration are present in less amounts but require a more active boneblack for their removal. Furthermore, overworking the most active black would quickly reduce its efficiency.

Washed sugar liquor forms the bulk of the liquor to be filtered so it is generally run on each filter for twenty-four to forty hours, depending on the cycle. The effluent is water white and above 99° purity and goes to the vacuum pans for boiling to granulated sugar. After many hours running the No. 1 liquor assumes a yellowish tinge, and it is then used for covering other filters, or is refiltered. Following the washed sugar liquor, remelt sugar liquors of 95° to 97° purity may be filtered, then "granulated sirups" (90° purity), which are sirups from granulated sugar strikes too dark for further boiling to granulated. Cloth-filtered raw sugar washings (affination sirup) of about 80° to 85° purity and sirups from remelt sugar strikes are the low-grade materials which are generally sent to the char filters. These and the granulated sirups are double and triple filtered to make light-colored liquors of low test for soft sugar manufacture or for fancy sirup production. The second and third filtrations remove color but do not materially raise the purity of the liquors.

Some refineries of limited char capacity do not filter anything of lower test than granulated sirups of 90° purity. Where such practice is followed the raw sugar washings are sent to the vacuum pans direct without cloth or char filtration and are boiled to remelt sugars.

388. Washing Off. *"Sweetening Off."* At the end of the filtration period the boneblack, so far as regards color removal and adsorption of impurities, is practically exhausted. The inlet valve is now closed and the level of the liquor in the filter is lowered by gravity or air pressure to that of the char. The head of the filter is removed and the char is leveled and then hot water (210° F.) is run upon it until the filter is filled. The head is replaced and hot water is run into the filter under pressure. The rate of flow of the water is about half that used for the liquor. The idea is to have the water displace the liquor remaining in the filter with as little mixing of the two as possible. This is so well accomplished in a well-running filter that there is no appreciable break in the density of the outflowing liquor for five or six hours after water is put on the char. When the dilute liquor has a density of about 40° Brix it is termed char sweet water, and the rate of flow is now further reduced and the filtrate is sent to the multiple-effect evaporator for concentration with other sweet waters. The density and purity of the sweet water fall rapidly. After two to five hours the water no longer contains enough sugar and is of too low purity to warrant its evaporation, and it is turned to waste.

The char sweet waters contain very high percentages of ash on the solids present and they also are high in colloidal material.[25]

The ash and colloids which have been adsorbed during the filtration are leached out of the black in the dilute runnings after water has been put on. It is obviously the best practice to keep the evaporated char sweet waters separate from other materials containing less ash and colloidal impurities. They are not generally returned to the black for refiltration, and the use of char sweet water for melting purposes should never be considered.

Washing to Waste. The washing to waste is continued for at least twelve to fourteen hours and the speed of flow again is increased to about 200 cu. ft. per hour. Washing for a longer period at a slower rate will give more effec-

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[25] Paine and Badollet, *The Planter*, **79**, No. 2 (1927).

tive results and is the practice when time permits. Much of the organic matter is so strongly held by the char that no amount of washing will remove it. At the completion of the washing period the water is driven out of the char by compressed air, 10 lb. pressure for two hours usually being sufficient; the bottom doors of the filter are opened and the char is discharged into the hoppers on top of the driers. (Fig. 99.) Lyle[26] gives an interesting discussion of a method of saving water in both sweetening off and washing to waste. This is done by running the filters which are on water in a modified tandem system.

389. Revivification of the Boneblack. The revivification of the black is accomplished by heating it to a high temperature out of access with the air. Special driers and retort kilns are designed for this purpose. The burning of the black, as it is usually called, distils off some of the organic impurities which the washing failed to remove, and carbonizes the remaining portion. The alteration of boneblack with use will be discussed later.

390. Char Driers. The wet char is passed by gravity through driers preliminary to the revivification. The driers are of various designs and in general consist essentially of a casing through which the waste gases from the kilns are drawn. Deflecting plates conduct the wet char slowly over the surface of this casing. The char enters the driers carrying about 18 to 20 per cent of moisture and leaves them with about 12 to 15 per cent. This type of drier is not very effective.

A much more effective drier has been devised by deVries and is reported to deliver moisture-free char to the retorts. The flue gases from the kiln are by-passed directly to the stack, while hot air is drawn from a casing around the cooler pipes below the retorts and blown into the inner casing of the drier. This heated air is forced through the wet char which is passing down by gravity between louvres as in the ordinary drier. The "wall" of char in the deVries drier is much thinner than in the regular type and the hot air blowing through this thin layer removes the moisture much more rapidly. It was formerly thought that some moisture in char was necessary to revivification, that is, that it was a steam distillation, but this has been disproved.

391. Char Kilns. The kiln consists of a furnace flanked on both sides by double or triple rows of upright pipe retorts, twenty retorts to the row, through which the char slowly passes. The commoner type is the double-row, or eighty-retort, kiln. The older-style retorts are of cast iron, of oval cross section, 3 in. by 9 in. or 3 in. by 12 in., from 8 to 10 ft. long. The combustion chamber occupies about one-quarter the width of the kiln, running through the entire length, and it is generally fired at both ends, the fuel being coal, oil, or natural gas. Fuel consumption with good kiln work is about 30,000 Btu. per cu. ft. of char burned.

The Buchanan kiln, not in general use in refineries in the United States, has a drier-retort-cooler tube combination all revolving as a unit at slow

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[26] *Loc. cit.*

speed—one revolution every thirteen to twenty-four minutes. Each kiln has twenty-two of these revolving drier-retort-cooler tubes, driven from gears at the bottom. Each retort is 12 in. in diameter and fitted with a ventilation pipe extending through its length and connecting with a similar pipe within the drier pipe.[27] The patents are quite old but the Buchanan kiln is still looked upon with favor in English refineries.

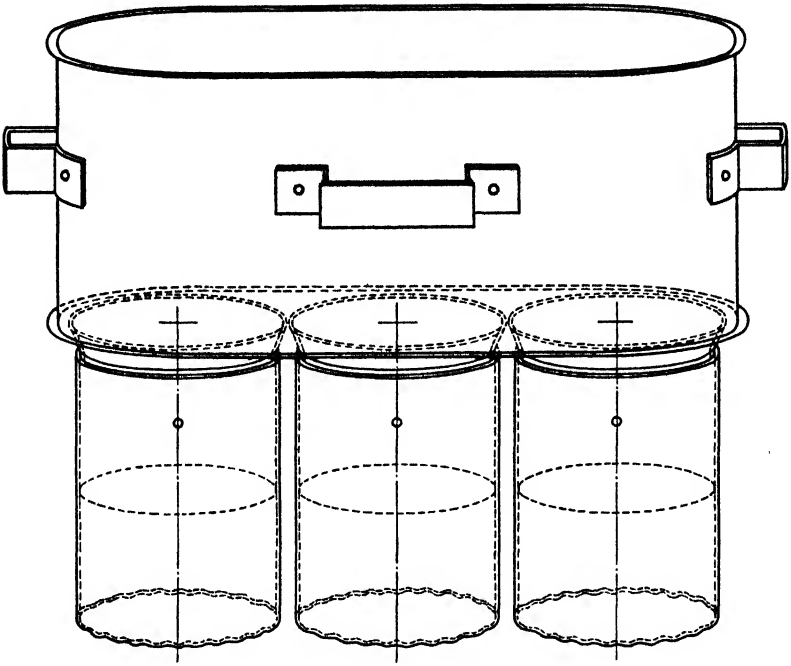

Fig. 101. Three-Tube Thin-Wall Retort.

An improvement in retorts of the past ten years is the triple-tube thin-wall type made of 26-gage heat-resisting steel containing about 18 per cent chromium. The triple tubes fit into a header of similar shape to the old-style cast-iron header, and a suitable fitting is also used at the bottom (Fig. 101). The thin-wall tubes are much lighter in weight, offer a readier heat transfer, present a larger surface to the heat for a smaller volume of char, have a much longer life, are more easily replaced, and in general are so advantageous that the cast-iron retort will soon be entirely eliminated.

The products of combustion leave the chamber near the top, are conducted downward along the length of the retort to ports at the bottom of the kiln,

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[27] See Misener and Fraser, *Proc. Sugar Ind. Eng.*, Aug. 10, 1941.

and thence by ducts in the brickwork up to the casing of the char driers. Recording pyrometers are now general for regulating the temperature, the practice in most refineries being to place the thermocouple between the rows of retorts in the center of the kiln at the hottest spot. Temperatures of 1000° F. or above are usual with the older-style cast-iron retorts, although many refineries with large kiln capacities operate at lower temperatures than this. The thin-wall retorts described above permit of lower kiln temperatures. Peepholes in the brickwork are used to judge the temperature of the retorts in the absence of a pyrometer, a dull red heat being necessary for ordinary revivification. At the base of each retort is fitted a sheet-iron cooler pipe identical in shape with the retort and extending below the kiln some 6 or 8 ft. The cooler pipe takes the hot char at the kiln temperature and cools it to the point where it can be released to the atmosphere without danger of the carbon burning. The speed of the char through the kilns is controlled by means of a cam arrangement actuating a small drawplate or tilt bucket at the end of each cooler pipe.

Each eighty-retort kiln will revivify about one filter (1200 cu. ft.) of char per twenty-four-hour day, or about 15 cu. ft. per retort per day. The char takes four to five hours to travel through the retort. Kilns may be forced to 50 per cent greater capacity than this but only at the expense of the furnace linings and retorts. From the cooler pipes the char falls into hoppers that deliver it to the conveying system (endless belts and bucket elevators) by which it is carried to the distributing system at the filter heads where it is run into an empty filter to go through the cycle again. (See Fig. 99.)

The char should never go to the filters at a temperature above 160° F. (many refiners prefer even lower temperatures) so that it is generally necessary to cool the black further on its way to the filters by passing it over pipes through which cold water is circulated. A simple method of cooling the char is to spray it with water as it passes along the conveyor belt beneath the kilns. All the water evaporates in the passage of the char through the conveyor system and the cooling is very effective and easily regulated.

392. Rotary Kilns and Other Designs. Many patents for rotary kilns have been issued but none has ever been used on a large commercial scale. A kiln of this type recently developed by E. W. Rice [28] has been operated in a small refinery. The Rice kiln is essentially a long revolving drum fired at one end by gas or oil and so arranged that the gases of combustion can be recirculated through the drum to regulate the quantity of excess air. Temperatures of 600° to 700° F. for ten to thirty minutes are reported as giving satisfactory activation. To avoid abrasion in handling the char, blowing through upright tubes without bends is recommended instead of the usual bucket elevators.

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[28] *Ind. Eng. Chem.*, December, 1939, p. 1516.

The Thermofor kiln[29] which is being used in the petroleum industry has been suggested as a possible method of reactivating char. In this kiln molten salt is pumped through the retort tubes while the material to be reactivated (clay in the petroleum industry) passes down through the kiln on the outside of the tubes so heated. The high cost of this kiln would seem to preclude its adoption in sugar refining.

In one small refinery a hearth furnace similar to the type used for reactivating diatomaceous earth (Sec. 368) is used for char reactivation. The appearance of the boneblack from this furnace indicated burning of the carbon but a recently designed furnace has eliminated this objection.

A study of char reactivation by J. M. Brown and W. A. Bemis[30] shows that with suitable experimental equipment it is possible to reactivate char completely in as little as fifteen minutes where the ordinary retort type of kiln takes from two to four hours. It may be that with the type of metal in the thin-wall retort and a better knowledge of the theoretical aspects of reactivation, a kiln will be developed which is much less cumbersome and costly than the present-type retort kilns.

Weinrich Decarbonizer. The reburning of the black carbonizes some of the organic matter which the washing failed to remove. This carbon remains in the pores of the black, and since it has no decolorizing power such as is possessed by the constituent carbon, it merely clogs the pores of the char and decreases its filtering value proportionately. The Weinrich decarbonizer, designed to remove this added carbon, is a revolving drum, slightly inclined to the horizontal, with a carefully regulated fire under it. The inert carbon is burned away as the char passes through the drum, thus increasing the porosity and prolonging the life of the boneblack.

The modern way of decarbonizing boneblack is to admit air in regulated quantities at the top of the cooler pipes while the boneblack is still very hot. This requires no apparatus and may be done in the regular cycle of revivification.

393. Renewal of Boneblack. Mechanical handling causes the grain of the boneblack to be broken down by attrition. The fine dust is removed by means of suction fans which draw air through the char as it falls over umbrella-shaped devices, generally placed at the discharge of the elevators just ahead of the char-distributing system to the filters. Besides this dust removal it is occasionally necessary to screen out the finest char (below 50 mesh) with wire-mesh screens of the mechanical shaking type. The dust may be used in making phosphoric acid paste for the defecation or sold for fertilizer manufacture and other purposes. The char may decrease in bulk somewhat by shrinkage in burning, but this has recently been questioned.

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[29] Simpson, Nicholl, and Payne, *Trans. Am. Inst. Chem. Eng.*, December, 1940, p. 841.

[30] *Ind. Eng. Chem.*, August, 1940, p. 1112.

These losses are made up by the addition of new boneblack, either a filterful at a time or a fixed amount to each filter at the beginning of the washing period. In refineries having two or more grades of black, the new black is naturally added to the highest grade, and the heaviest screening is done on the lowest grade. As new black is added to the first grade, an equal volume is thrown from the first to the second and from the second to the third grades to maintain the proper proportions. As will be explained later, new black must be washed and burned before use. From 15 to 30 per cent of new boneblack is added yearly, depending on char capacity and other factors. The boneblack in a refinery is therefore completely renewed every three to six years.

394. Char Separator. An effective specific gravity char separator for removing inert material from boneblack in service was devised by J. M. Brown and W. A. Bemis of the Revere Refinery, Boston, Mass.[81] The separator consists of a grasshopper conveyor plus an adjustable screened deck through which a powerful current of air passes. The deck surface consists of a perforated plate through which air is blown at the rate of 1700 to 3100 cu. ft. per minute, the rate being determined by the type of char to be cleaned. Deck baffles about 1 in. apart run practically the entire length of the deck. The deck moves about $\frac{3}{16}$ in. longitudinally four hundred times a minute. The sidewise and endwise slopes of the deck are also variable. The bone char is fed onto the deck, and the dust is collected at the receiving end through a dust hood, fan, and cyclone arrangement. The lighter particles of bone char are raised by the current of air high enough to drift over the baffles and the heavy particles, independent of size, move toward the far end and are discharged there as discard. The good bone char is discharged over the side of the apparatus and returns to the system.

Bone chars of varying densities and degrees of activity may be obtained at will by varying the lateral or longitudinal tilt of the table and by varying the air flow through the perforated deck.

As will be shown later the effectiveness of boneblack varies inversely as the weight per cubic foot. Therefore this separator offers a way of removing the useless portion of the black and replacing it with new black. Knowles has figures[82] that *new* boneblack, separated on this gravity separator, gives fractions of widely varying specific gravity and decolorizing power. The heaviest fraction amounting to 14 per cent of the black showed a color adsorption of only 76 per cent as compared with 90 to 92 per cent for the lighter portions.

395. Composition of Boneblack and Its Alteration by Use. Analyses of new boneblack of American manufacture and of the same black after use, are given here.

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[81] *Ind. Eng. Chem.*, September, 1934, p. 918.

[82] Private communication.

	NEW BLACK	HIGH-GRADE SERVICE BLACK	LOW-GRADE SERVICE BLACK
Carbon	8.55	6.16	4.62
Insoluble silica	0.25	0.33	0.50
Calcium sulfate	0.06	0.82	1.01
Calcium sulfide	0.05	0.15	0.26
Calcium carbonate	8.90	4.50	3.75
Iron	0.06	0.20	0.28
Undetermined (calcium phosphate)	82.13	87.84	89.58
	100.00	100.00	100.00
Weight per cubic foot, average loose and packed	45.0 lb	64.0 lb	74.0 lb
Percentage between 12 and 30 mesh sieves	84.8	79.01	72.5

Boneblack always contains some nitrogen, but the role this plays in the filtration has not been determined. It had long been believed to be essential to the decolorizing power and Patterson[33] and later Hall,[34] extracted a nitrogenous body from boneblack by digestion with sulfuric acid, a few drops of which were claimed to have the decolorizing power of several grams of black. When precipitated on inactive wood charcoal this material also was supposed to give a highly active carbon, but these results were explained shortly after by Tanner[35] as being due to the decolorizing action of the sulfuric acid on the caramel used in the tests and not to the nitrogenous body. Hauge and Willaman[36] failed to find any decolorizing power in the nitrogenous body so extracted, and other investigators have presented arguments against the decolorizing power of the nitrogenous constituents of the char which seem to be conclusive. New black contains a considerable amount of ammonia salts, which must be removed, since they would have a detrimental effect upon the color of the filtered liquors. For this reason, new char must be thoroughly washed and burned before use for filtration.

As is shown by the analyses, the composition of char alters with use. The carbon may either increase or decrease, depending on the airtight condition of the kilns; whether air is purposely admitted at the top of the cooler pipes; the temperature of revivification; the amount of organic impurities adsorbed by the char, i.e., the extent of the exhaustion of the boneblack; the extent and effectiveness of the washing process; the thoroughness of revivification; and probably other less evident conditions. In recent years the tendency seems to be for the carbon to decrease in chars burned at temperatures over 1000° F., particularly if only high-test liquors are filtered. This decrease may reach an equilibrium at 4 to 5 per cent of carbon, but some refineries

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[33] *J. Soc. Chem. Ind.*, **28**, 700 (1909).

[34] *The Planter*, **68**, No. 2 (1922).

[35] *Ind. Eng. Chem.*, **14**, p. 441 (1922).

[36] *Ind. Eng. Chem.*, **19**, p. 943 (1927).

have reported carbon content below 2 per cent (without apparent air leaks in the kilns). Lyle[37] reports this same tendency (in English practice) of the carbon to decrease rather than to increase with use. The carbon in an overworked and underburned boneblack which the writer examined some years ago rose to 16 per cent, but this was later reduced to 10 per cent by successive revivifications in the Weinrich decarbonizer (Sec. 392).

The calcium sulfate increases by the removal of sulfates from the liquors and the water. The calcium sulfide tends to increase as the sulfates increase, owing to the reduction of the latter by the organic matter during the burning. The calcium carbonate drops sharply during the first few washings and burnings and finally tends to reach a balance around 4 per cent.

The calcium carbonate may be rapidly depleted in a black by the filtration of acid liquors. It may be replaced somewhat by increasing the lime in the on-going liquors (8.0–8.4 pH), but a more practicable method which is employed in several refineries is to add lime (or more generally ground limestone) to the revivified black as it leaves the kilns. The limestone is more effective, and it is best added in the form of "chicken grit" of about 10-mesh size. Either powdered limestone or hydrated lime is largely removed by the fans as the dry boneblack passes through the dust-collecting system and therefore is lost, but the grit continues in the black until used up by excess acidity of the liquors.

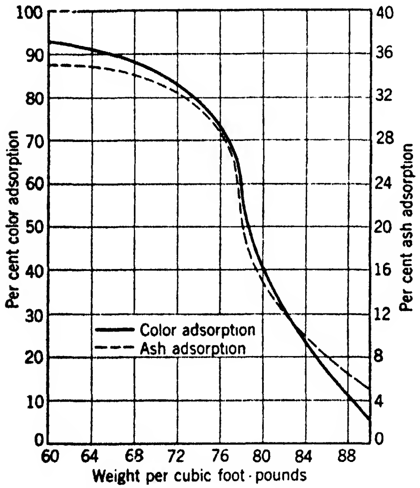

FIG. 102. Color and Ash Adsorption and Weight per Cubic Foot.

The iron increases slowly. Of all these impurities the calcium sulfide and iron are the most objectionable, since appreciable quantities of these two constituents will give a greenish color to the filtered liquors and a gray cast to the sugars boiled from them. A boneblack may become unfit for use if the calcium sulfide content exceeds 0.4 per cent. Thorough washing and burning, together with the adequate addition of new black, will prevent the sulfide content from becoming abnormal.

396. Weight per Cubic Foot. The weight per cubic foot has long been recognized as having a relationship to the activity of the char. Brown and Bemis[38] have worked out the relationship between ash and color adsorption (Fig. 102) which indicates a sharp reduction in these functions in char heavier than 76 lb. per cu. ft.

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[37] *Technology for Sugar Refinery Workers,* London, 1941, p. 265.

[38] *Ind. Eng. Chem.,* September, 1934, p. 920.

Up to recently it was supposed that the char increases in weight per cubic foot because of shrinkage, but A. B. Babcock (before Sugar Industry Engineers, January, 1944) has shown that no such shrinkage occurs. The increase in weight is due to the clogging of the pores of the char by adsorbed foreign matter from the liquors. The grains of the char also wear smooth with use and therefore pack more closely. Since the action of char is due to adsorption, which is a surface phenomenon, the less porosity the less adsorption, hence the relationship between weight per cubic foot and activity.

397. Bauxite as a Substitute for Boneblack. The use of bauxite for decolorizing and purifying sugar solutions is described later (Sec. 405). (This material has largely replaced boneblack for the decolorization of lubricating oils.) A publication of comparative tests[39] shows that with both washed sugar liquor and raw sugar washings (wash sirup) the removal of ash by bauxite is over twice as great as with boneblack, and the invert removal is also much greater for the new material. Color removal is about the same and the color tone of the filtered low-grade liquors from the bauxite is reported good. Plant scale operations have developed difficulties which may or may not be corrected with greater knowledge and experience. The low cost of the bauxite, and the fact that it may be reactivated at relatively low temperatures (800° F.) in the presence of air make it an attractive prospect as a substitute for boneblack. An interesting difference is that bauxite revivified at high temperatures (900° F. or above) gives low pH filtrates, though the opposite is true of the relationship between boneblack revivification temperatures and pH of filtrates. (See p. 325.)

398. Action of Boneblack on Sugar Solutions. Boneblack is primarily a decolorant, but its ability to remove organic and inorganic impurities adds enormously to its value to the refiner. It is now generally agreed that it acts by adsorption, following Freundlich's equation which is, that for any given temperature

$$X/M = KC^{1/n}$$

where X = amount of dissolved substance adsorbed by M grams of char.

C = the concentration of the dissolved substance at equilibrium (i.e., after the adsorptive action has ceased).

K and $1/n$ are constants, depending on the amount and kind of the solution treated with the char.

The term X/M in the equation is really the work done by a unit weight of boneblack or carbon, and the equation states that the work done by a unit weight of carbon (or boneblack) is proportional to some power of the concentration of the dissolved substance at equilibrium. Within the working limits for sugar solutions the higher the temperature the greater the adsorption. The upper limit for sugar work is about 180° to 190° above which point inversion and decomposition become possible factors.

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[39] W. A. LaLande, Jr., *Ind. Eng. Chem.*, January, 1941, p. 108.

Color Adsorption. M. T. Sanders[40] and F. W. Zerban[41] showed that the adsorption formula as given above is valid for color removal by all carbons including boneblack when the color units of Meade and Harris[42] are substituted for the concentrations X and C. (Other color units based on the extinction coefficient (Sec. 594) may also be used.) It should be noted that adsorption isotherms when plotted for X/M and C on logarithmic, cross section paper are straight lines.

The claim that the decolorizing power of boneblack rests in the nitrogen content has been largely refuted (see p. 321) and the carbon deposited on the bony structure of the black at the time of manufacture is now recognized as the color-adsorbing medium. Refiners have long known that the adventitious carbon deposited in the pores during revivification not only has no decolorizing value but also is actually a detriment in that it reduces the porosity of the black and in consequence its activity. H. I. Knowles[43] suggested that there is an optimum carbon content giving the highest decolorization and in experiments in which he decarbonized and recarbonized chars in the laboratory found the optimum decolorizing value at a carbon content of about 6 per cent. Some boneblacks which were in refinery service were studied by him during twenty months and found to be at their optimum decolorizing value when the carbon had reached 7 per cent. It seems probable that this figure would vary considerably for different chars, and that other conditions might have a greater effect on the decolorizing power, e.g., the alkalinity of the black and the extent of the revivification. It has already been mentioned (p. 322) that refinery service blacks with carbon contents as low as 2 per cent have appeared to give satisfactory results.

The pH of the solution going on the black is of great importance as was shown by Hauge and Willaman[44] in a study which ascribed to the disregard of this feature most of the discrepancies and contradictions in the work of different investigators on decolorization by vegetable carbons. They showed that caramel is readily adsorbed by boneblack only at low pH values, i.e., much higher acidities than are encountered in practical sugar work. Experiments such as these on pure caramel have probably been the source of the long-expressed belief that the reddish and brown coloring matters in raw sugars are hardest to remove by boneblack. Wayne[45] in a careful investigation of decolorizing efficiency by spectro-photometric analysis showed that no such generalization concerning caramel types of coloring matter can be drawn. The colors due to the polyphenols of iron (grayish-green and greenish-brown tints in washed sugar liquors) are more easily removed so far as percentage of color removal is concerned than the red coloring matters, so long as complete

[40] *Ind. Eng. Chem.*, **15**, 784 (1923).
[41] Unpublished, presented before Am. Chem. Soc., 1921.
[42] *Ind. Eng. Chem.*, **12**, 687 (1920).
[43] *Ind. Chem. Eng.*, **19**, No. 2 (1927).
[44] *Ind. Chem. Eng.*, **19**, No. 8 (1927).
[45] *Ind. Chem. Eng.*, **18**, No. 8 (1926).

or nearly complete decolorization is not attempted. When the decolorization is carried to the extent that the refiner must carry it to give him liquors suitable for refined sugars, a residual color is encountered in the gray-green sugars that is highly resistant to char adsorption, and the resulting sugars have a grayish cast, whereas the red coloring matter from incipient caramelization may be completely removed, yielding white liquors free of a gray cast.

With intensive char filtration 80 to 90 per cent of all the color entering the refinery may be removed. The high-grade liquors (washed sugar liquor 99° purity) are decolorized practically 100 per cent.

Ash Adsorption. The removal of inorganic matter is due in general to the bony structure of the char, as may be proved by burning off the carbon, when the residue will give as great, or sometimes greater, ash removal than before ignition. The selective action of boneblack for various kinds of salts was brought out a great many years ago. The phosphates, sulfates, and carbonates are readily adsorbed, but the chlorides and nitrates are adsorbed to a much lesser extent. Knowles [46] corroborated these facts as did Rice and Murray,[47] and they showed as well that the nature of the base was of equal importance. Iron and calcium are more readily adsorbed than sodium and potassium. Similar selective action is evidenced for the organic salts. A considerable portion of the ash that is adsorbed by the boneblack during filtration is released to the thinner runnings and sweet waters after water is put on. This leaching out of the ash makes the char sweet waters very high in ash-sucrose ratio and their return to the char filters after concentration should be avoided.

An important investigation on the action of boneblack by T. B. Wayne [48] deals largely with ash adsorption. His findings may be summed up as follows: (1) The percentage of ash adsorbed by a char increases regularly with increasing alkalinity of the char itself up to a pH of 9.6 as measured on a distilled-water extract of the char. (2) The pH of the boneblack is directly affected by the temperature of revivification, e.g., a char burned to 900° F. (480° C.) (as measured by a pyrometer in the kiln) gave a water extract pH of 7.0, the same char burned to 1000° F. (538° C.) gave 8.4 pH, and when this char was burned to 1150° F. (620° C.) the pH was 9.3. (3) From conclusions (1) and (2) it follows that ash adsorption is increased by increasing the temperature of the revivification. (4) The pH of the filtered liquor is increased as the pH of the char increases. (5) Varying the proportion of char to liquor does not alter the pH of the filtrate. This in conjunction with (4) shows that the alkalinity of the black is held in an adsorbed state and is liberated to the liquor in exchange for some of the constituents of the liquor until an adsorption equilibrium is reached. (7) The caustic soda test (Sec. 429) may be colorless, i.e., excellent revivification may be indicated, but if the pH of the black is not high enough due to too low a temperature of revivifica-

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[46] *Loc. cit.*, **19**, 222 (1927).

[47] *Ind. Eng. Chem.*, **19**, 214 (1927).

[48] *Ind. Eng. Chem.*, **20**, No. 9 (1928).

tion, acid liquors may result, even though both the black and the on-flowing liquors may be above 7.0 pH.

This last finding is demonstrated by Wayne by filtering neutral calcium acetate solution (pH 7.3) through a char burned at various temperatures. The results are shown as follows:

pH of Char Extract	pH of Calcium Acetate Solution On	pH of Calcium Acetate Solution Off
6.9	7.3	6.0
8.2	7.3	7.0
9.6	7.3	8.4

Some refiners have long recognized that poorly burned boneblack in refinery service yields acid liquors and increases inversion losses. This was ascribed to various causes, but the above findings show it to be due to a form of hydrolytic adsorption, i.e., a breaking up of neutral salts into acids or acid salts within the black.

Wayne's conclusions in this investigation bear out many points which the writer has observed in actual char-filter practice but which have lacked scientific corroboration previously. Principal among these are the need of relatively high temperatures and exact control in revivification, the fallaciousness of the idea of overburning at practical kiln temperatures and the fact that the pH of the filtered liquors is not dependent on the pH of the on-flowing liquor but on the condition of the boneblack itself.

From 20 per cent to 50 per cent of the total ash entering the refinery may be adsorbed by the boneblack. Many factors will influence the variations between these limits as may be judged from the previous discussion.

Adsorption of Invert Sugar. There is no question that the reducing sugars in the char-filtered liquors are lower than before filtration, particularly during the earlier runnings, but it is doubtful whether this is a selective adsorption or a destruction due to the alkalinity of the black. Another point in question is whether the glucose is leached out after water is put on as in the case of the adsorbed ash. In an earlier edition of this book the present writer stated that this leaching out did occur, the conclusion being reached from observations of filters in practice. Blake[49] concluded from similar observations that levulose is selectively adsorbed in excess of dextrose[50] but that later this was given up to the sweet waters. An extended attempt to prove this point by laboratory experiment[51] has failed to show a single instance of such leaching out after the disappearance of the invert sugar (or glucose) nor have any char filters been found in house practice since pH

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[49] *The Planter,* **65,** No. 22 (1920).

[50] This selective adsorption of levulose from invert sugar is reported by Nakinamovitch and Zelikman; *Facts About Sugar,* Dec. 3, 1927 (abs.).

[51] Meade and Baus. Presented at Philadelphia American Chemical Society Meeting, 1926.

control was developed where the glucose ratio in the sweet waters is higher than in the highest glucose-containing material going on the black, provided all materials including sweet waters are above 7.0 pH. It appears that the apparent leaching out occurs only when acid conditions occur in the sweet waters or thin liquors, which would lead to the conclusion that the increase in glucose observed by some investigators is due to an inversion and not a leaching out. However, it may also be that the alkaline substances formed in the char during revivification aid in the adsorption of the glucose as they do in the ash adsorption as shown by Wayne, and that when this alkalinity disappears the glucose is released to the sweet waters.

The matter is of practical importance to the refiner since it involves the question whether the refinery should show a glucose balance or a glucose removal for its entire operations, if no inversion or destruction of glucose is taking place elsewhere.

Adsorption of Colloids. Much of the coloring matter of sugar solutions is colloidal, but there are also other colloidal non-sugars present. Paine and Badollet[52] studied colloid elimination during char filtration and found that the irreversible type (high in ash; do not readily redisperse when dried and taken up in water again) are preferentially and completely removed during char filtration whereas reversible colloids (gummy materials) are never completely adsorbed and are leached out in the sweet waters.

REFINING WITH VEGETABLE CARBONS AND OTHER PROCESSES

399. Introductory. Although boneblack is still the standard decolorant for large refineries in the United States, the use of activated vegetable carbons for this purpose is fairly general in Europe and is gaining ground in the United States and tropical America. Chlorine as a decolorizing agent has also come into increasing use.

It naturally follows that where vegetable carbons or chlorine are used for decolorizing, the process of refining is much the same except for the char-filter station. Because of the fact that most of the vegetable carbon or chlorine plants are smaller than those using boneblack, their processes are generally less elaborate than in the larger boneblack plants.

The various carbons and the chlorination process have been advocated by those commercially interested but it is unfortunate that no unbiased study of the various methods in comparison with boneblack has been published. Figures on costs, yields, and efficiencies have been given in controversies,[53] but it cannot be said that an authoritative study by disinterested and scientific investigators has been carried out in the United States covering these important points.

400. Decolorizing Carbons. Practically any waste vegetable material such as seaweed, peat, bagasse, press cake, rice hulls, or sawdust may be

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[52] *Facts About Sugar,* **22**, No. 3 (1927).

[53] E. E. Chapin and J. A. King, *Intern. Sugar J.*, November, 1939, p. 427.

used as the basis for a decolorizing carbon, and the number of patents issued is large, although relatively few of the products have proved of value commercially. The cost of production does not depend on the material carbonized but on the process used for the activation of the carbon. The literature on the subject is extensive, but much of it deals with the use of carbons for water purification, gas absorption, and processes other than sugar refining.[54] The theory of adsorption by carbons is similar to that for boneblack (see p. 322), and details on the subject may be had by reference to M. T. Sanders' articles.[55]

401. Comparison of Boneblack and Decolorizing Carbons for Refining. Blowski and Bon[56] made a comparative study of carbons and boneblack for refining purposes and gave the following advantages and disadvantages of the two processes:

Boneblack. Advantages.

1. Elimination of non-sugars, resulting in a decreased molasses production and an equivalent increased sucrose recovery.
2. Ease of regeneration by washing and kilning.
3. Small replacements, amounting to between 13 and 15 per cent per annum of the char in process.
4. Highly developed technology.

Boneblack. Disadvantages.

1. Large investment in plant.
2. Large amount of material to be handled.
3. Large wash-water consumption.

Decolorizing Carbons. Advantages.

1. Simplicity of process. The amount of carbon required is small, and the time in process is short.
2. Reduced inversion losses, owing to the shorter time that liquors are in process.
3. Smaller investment necessary.
4. Small wash-water requirements.

Decolorizing Carbons. Disadvantages.

1. Low non-sugar elimination.
2. High losses in regeneration. Losses at least 5 per cent per cycle.
3. Increased remelt boiling. Because of negligible elimination of non-sugars at least 140 per cent more remelts must be boiled.

These general considerations still hold both for carbons and chlorination. To the advantages of the boneblack process might be added the ability to make specialties such as large-grained sugars for manufacturing and candy-

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[54] See M. E. Barker, "The Mechanism of Charcoal Activation" (with bibliography), *Ind. Eng. Chem.*, September, 1930, p. 926.

[55] *Ind. Eng. Chem.*, **15**, No. 8, 784, 788 (1923).

[56] *Ind. Eng. Chem.*, **18**, No. 1 (1926).

making, soft sugars, and the like. So far as is known no plant not employing boneblack has ever attempted to make these sugars in competition with the boneblack process. Knowles[57] studied the refining value of boneblack and decolorizing carbons and found that "the excessive cost of application" precludes the substitution of carbon for boneblack in an established boneblack refinery.

For small plants where granulated sugar only is to be made, the carbon and chlorination processes will probably be of greatest interest. For large units, looking to produce a full assortment of refined sugars, the boneblack process is the only practicable method on the basis of present knowledge.

402. Vegetable Carbon Processes. In general, the process used for all carbons is much the same. The simple refining process already described where only high-grade liquors are filtered is used with carbon substituted for boneblack. Two-stage treatments with the decolorizing carbon are the usual practice. (See Fig. 103.)

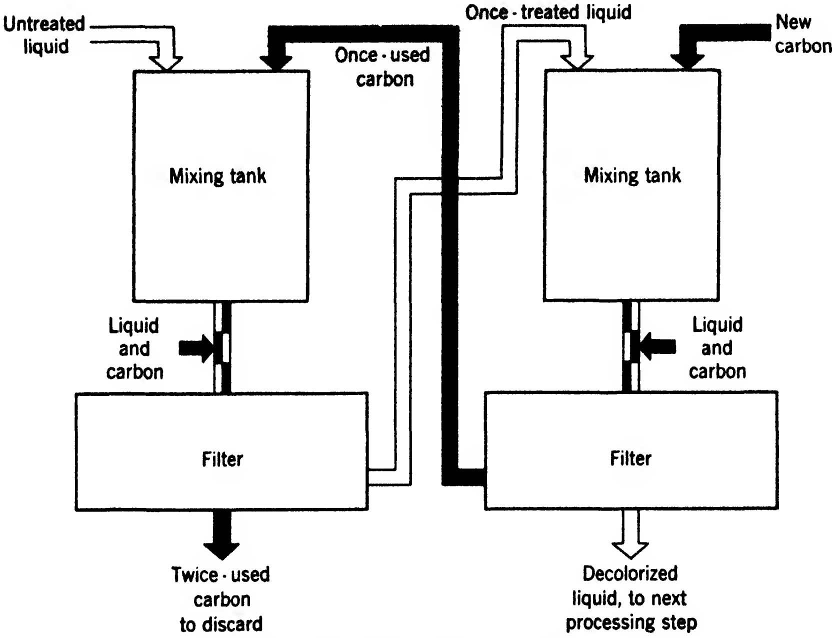

FIG. 103. Two-Stage Carbon Treatment.

The theory of countercurrent two-stage use of carbons has been discussed in detail by Sanders,[58] and a simple method for applying the theory in

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[57] Before American Chemical Society, New York, April, 1935.

[58] *Ind. Eng. Chem.*, August, 1928, p. 791.

actual practice has also been worked out.[59] A typical process in a modern plant using Darco has been described in detail with a flow diagram of the process.[60] Pressure filtration of the washed-sugar liquor is followed by treatment with once-used Darco, and the liquor thus treated is sent through Vallez filters. This once-treated liquor is then treated with the required amount of revivified Darco (1.6 per cent on solids) and is again filtered in Vallez filters, from whence it goes to the pans. The Darco cake from the second treatment returns for reuse in the first treatment, and the twice-used Darco from the first treatment is sluiced to plate-and-frame presses, washed free of sugar, then dried in a drum drier. The revivification kiln is a rotating inclined tube in an oil-fired furnace; a mixture of steam and air is admitted to the tube in proper proportions to effect the revivification. Treatment with dilute hydrochloric acid follows, and finally neutralization with caustic soda to 7.0 pH. The efficiency of the reactivated Darco is reported as 85 to 90 per cent of the original Darco, and recovery is 92 to 94 per cent of the spent material going to the kiln.

Refining with Norit, another well-known carbon, in conjunction with a raw sugar mill in Cuba is described as following much the same process as above, except that the sugar is sent from the raw factory already washed.[61] The exhausted Norit is revivified by first boiling with 2 per cent HCl, filtering, and then mixing with alkaline water (caustic soda or carbonate), followed by a second heating and filtration, after which it is revivified with heat in two stages at 400° and 650° C.

The recent tendency in refineries using carbon is to dispense with the revivification entirely (which has never been a thoroughly satisfactory process) and to discard the carbon after it has been used the second time.

An efficient application of this method is in operation at Georgia Refinery, Louisiana, where vegetable carbon is used in conjunction with the Williamson system (see Sec. 374) and phosphoric acid-lime defecation. The Jacobs clarifier is used as described on p. 304. A modification of the usual practice is the secondary clarification of the scums from the first clarification, i.e., the scums from the first clarifiers are diluted somewhat, re-aerated, and then sent to a secondary clarifier from which the thick mud goes to plate-and-frame presses. (The phosphate treatment removes about 45 per cent of the color in Cuban raws by this process.) The carbon (Darco) is used in the regular two-stage countercurrent method described above and then is discarded. Remarkably low percentages of carbon are reported in this process; 0.15 to 0.20 lb. per 100-lb. melt with Louisiana raws and 0.25 to 0.40 lb. on Cubans.[62] The decolorized liquors are of such a grade that four boilings of white sugar are made before the sirup is sent to remelt.

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[59] See *Handbook for Counter-Current Treatment with Activated Carbon* and *The Darcograph,* both published by the Darco Corp., New York.

[60] *Facts About Sugar,* June, 1937.

[61] Irazoqui, *6th Annual Proc. Assoc. Cane Sugar Tech. Cuba* (1932).

[62] Harold Jacobs, personal communication.

403. Carbons and Boneblack in Conjunction. This combination has not been tried to a great extent in this country, but it offers some possibilities that warrant serious consideration. Dedek and Kacl[63] found that carbon acting before boneblack (never in the reverse order) increases the ash adsorption because the active carbon splits up the organic salts of sodium and potassium by hydrolytic adsorption, and adsorbs the liberated anions, while the boneblack adsorbs the cations. The work of Paine and Badollet on adsorption of colloids by boneblack (see p. 327) shows that boneblack adsorbs irreversible colloids much more readily than the reversible type (gums, etc.), and it is well known that carbons adsorb the reversible colloids more easily, another evidence of complementary action of the two types of decolorants. The selective removal of color by the carbon and by boneblack is also different which is another argument for their interaction.

One plant in the United States, specializing in refined sugar sirups, uses boneblack followed by vegetable carbon for the production of high-test refined sirups. Experiments at this plant proved little difference in adsorptive power, whether the boneblack preceded the carbon or vice versa, but the removal of char taste and odor by the carbon proved an important reason for using it after boneblack. Several boneblack refineries have used carbons to remove taste and color from sirups and "liquid sugars."

404. Decolorization with Chlorine. *The Sucro-Blanc Process.*[64] Sucro-Blanc is a high-grade calcium hypochlorite. The use of the process has shown a steady increase in recent years, both in connection with raw sugar factories and in independent refinery units.

The affined sugar is melted at about 110° F. by the batch system in tanks with suitable stirring devices for quick melting. (See flow diagram, Fig. 104.) The Sucro-Blanc treatment consists of the addition of phosphoric acid followed by the Sucro-Blanc itself, which is allowed to remain in contact with the sugar melt for about fifteen minutes with vigorous stirring. The pH is adjusted by the addition of lime to the proper point, and no other pH adjustment is necessary throughout the process. This Sucro-Blanc treatment removes from 90 to 95 per cent of the color.

The treatment melt is then pumped through an aerator to the Edson clarifier, designed particularly for the Sucro-Blanc process (see Sec. 374). By the action of the impregnated air, the gas liberated by the hypochlorite treatment and the heat currents, the impurities are raised to the top of the clarifier in a very compact mass and at a much lower temperature than is necessary in other clarifiers of this type. The floating scums are continuously removed by the slow-operating chain drag. The clear liquor is continuously drawn off and is carried to the second treatment tanks where it is again treated with Sucro-Blanc (approximately 25–30 per cent of the amount used in the original treatment) giving a further color removal. The second

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[63] *Facts About Sugar,* July 30, 1927 (abs.).

[64] Descriptions supplied by A. P. Fowler, Sucro-Blanc, Inc., New York.

treated liquors are then treated with a small amount of filter aid and pumped through suitable filter presses or pressure filters directly to the pan floor. A very small quantity of sodium hydrosulfite is added at this stage to make certain that no free chlorine remains in the liquor. A bright, low-color, clear liquor results from this treatment.

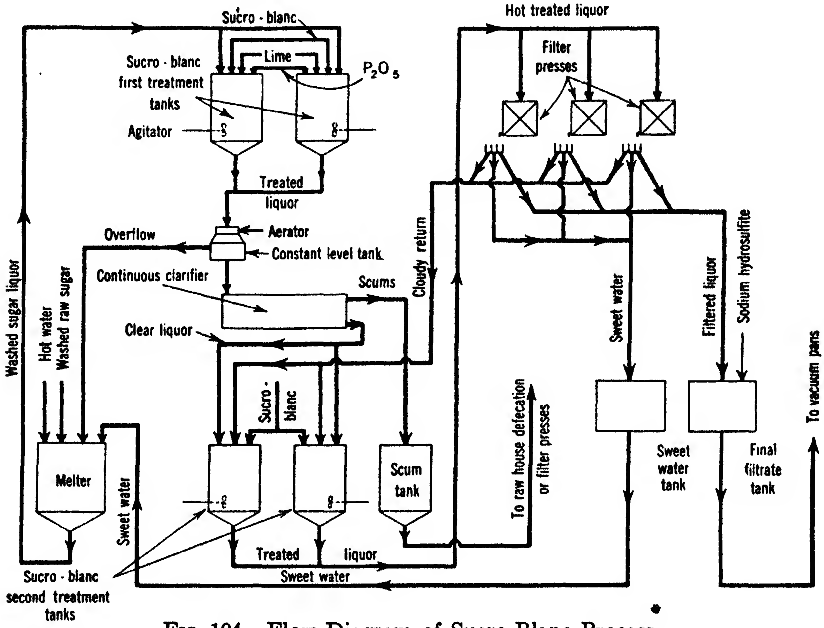

FIG. 104. Flow Diagram of Sucro-Blanc Process.

405. Sucro-Blanc Sucrocel Process. In this process the affined melt is prepared as described above by treatment with phosphoric acid, the Sucro-Blanc, pH adjustment with milk of lime, and subsequent clarification through the Sucro-Blanc continuous clarifier. The clarified liquor is then passed through percolators containing Sucrocel, which is an activated mineral ore (bauxite, see p. 323) of very high adsorptive powers. The percolation of the liquors is similar to the passing of liquors through boneblack, but the size of the individual percolators and of the entire percolating plant is only about one-tenth the size of a similar capacity boneblack plant, because contact between liquor and Sucrocel is only forty-five minutes to one hour, and only 5 per cent Sucrocel on sugar solids in the melt is employed. Further advantages of Sucrocel are that three or four cycles of liquor may be run with washing only between cycles, thereby reducing the cost of reburning. [Practical difficulties in reactivating the bauxite are encountered in actual practice, from latest reports. (*G.P.M.*)]

The percolation through Sucrocel removes up to 50 per cent of the non-sugar solids and from 92 to 95 per cent of the coloring matter of the original melt. The economies claimed for the use of the Sucro-Blanc Sucrocel process are as follows:

1. Elimination of pressure filtration, with reduction in labor cost, filter cloth, and filter aid.[65]
2. Savings in steam because of high Brix liquors which may be run through the process.
3. Increased yield due to removal of non-sugars by Sucrocel.

406. Purification or Refining by Ion Exchange. The removal of impurities from sugar solutions or juices by ion exchange adsorbents is a development of very recent years which may prove to be the most revolutionary advance in modern sugar processes. In the beet sugar industry the method has been introduced on a plant scale,[66] and active experimenting is being done with cane sugar solutions. In its fundamentals, the process consists of treating the sugar solution first with a cation exchanger which replaces the cations (calcium, magnesium, sodium, and potassium) with hydrogen ions, converting the neutral or alkaline salts into the corresponding acids. This reduces the juice to a pH as low as 2.0. Then the acid juice is treated (by percolation) with an anion exchanger which removes the free acid, leaving the juice alkaline again, practically water white and ash free. In practice a vegetable carbon treatment between the cation and anion exchangers has been found of practical advantage in reducing the load on the anion exchange bed. The exchangers can be regenerated by treatment with sulfuric acid and caustic soda respectively. The ion exchangers are synthetic organic compounds.[67]

The spectacular results, complete decolorization and purity increase from 88° to 99° with only a few percolation tanks as equipment, are most encouraging, but there are several obstacles which may prevent general adoption, especially in cane sugar work. The exchange must be carried out on thin solutions (15° Brix) and the very low pH's during the process necessitate cooling the solutions to 20° C. to prevent inversion. The exchangers themselves are expensive and the cost of the regenerating chemicals is high. Some or all of these objections may be overcome by further experimentation, but the present status (1943) offers more promise for beet than for cane sugar work.

CRYSTALLIZATION OF THE SUGAR

407. General. The procedures so far given in this chapter have resulted in liquors more or less thoroughly decolorized by boneblack or other processes. Boneblack is generally used to produce many grades of sugar, and therefore the sections which follow deal with the boneblack refineries, but it is to be

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[65] These advantages are those of the Williamson defecation system which is employed in the Sucro-Blanc Sucrocel process. (G.P.M.)

[66] Weitz, *Sugar*, January, 1943.

[67] See Tiger and Sussman, *Ind. Eng. Chem.*, February, 1943, p. 186.

understood that the methods described for producing the ordinary granulated sugar also apply to those refineries which use vegetable carbons or chlorine.

408. Classification of the Liquors. The liquors are classified at the filter outlet pipes according to their purity and color and are distributed to storage tanks near the vacuum pans. In the continuous system of filtration, in which no two filters are at the same phase of their cycles, all grades of liquors are flowing at the same time. The liquors are classified about as follows. *First liquor:* filtered washed-sugar liquor, water white and of 99° to 99.5° purity; *second liquor:* filtered granulated sirups, off white or slightly yellow and of 90° to 93° purity; *third liquor:* filtered or double-filtered washings or low-grade meltings, golden yellow and of 84° to 87° purity; *fourth liquor:* last runnings, too dark to make granulated sugar and of 75° to 80° purity. As has been stated above triple filtration of these liquors for soft-sugar work is common practice.

Those refineries where affination sirups are boiled to remelt without char filtration generally have only two grades of char-filtered liquors.

Pressure filtration with inert filter aid of the filtered liquors after leaving the boneblack is practiced in many refineries to remove any faint haze or cloud from the liquors. A small portion of phosphoric acid is sometimes added before this pressure filtration. Since there is so little filterable material in the char-filtered liquors the cycle of these press filters is very long and the amount of filter aid used is not great. In those refineries using Williamson defecation this secondary press filtration is not generally practiced as hazy liquors are rare.

409. Boiling to Grain. The vacuum pans used in refining do not differ materially from those of the raw sugar factory. The white sugar pans are usually of a lesser height in relation to their diameter so as to obtain greater heating surface per volume and to speed up the boiling. Twelve-, 14-, and even 16-ft. diameter pans are employed. Calandria pans are used for remelt sugars and also for white sugar strikes in some refineries. The use of pan-control instruments is quite general; in fact many of the advances in automatic pan boiling have been devised and perfected in refineries in the United States. (See Sec. 240.)

The Webre pan with forced circulation (Sec. 230) has been found effective for both soft sugars and refinery low-grade strikes. Most refineries now evaporate in multiple effect a large part of the No. 1 liquor coming from the char filters, generally to a Brix of 67° or even 70°. This heavy liquor is used to charge the pans, thereby economizing both in time and in fuel. White sugar strikes using this heavy liquor are frequently boiled in as short a time as one hour. In general the boiling time is an hour and a half to two hours.

The general principle of pan manipulations (see Chapter 11) is the same in the two branches of the industry, but from the nature of the product, greater care must be exercised in the refinery because of the necessity of boiling sugars to certain specifications of size and hardness of the crystals.

It is now almost universal practice to use seed graining for making sugars of a grain size larger than the ordinary table granulated. A calculated amount of seed grain of the proper size is drawn into the pan when the liquor has reached the graining point (see Sec. 247), and these crystals grow as the pan boiling progresses until they are the desired size when the pan is full.

In boiling granulated and other hard sugars, a high pan temperature is customary (160°–180° F.), whereas for soft sugars, in which a small spongy grain is desired, low temperatures (110°–130°) prevail.

Scroll Calandria Pans. A pan of special design built for the refinery of Tate and Lyle in London [68] has a scroll calandria which is made up of special heating elements consisting of two special steel plates welded together at top and bottom to form a long rectangular cross section with sharp edges at top and bottom. This type uses steam at 70 lb. pressure, and although having the form of a calandria pan it is in effect a coil pan with special-shaped spiral heating elements. There are many unique features to these unusual pans, among which are a steam-jacketed bottom and belt around the calandria; very great height in relation to diameter (35 ft. high, 14-ft. diameter); a motor-operated discharge valve 4 ft. in diameter, and a vapor space the same diameter as the pan. The purpose of this special design is to permit the boiling of very dense strikes, so heavy that they must be lubricated with saturated white sirup in order to discharge and purge the massecuite. Yields of granulated sugar of 64 per cent are reported for first boilings as against 50 per cent yield or less with ordinary methods. The steam savings are naturally very great, but there is considerable darkening of sirups during the boiling process to these heavy densities.

410. Refinery Boiling System. Selected water-white first runnings of filters are used for the fancy grades of refined: cubes, cut loaf, confectioners' sugar, etc. The bulk of the output is the granulated sugar of commerce, called in the trade standard fine granulated or extra fine granulated, and the boiling system is planned for the production of this grade. The system generally employed is to have one or more pans continually working on straight liquor strikes, while the sirups purged from two of these strikes are combined and boiled in another pan to first sirup strikes. These strikes yield still lower-purity sirups which are combined and boiled to second sirup strikes. The sirups purged from these second sirup strikes combine to make a strike which yields a sirup of about 90° purity, and this is sent back to the char house for refiltration where it is called granulated sirup, or sirup for boneblack, and returns to the pan floor as second liquor. The second and third liquors are boiled to make off-granulated strikes. The sugars from these different grades of strikes are mixed to make a product of uniform quality.

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[68] Oliver Lyle, *Intern. Sugar J.*, July, 1939, p. 262.

The sirups from the off-granulated strikes are boiled with fourth liquor or with unfiltered affination sirups for high remelt strikes of 75° to 80° purity. The sugars of these strikes are washed to a high test (98° purity) and are melted directly with the washed raw sugar. The sirups from these strikes are boiled back on a footing of fourth liquor or affination sirups to make strikes of 64°–66° purity. These magmas are discharged into crystallizers where they are kept in motion during two to three days. They are then purged without the use of wash water and yield a sugar from 85° to 90° purity. This sugar may be mixed with high-test raws and washed with them, or it may be melted directly with other low-grade raws and be submitted to the processes already described. Preferably, the sugar is mingled with a heavy sirup of low test, repurged, and washed to give a 96°–98° test sugar which is then melted and sent to the defecators with the high remelt sugars. The use of high-speed centrifugals and the hot mingling process (Sec. 293) for purging the crystallizer strikes results in a high-test sugar without the necessity for repurging. The sirups purged from these low-grade remelt strikes (or crystallizer strikes) form the residual sirup or barrel sirup of the refiners.

In the days when soft sugars were produced by some refineries to the extent of 25 to 30 per cent of the total output the pan boiling system of the soft-sugar houses revolved around the production of softs, although the high-test liquors were boiled to granulated sugar in much the same way as that described above. Now few American refineries produce soft sugars beyond 10 per cent of their output so the boiling of soft sugars, though still a very important and profitable part of refining, has been more or less subordinated to the production of granulated.

Soft sugars are graded according to color, varying from an almost pure white to a deep brown sugar. The desirable characteristics from the trade point of view are: texture, which should be spongy and free from any sharp crystalline appearance; color, which besides conforming to the desired grade should be free from grayish or greenish tones; flavor, freedom from harsh or salty taste. The refiner seeks in addition to these keeping quality and low polarization. Soft sugars to attain these characteristics must be boiled from liquors free of turbidity, low in total color, and free from greenish-gray cast, relatively low purity, low in ash, and high in invert sugar content. These characteristics of the liquors are in part due to the character of the raws being melted but are largely dependent on char filtration. Given the grade of softs which the market requires, liquors conforming to the characteristics and proper test are selected for the production.

For the light yellow soft sugars, char-filtered granulated sirups and double or triple char-filtered affination wash sirups of 86°–88° purity will form the basis of the strikes. The sirups from these strikes, reboiled with a footing of the lighter-colored liquors, will yield the middle grades of softs, and the darker grades are made from once char-filtered affination sirups or from defecated and press-filtered affination sirups. The varying demand for the different grades necessarily precludes the possibility of a fixed boiling system for soft sugars.

411. Evaporation of Sweet Waters. The various sweet waters in a refinery are handled in one of two ways. Those of high purity, e.g., those produced by filtering the mud waters of the defecation system, are used for melting the raw sugar, and those of lower purity (dark press waters, char sweet waters; bag-wash waters, etc.) go to the multiple-effect evaporator for concentration to a sirup of 60° Brix (concentrated sweet water) which is boiled to remelt. The multiple effects generally installed in the older refineries are of the horizontal tube type (Lillie, Swenson, etc.) although the standard evaporator used in raw houses serves equally well. In many refineries it is customary to use one or two bodies of the multiple effect for evaporating char-filtered liquor to heavy density (67°–68° Brix) to save steam at the vacuum pan station.

DRYING AND FINISHING THE PRODUCT

412. Centrifugal Work. The separation of the crystals from the sirup in the massecuites is effected by centrifugal machines such as are used in the factories. The white sugars are very thoroughly washed with hot or warm water in the machine to remove all adhering sirup, the amount of water used per charge being accurately measured by automatic devices. The quantity of water varies with the grade of massecuite, first liquor strikes requiring from 12 to 20 lb. of water and off-granulated strikes as much as 40 lb.

Soft sugars are not washed in the centrifugals except in rare cases; the time of purging is used to regulate the color to some extent by leaving more or less sirup on the grain.

Because of the nature of the product, the centrifugal work of a refinery is in general more carefully controlled than in a raw house, and the centrifugal machines are supplied with automatic devices for timing the cycle, adding the water, braking, discharging, etc. The use of high-speed centrifugals (see Sec. 291) with heating devices in the minglers is coming into general practice.

The purging of white sugar massecuites by vacuum filters of the Oliver type has been experimented with but not adopted on a commercial scale.[69]

413. Drying the Sugar. The moist sugars discharged from the centrifugal machines contain about 1 per cent of water and are conveyed to distributing bins above the granulators by scroll or belt conveyors and bucket elevators. The bulk of the white sugar is sent through the granulators for drying.

The granulator is so called because it separates the crystals from one another. The granulator is an iron drum (Fig. 105) about 6 ft. in diameter and 25 ft. long, slightly inclined to the horizontal toward the discharge end, and revolving on rollers. A series of narrow shelves attached to the inside of the drum, longitudinally, serve to lift the sugar and let it fall through heated air as the drum revolves. The shelves generally have saw-

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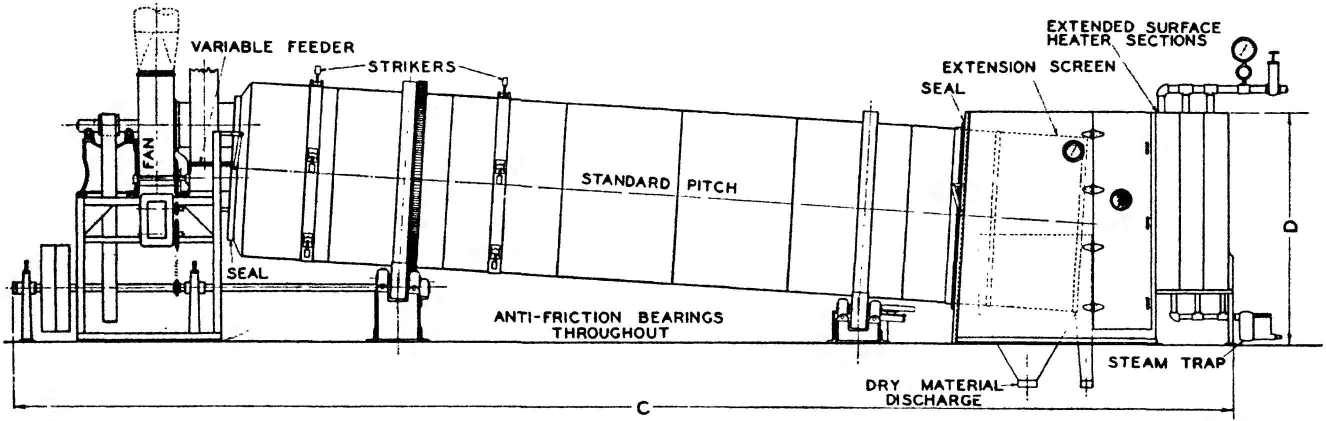
[69] Godefroy, *Ind. Eng. Chem.*, **25**, No. 9, 964 (1933).



Hersey Mfg. Co.

Fig. 105. Granulator Drum.

toothed edges which distribute the curtain or storm of sugar more regularly as it falls through the heated air. The air is heated either by means of a steam drum extending through the middle of the granulator or by a large group of steampipes at the discharge end of the drum. (The latter is more common in modern installations.) The air is drawn over the pipes and through the granulator by an exhaust fan which removes the sugar dust. The illustration is of a Hersey granulator, the usual American type.

Two granulator drums are usually operated in series, one above the other. The sugar leaves the upper drum partly dried and quite hot, hence very little steam is required to complete the drying in the lower granulator. The sugar should leave the lower drum comparatively cool, preferably below 110°. A third drum, connected in series with the other two and containing no heating element, is sometimes used to cool the sugar. Some modern refineries supply filtered and washed air to the granulators to avoid the entrance of dust and microorganisms into the sugar.

Single-pass granulators, i.e., in which the sugar passes through one drum only, are not uncommon. An unusual arrangement of single-pass drums is in the new Tate and Lyle Refinery, London. The angle of tilt of the granulator is variable.[70] The tilt is increased when the sugar is drying well so that there are fewer falls during the passage of the crystals, and the tilt is reduced for poor drying or fine-grained sugars. Dry, unscratched and cool sugar is reported as produced by this system.

For large-grain sugars the granulator type of drier is not usually employed as the falls tend to scratch the crystals. Multi-pass belt driers, riffles, and other means are employed, as well as small-diameter drums in which the crystals roll in a warm air current without falling.

414. Roto-Louvre Drier. An account of the Link-Belt Company's Roto-Louvre drier by Morton Goldstein[71] indicates that this new type of drier has distinct advantages over the old granulator. The Roto-Louvre drier consists of an outer cylindrical shell or drum (6 ft. by 18 ft.) similar to the conventional type granulator but with an inner shell made up of louvre plates which are attached to full-length tapering radial plates, forming an inner shell of gradually increasing diameter as it approaches the discharge end. Therefore the space between the outer and inner shells is partitioned into many hot-air passages, the only exit of which is the space between the louvre plates.

Wet sugar is fed at a uniform rate into the inner drum, over the louvre plates, and while the drier rotates, the bed of sugar is slowly tilted in the direction of rotation, causing it to fall over onto the top surfaces of adjacent louvre plates. The sugar travels through a spiral path to the discharge end of the drum, keeping constantly but gently in motion.

Hot air is regulated automatically by the sloping inner shell which causes the sugar bed to be thinnest at the feed end and become gradually thicker

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[70] *Intern. Sugar J.*, July, 1939, p. 266.

[71] Before Sugar Industry Engineers, December, 1942.

as it flows forward. The thin bed offers least resistance to the passage of hot air, and consequently the greatest air volume passes through the bed at the feed end where the sugar is wettest.

415. Screening the Sugar. The sugar falls from the granulator to a set of screens which classify it according to the size of the crystals, remove the lumps and coarser dust, and deliver the products into the packing bins. The older screens are of the bolter type, consisting of a revolving horizontal casing, inclined slightly, covered with wire screen of the desired mesh through which the sugar falls, and the tailings are delivered at the far end of the bolter. The more modern screening devices are of the vibrating or tapping types. These consist of flat screening frames, inclined at about 35° to the horizontal, down which the sugar flows while mechanical devices tap or vibrate the screening surface to cause the crystals to go through the meshes.

The Rotex type of screen is coming into increasing use both for ordinary granulated and for large-grained specialties. This type has nearly horizontal screening surfaces which are rotated in an endwise and sidewise motion. Rubber balls held in compartments below the screening surface bounce against the wire meshes and keep them clean. Generally speaking the various sizes of sugar on the market are boiled to the given size and not graded by screening. The screening is for the purpose of removing the undesirable portions.

416. Dust-Collecting System. The fine crystals drawn off by the ventilating fans in the granulators are caught in ordinary Cyclone dust collectors to be sold as superfine or fruit sugar, but a portion is too fine to be caught in this way and passes on to wet-type dust collectors of which there are several designs. The most recent of these consists of a chamber filled with a staggered set of Bakelite rods down each of which a stream of hot sweet water flows in spiral grooves. The dust-laden air passing by the whirling streams of hot liquid is completely freed of sugar.

417. Packing the Sugar. The screened sugar is packed direct as it comes from the screens, or may be sent to storage bins for packing during the daytime only.

Much of the refined sugar goes into 100-lb. bags (either cotton-lined burlap, heavy cotton, or five-ply all-paper) into which the sugar is automatically weighed and then sewed (see Fig. 106). Barrels, which formerly were in greatest demand, are now packed in relatively small proportions. Small cotton packages, paper packages, and cartons are all automatically filled, weighed, and closed, the multiplicity of grades and packages making the finishing house of a large refinery an elaborate establishment in itself.

One representative refiner reports sixty-two different types of sugar in two hundred and seventy-seven packagings.[72] The most recent tendency in sugar packing is toward paper containers.

418. Grades of Sugar. *Granulated.* Granulated sugar forms a large part of the output of all refineries and the total product of the smaller establish-

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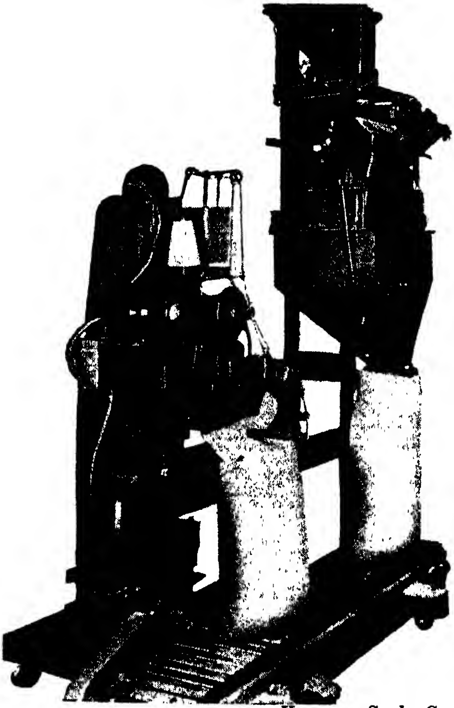
[72] Wills, *Facts About Sugar,* July, 1938.



Hoepner Scale Co.

Fig. 106. Automatic Scale and Sewing Machine.

ments. The ordinary commercial granulated used to be sold in two grades, standard fine and extra fine, but the demand is for finer crystals, and most refineries now sell only the extra fine grade. Some confusion arose because the term extra fine was believed to mean an extra quality sugar rather than a finer grain.

A still finer grade, consisting either of fine screenings or the fine crystals drawn off by the granulator fans, is called fruit granulated, fruit powdered, dessert sugar, berry sugar, or superfine. Coarser grades than the regular granulated are sanding sugar, manufacturers' granulated, medium coarse, coarse and extra coarse. These larger crystal sugars are used by bakers, candy-makers, bottlers, and other manufacturers. Brilliant or Candy A sugar used to be produced as a large-grain sugar packed in barrels while moist, as it left the centrifugals, but these moist sugars have disappeared from the market.

Cube and Tablet Sugars. The various kinds of lump and loaf sugars are made by mixing sugar of the proper grain with a heavy white sugar sirup to form a moist mass. This is then variously treated by pressing or molding to form the cubes, or slabs, after which the cubes or slabs are dried in hot closets. The cubes are ready for packing after they have been dried. The slabs and bars are cut or sawed to form cut and sawed loaf sugar. These are broken down in making crushed loaf sugar.

Powdered, Pulverized, or Confectioners' Sugars. These are made to various degrees of fineness by grinding coarse granulated in specially designed mills, which are generally of the hammer type revolving at 3500 rpm. The sugar may be air-separated, screened through bolting cloth or, in the more modern mills, passed direct to the package. The powdered sugar designated as 4X or 6X contains 3 per cent of corn starch to prevent caking and all will pass through a 100-mesh screen. Tricalcium phosphate (1 per cent) to replace the starch has been used to some extent as a preventive of caking in these sugars.

Soft Sugars. The soft yellow and brown sugars are classified according to a series of arbitrary trade color standards numbered from 1 to 16.

The grades from No. 1 to No. 5 are so nearly white as to be rarely called for, and the very dark brown sugars No. 15 and No. 16 are also in little demand. Most refineries produce only five or six grades of softs, designating these by trade names, as well as numbers. The most popular grades are No. 8, which is light yellow in color; No. 10, which is a deep yellow or light brown; and No. 13, a deep brown.

The soft sugars are packed while moist, generally after screening or aerating and cooling. It is usual to pack these sugars in moisture-proof containers, such as laminated asphalt paper, wax paper, or rubberized paper sheets between other layers of kraft paper. The paper may serve as a liner for a textile bag or may be a part of an all-paper container.

419. Liquid Sugar, Inverted Sirups. A new phase of marketing refinery products has arisen during the past decade in the sale of high-test liquors under the name of liquid sugar. These liquors are about 68° Brix and of two or three grades, the most popular of which is water white. They are produced in the refineries located in large manufacturing centers and delivery is made in glass-lined tank trucks. The price is based on the sugar content with an allowance for the fact that no package is required and no crystallization or drying has taken place.

Inverted refined sirups have also become an important factor in the marketing of sugar to ice cream manufacturers, bakers, candy-makers, etc. These sirups may consist entirely of invert sugar, that is, may contain no uninverted sucrose, but more generally are made of a mixture of refinery liquor or filtered sirup and a portion of invert sugar sirup sufficient to prevent crystallization. These sirups vary in color and analysis to suit the needs and purposes of the buyer. The invert sirup is made by inverting refined sugar either with hydrochloric or sulfuric acid (see Sec. 333) or by a special strain of yeast.

420. Refinery Sirup, Barrel Sirup, Refinery Blackstrap. The end product of the refinery is the sirup purged from the lowest-grade remelt strikes which have been cured in crystallizers. In those plants which do not char-filter their low-grade materials, this product is refinery blackstrap and does not differ materially from cane factory blackstrap, although it is usually less viscous. It is used for the same purposes, viz., alcohol manufacture, cattle feed, yeast (see Chapter 14). In refineries which employ intensive char filtration the final sirup is frequently char-filtered also, and then boiled smooth, to about 80° Brix. This product, known as filtered barrel sirup, is used for blending with other sirups, but it is not in as great demand as before the days of inverted sirups. Thirty-five years ago this product was shipped in oak barrels (hence the name) but today tank cars and tank trucks (or more rarely steel drums) are the means of transporting.

This sirup varies in composition through rather wide limits, depending on the class of sugar from which it is derived and upon other factors. A typical analysis would be as follows: Brix 82°; polarization 32 per cent; sucrose 36 per cent; reducing sugars, 20 per cent; ash 8 per cent; water 24 per cent; organic non-sugar (by difference) 12 per cent.

421. Yield of Refined Sugar. The yield of sugar in a refinery is expressed in percentage terms of the raw sugar melted. The following figures are illustrative: polarization of the melt, 97°; granulated yield, 95.0 per cent; sirup yield 4 per cent at 32° polarization (1.28 per cent of melt). Pol lost in manufacture 0.72 per cent.

Where soft sugars, inverted sirups, and other products besides granulated sugar are made these are calculated to available granulated and sirup as will be shown later (Sec. 423).

REFINERY TECHNICAL AND CHEMICAL CONTROL

422. Introductory. The technical control of a refinery is more positive and direct than that of a raw sugar factory because the weighing and analysis of the raw material entering the refinery offer no difficulty. All the control figures are computed upon the basis of the weight of raws. A technical statement may be made up with comparatively few figures: the analysis and weight of the raw sugar melted; the analyses and weights of the refined sugars and sirup produced; the stock-in-process figures for the beginning and end of the period.

The normal control of a refinery is generally on a polarization basis but a true sucrose balance should be made and used as a check on polarization figures.

423. Technical Control. Each package of raw sugar is weighed and sampled as it is dumped, and the samples are polarized at convenient intervals, usually twice daily. Complete analyses are made weekly, or as often as desired, of a weighted sample made up from the daily melts. Besides the average polarization, which is computed from the component polarizations, the invert sugar, moisture, ash, and organic non-sugars are determined by the usual methods of sugar analysis. The analysis of the melt of the technical period is computed from these analyses and the weights of material which they represent.

Most refineries base their loss figures on the settlement weight and test by which the raw sugars are purchased. Where dutiable sugars are imported the customs weights and tests serve as checks on the plant laboratory.

The refined sugar is automatically weighed as packed. No analysis of the granulated sugar is made and its polarization is always taken as 100°. Soft sugars are weighed, sampled, and analyzed, as are the raw sugars, and a weighted average analysis is calculated for the period.

The barrel sirup is sampled and measured by lots, and the density and polarization of each are determined. The weighing may be conveniently done by means of the Pneumercator (sec Sec. 778). Weekly complete analyses are made of a composite sample, and a weighted average analysis for the technical period is computed. At the end of every period—usually the technical periods include four weeks—an inventory of the material in process is made. All material containing sugar is measured and sampled. The density (Brix by hydrometer) and apparent purity of the samples are determined. The pounds of solids are computed from the volume of the material in cubic feet and the pounds solids per cubic foot, as indicated by the density. The solids multiplied by the purity give the pounds pol. The solids less the pounds pol give the solid impurities. In this way the total solids, total pounds pol, and total impurities in the stock are ascertained. To compute these figures to available sirup and available granulated sugar, it may be assumed that all the impurities will go to make sirup of the same composition as that produced during the period.

Then:

$$\frac{\text{Pounds solid impurities in stock} \times 100}{\text{Per cent solid impurities in sirup produced}} = \text{Pounds available sirup in stock} \quad (1)$$

$$\frac{\text{Pounds available sirup in stock} \times \text{Polarization of sirup produced}}{100} = \text{Pounds pol in sirup in stock} \quad (2)$$

$$\text{Total pol in stock—pol in sirup in stock} = \text{Pounds available granulated in stock}$$

Example. Assume a stock in process of 1,180,000 lb. solids
850,000 lb. pol
330,000 lb. solid impurities.

Assume a barrel sirup produced as containing:

	Per Cent
Polarization	34.2
Water	22.1
Solid impurities (ash, glucose, organic)	43.7
	100.0

$$\frac{330{,}000 \times 100}{43.7} = 778{,}032 \text{ lb. available sirup} \qquad (1)$$

$$\frac{778{,}032 \times 34.2}{100} = 258{,}261 \text{ lb. pol in sirup in stock} \qquad (2)$$

$$850{,}000 - 258{,}261 = 591{,}739 \text{ lb. available granulated in stock}$$

The increase or decrease in the available granulated in stock in process at the end of the period as compared to that at the beginning is added to or subtracted from the actual production of granulated for the period; the same calculation is made with regard to the sirup produced and the sirup in stock. These are *net* productions of sugar and sirup for the period.

These net weights produced are each divided by the weight of raws melted to give the net productions per cent melt. The analyses of the granulated, the soft sugars, and sirup are calculated to per cents of the melts by multiplying each constituent of the analysis of each of these three products by the percentages of the product in terms of the melt. The various constituents of these analyses per cent of the melt can be totaled to give the analysis of the combined output of the refinery in terms of the melt. A comparison of this analysis with that of the melt itself gives the increase or decrease of each constituent during the process of refining.

To illustrate this, let us assume a set of net yields for a refinery period and calculate the polarization loss for the period:

Polarization of the melt	97.0°
Net yield of granulated	92.20 per cent
Soft sugars produced	3.5 per cent at 89.0° polarization
Net sirup production	3.3 per cent at 32.0° polarization

Then,

Pol in melt		97.0
Pol in granulated per cent melt	$= 92.20$	
Pol in soft sugars per cent melt	$= \frac{3.5 \times 89.0}{100} = 3.12$	
Pol in sirup per cent melt	$= \frac{3.3 \times 32.0}{100} = 1.06$	
Pol in total product per cent melt		96.38
Pol lost in refining per cent melt		0.62

Following this same method of calculation, the gain or loss of invert sugar, ash, water, and organic non-sugar may be traced.

424. Chemical Control. The chemical methods employed in a refinery are similar to those used in factory work as described in the sections of this book devoted to analytical procedure. Clerget sucrose as well as direct polarization are determined on entering raws, barrel sirups, and soft sugars in order to check the pol balance previously described.

The routine control of the refinery depends largely upon determinations of the apparent purity of the materials in the successive steps of the process. The char filter and pan work are entirely controlled on a basis of purity. The number of these purity tests required day and night is so large that the work is usually conducted in a separate laboratory, on or near the vacuum pan floor, by men trained to do this testing only. This leaves the chemists free to do the analytical work involved in the technical control.

The dilute purity method of Casamajor is used in this work. The material, liquor, magma, or sirup, is diluted to any convenient density, usually between 15° and 20° Brix, and the corrected Brix is determined. A part of the solution is clarified with Horne's dry subacetate of lead, if such preparation is necessary, and after filtration it is polarized directly. The polariscope reading multiplied by the factor corresponding to the degree Brix gives the purity coefficient. The factors are computed from the formula

$$\text{Factor} = \frac{26.0 \times 100}{99.718 \times \text{sp. gr. } (20^\circ/20^\circ) \times \text{Brix}}$$

which is Casamajor's formula as modified by Rice for temperatures of 20° C. A table of these factors is given on p. 766. The figuring is simplified by the use of the expanded Horne's table of purities for use in refinery control, given on p. 767.

425. Impurities Control. The English refineries employ a control by impurities rather than by purity[73] which has certain obvious advantages. The determination of ash in high-test materials by electrometric methods (see Chapter 22) can be more rapidly carried out than a polarization and it gives a much closer check on the quality of such materials than does the purity which at best is correct to ±0.1 per cent. The small quantities of invert sugar present in such high-test materials are also determined by rapid routine methods and these in turn give a much clearer idea of the quality of the liquors and sugars than could purity determinations. A close color control is also maintained in the English refineries, but this is generally customary (though possibly not to as great an extent) in sugar refineries in the United States.

426. pH Control. This has become an essential part of refinery control work. The various methods that are used are as given in Chapter 23. The regulation of the reaction at the blowups has already been referred to (p. 307) and maintenance of pH within close limits is customary at this station. Tests of the pH of the materials going on and coming off the char are also frequently made and the pH of the water extract of the revivified char has been shown to be of the greatest value (p. 350). A careful study of the whole subject of pH control in a large refinery has been published by Blowski and Holven.[74]

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[73] Lyle, *Technology for Sugar Refinery Workers*, London, 1941, p. 317.
[74] *Ind. Eng. Chem.*, 17, No. 12 (1925).

SPECIAL ANALYTICAL METHODS

427. Available P_2O_5 in Phosphoric Acid Paste. Ten grams of the well-mixed paste are weighed rapidly and washed into a liter flask. Make to the mark, and shake thoroughly for fifteen minutes to effect solution of soluble phosphates. Filter a small portion of the suspension, and pipet 10 ml. of the clear filtrate into a 300-ml. beaker. Add about 5 grams of ammonium nitrate crystals and dissolve. Heat the phosphate solution to 65° in a water bath and add 75 ml. of filtered ammonium molybdate solution all at once into the phosphate solution. Stir vigorously and allow to stand at 65° for a half hour; allow the precipitate to settle thoroughly. Decant the clear supernatant liquid through a funnel with a moderately fast filter paper. Add about 30 ml. of cool distilled water, let settle, and decant again. Repeat three or four times, then transfer the remainder of the precipitate into the filter. The precipitate in the filter paper is then washed with water until the combined filtrations of two successive washings yield a red color to phenolphthalein upon the addition of one drop of standard alkali. The filter paper and precipitate are then returned to the original precipitation beaker, 100 ml. of water are added, and the paper is stirred to almost complete disintegration by means of the stirring rod. Then add from a buret a measured amount of 0.3238 N potassium hydroxide, free from carbonates, until the yellow precipitate is all dissolved by vigorous stirring. Just before all the precipitate is dissolved add phenolphthalein as an indicator. Titrate back the excess of alkali by means of 0.1619 N HNO_3,

$$\text{Available } P_2O_5 = \text{Ml. } 0.3238\ N\ \text{KOH} - \frac{\text{Ml. } 0.1619\ N\ \text{HNO}_3}{2}$$

428. Analysis of Boneblack. *Preparation of the Sample.* After thoroughly mixing the sample reduce it to 200–300 grams by subsampling. Pass a magnet through a thin layer of the sample to remove particles of iron that may have gotten into it from the retorts and filters. In a porcelain mortar grind about 100 grams of this prepared char to a powder, all of which should pass through a 100-mesh sieve. The ground sample must be kept in a tightly stoppered bottle to prevent absorption of moisture.

Moisture Determination. Heat 5 grams of the unground portion of the subsample for four hours at 110° C.

$$\text{Loss of weight} \times 20 = \text{Per cent moisture}$$

Carbon and Insoluble Matter. Treat 2 grams of the ground char with 10 ml. of concentrated hydrochloric acid and 50 ml. of water. Boil gently for fifteen minutes, filter through a tared Gooch or alundum crucible, and wash the residue with water to the disappearance of chlorides. Dry the crucible and contents at 100° C. and weigh; ignite to constant weight over a flame or in a muffle.

$$\text{Loss on ignition} \div 2 \times 100 = \text{Per cent carbon}$$

$$\text{Residue after ignition} \div 2 \times 100 = \text{Per cent insoluble matter}$$

Determination of Calcium Sulfate. To 25 grams of the powdered char, in a 250-ml. flask, add 25 ml. water and 100 ml. concentrated hydrochloric acid, gradually. Boil fifteen minutes. Add about 100 ml. water, and after cooling to room temperature, dilute to the mark with water, mix, and filter. Evaporate 200 ml. of the filtrate (= 20 grams of char) to about 150 ml. Proceed with the analysis as is described on p. 665, using the concentrated solution obtained as above. The barium sulfate precipitate should first be washed by decantation.

Calculation:

$$\text{Weight barium sulfate} \div 20 \times 0.5833 \times 100 = \text{Per cent calcium sulfate } (CaSO_4)$$

Determination of Calcium Sulfide. To 25 grams of the powdered char in a 250-ml. flask add 0.5 gram of potassium chlorate, then 25 ml. of boiling water, and follow this with 100 ml. concentrated hydrochloric acid, *added very slowly*. Proceed as in the determination of calcium sulfate, described above. Great care must be exercised in adding the acid, very slowly at first, in order that no sulfur be lost as hydrogen sulfide (H_2S). The barium sulfate obtained in this analysis corresponds to the sum of the sulfide and sulfate in the char. Subtracting that already found for the calcium sulfate leaves the barium sulfate equivalent to the calcium sulfide.

Calculation:

$$(\text{Total barium sulfate} - \text{Barium sulfate derived from the calcium sulfate}) \div 20 \times 0.3091 \times 100 = \text{Per cent calcium sulfide } (CaS)$$

Volumetric Determination of the Iron.[75] The following reagents are required:

1. Standard potassium permanganate: Dissolve 4 to 5 grams of the salt in 1000 ml. of water. Check this solution against an iron solution of known strength, prepared as follows. Dissolve 2.5 grams of piano wire, or of the grade of iron wire that is prepared especially for standardizing, in a small quantity of hydrochloric acid, and dilute this solution to 250 ml. in a graduated flask. Use 50-ml. portions of this solution under the conditions of the analysis, as below, in standardizing the permanganate.

2. Phosphoric acid and manganous solution: Dissolve 50 grams of manganous sulfate crystals in about 250 ml. of water, with the addition of a few drops of sulfuric acid; add 250 ml. of phosphoric acid solution of 1.3 specific gravity, followed in order by 150 ml. of water and 100 ml. of concentrated sulfuric acid. The phosphoric solution may be prepared from the 85 per cent acid (H_3PO_4).

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[75] Adapted from Clowes and Coleman, *Quantitative Analysis,* fifth edition, p. 206.

3. Stannous chloride solution: Dissolve 30 grams of pure granulated tin in 125 ml. of concentrated hydrochloric acid, with heating. Solution is promoted by the addition of a few pieces of platinum foil. Dilute the solution with 250 ml. of water and filter it through asbestos. Add 250 ml. of concentrated hydrochloric acid and 500 ml. of water to the filtrate.

4. Mercuric chloride solution: Dissolve 50 grams of the salt in 1000 ml. of water.

Proceed with the analysis as follows. Ignite 10 grams of the powdered boneblack and digest the residue with 30 ml. of concentrated hydrochloric acid, with gentle boiling during fifteen minutes. Filter the solution through a Gooch crucible and wash the residue thoroughly with small quantities of hot water. Heat the filtrate, contained in a large Erlenmeyer flask, to nearly boiling and add the stannous chloride solution to it drop by drop until the yellow color disappears. Add 60 ml. of the mercuric chloride solution, all at once, and mix well by shaking the flask; add 60 ml. of the phosphoric acid and manganous solution and 600 ml. of water.

The titration of the material prepared as above may be conducted in the flask placed over a white background or the solution may be transferred to a large porcelain dish. The flask, in the latter case, should be thoroughly washed and the washings added to the solution in the dish. Add the standard permanganate solution from a buret, with constant stirring, until the liquor assumes a faint pink color, which should disappear after three or four minutes' standing.

Make a similar titration of the solution prepared with the iron wire to ascertain the iron value of the permanganate solution. The percentage of iron in the char may be readily calculated from the data obtained in the two titrations.

Calcium Carbonate and Phosphates. The calcium carbonate may be determined by the methods on p. 664.

The percentage of phosphoric acid is of no particular significance, except when the spent char is to be sold on a basis of its fertilizing value. In this event, the customary methods of agricultural analysis are used.

Grist Test. Frequent determinations of the grain size of the boneblack are useful. A mechanical screen of the Ro-Tap type should be used, 100 grams being a convenient sample. Hardness tests on boneblack have been suggested by Knowles, Horne, and others based on the principle of determining the extent of grinding of the char in a ball mill under regulated conditions. The test is described by Knowles as follows:

> For purposes of test, the sample of boneblack is sieved to obtain the 16 × 24 fraction. One hundred grams of this fraction are sieved on 24-mesh and 50-mesh sieves for ten minutes, using a Ro-Tap mechanical sieve shaker. The portions passing the 24-mesh and 50-mesh sieves are weighed. The three fractions are reunited and put in a ball mill (turnip-type) with 10 steel balls ⅝ inch diameter. After rotating the mill for fifteen minutes, or for 450 revolutions, should the speed be somewhat different than 30 r.p.m., the char is removed and resieved. The total net loss on 24-mesh has been termed the

"shrinkage number," and the net gain through 50-mesh has been designated as the "discard number," it being assumed that in practice char finer than 50-mesh would be removed by screening.

Duplicate tests give results that seldom vary more than 0.5 for the shrinkage number and 0.2 for the discard number.

Weight per Cubic Foot. Both the loose and packed weights are required. These weights are calculated from the apparent specific gravity of the char under the two conditions.

Loose weight: Fill a tared 500-ml. flask loosely with char. The flask should not be shaken or tapped during filling. The weight of this char divided by that of an equal volume of water gives the apparent specific gravity of the sample. Calculate the weight as below.

Packed weight: Proceed as before except that the flask should be shaken and tapped constantly while filling until no more char can be added to the flask below the mark. Multiply the apparent specific gravities by 62.5 to ascertain the weights of a cubic foot of the char.

429. Thoroughness of the Revivification. The test of the efficiency of the revivification is of great value in the control of the kilns. Tests are made at very frequent intervals throughout the operating day on the char as it issues from the kilns.

To a measured volume of char add an equal volume of sodium hydroxide solution of 9° Brix. Heat the mixture to boiling for two minutes in a copper beaker, and decant the soda through filter paper or cotton into a test tube. A properly burned char will impart no color to the soda solution. A yellow or a brown color indicates a poor revivification (underburning), with the depth of the color directly in proportion to the amount of organic matter remaining in the black. An excess of sulfides will give a greenish cast to the solution.

René Baus[76] proposed the use of permanent standards made of acidified brom-thymol blue for the comparison of these tests. As modified since the publication, the standards are made as follows. To test tubes containing 35 ml. of 0.5 per cent acetic acid are added 0.2 ml., 0.4 ml., 0.6 ml., etc., up to 2.0 ml. of the 0.04 per cent brom-thymol blue used as pH indicator. These ten standards are numbered arbitrarily from 1 to 10, 1 being the lightest and 10 the brownest color. Soda tests are compared with these and the results may be reported numerically.

The pH of the water extract is possibly of greater importance than the soda test. (See work of Wayne, p. 325.) This is determined by shaking up the freshly burned char with neutral water, then filtering through paper or cotton that is free of acidity and determining the pH (generally on the phenolphthalein range). If the pH is above 8.4 (that is, pink to phenolphthalein) the alkalinity of the boneblack is satisfactory (though a pH as high as 9.0 is preferable). If below this the temperature of revivification should be raised,

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[76] *Ind. Eng. Chem.*, **19**, No. 11 (1926).

no matter whether the soda test shows colorless or not. Formerly, a pink test with phenolphthalein on the water extract of the char was considered as an indication of overburning, though many refiners recognized that moderate overburning in this sense was a virtue rather than a fault. Now it is known that there is little possibility in ordinary kiln work of reaching the limits of true overburning (pH of 9.6+).

430. Decolorizing Power. Laboratory filtrations for comparing the decolorizing and adsorptive powers of chars may be carried out in cylindrical

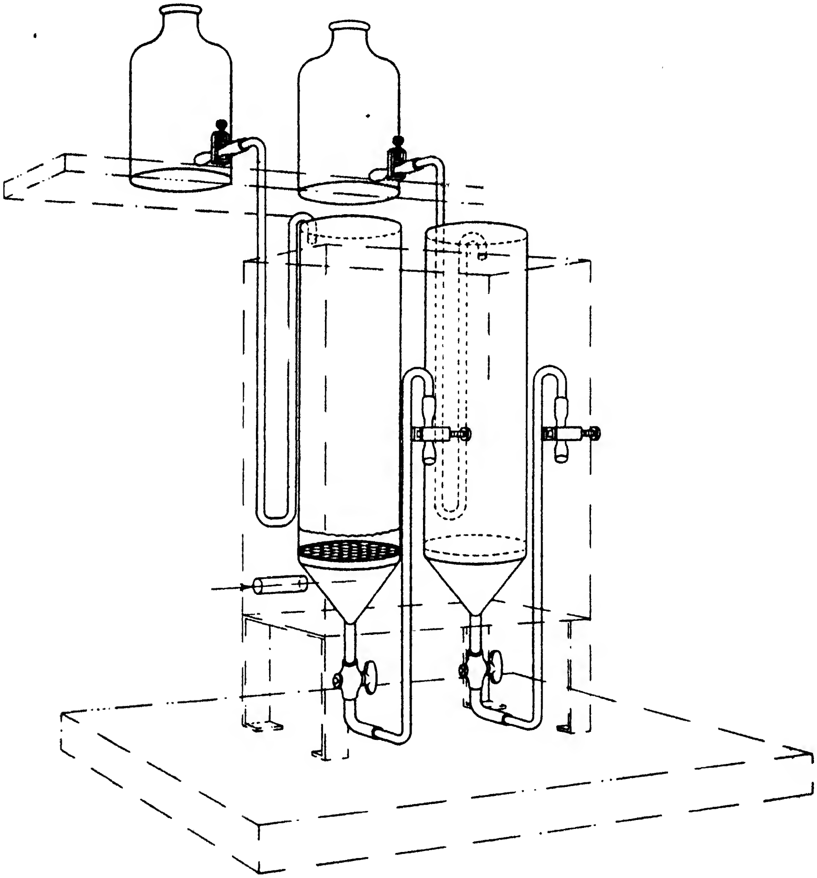

FIG. 107. Laboratory Boneblack Decolorization Apparatus.

copper funnels of convenient dimensions, e.g., 4 in. in diameter by 15 in. high. Each funnel should be provided with a small cock at the bottom. The char should rest upon a perforated copper plate covered with cloth. The funnels or filters should be immersed in a water bath provided with suitable openings for the outlet cocks, and should be filled to within a few inches of the top with the chars to be compared. The volume of the char should be the same in each of the filters.

A suitable solution for comparisons is prepared by dissolving raw or washed sugar to form a liquor of about 60° Brix clarified by filtration with kieselguhr or phosphoric acid and lime as in the refinery. The liquor should be adjusted to a fixed pH, say 7.5 or 8.0, as desired. This liquor should be heated to about 165° F. and equal portions of it should be added to each filter, little by little, as under service conditions, to avoid forming air pockets. After covering the char, the remainder of the liquor may be poured into the filter. Maintain the temperature of the water bath at 160°–170° for several hours and then draw off the filtered liquor from the outlet cocks. Compare the colors of the filtrates and unfiltered liquor and make complete analyses of each. The percentage absorption of color, ash, and organic non-sugars, as well as the improvement in purity and the change in the glucose ratios may now be calculated for each char, using the constituents of the unfiltered liquor as a basis. A supply of a good grade of boneblack should be kept as a standard of comparison, if many such tests are to be made. A convenient laboratory apparatus is shown in Fig. 107.

It is essential in all tests of this character that the conditions of the experiment shall be identical for all the samples of black to be compared. Certain points of the procedure must necessarily be arbitrary and the conditions can best be chosen to suit the particular requirements of the experiment. In so far as it is possible, factory conditions as regards temperature, ratio of char to sugar, density of the liquor, etc., should be maintained. Freshly burned black should be used in these tests as the adsorptive power changes on cooling and exposure to the air.

SUGAR ANALYSIS
AND CHEMICAL CONTROL OF THE FACTORY

CHAPTER 17

SUGARS AND OTHER CONSTITUENTS OF THE CANE AND ITS PRODUCTS

431. Sugars. In the manufacture of sugar from cane the sugars of importance in the analytical work are sucrose, dextrose, and levulose.

Sucrose, or cane sugar, is the most important of these. Dextrose and levulose, and possibly other reducing sugars, frequently grouped with them by cane sugar chemists under the name glucose, are of importance on account of their influence in analytical and manufacturing processes. The sugars are classed chemically as carbohydrates, i.e., they are chemical compounds of carbon with hydrogen and oxygen in the proportions found in water. One of the physical characteristics of the greatest importance to the sugar chemist is the property of the carbohydrates of rotating the plane of a beam of plane polarized light. (See Sec. 439.)

432. Sucrose, Saccharose, or Cane Sugar. This sugar is very widely distributed in the vegetable kingdom and in its pure state is the refined sugar of commerce. In a classification of the sugars it belongs to the disaccharides, derivatives of hexoses, and its formula is $C_{12}H_{22}O_{11}$. The commercial sources of cane sugar are the sugar cane, the sugar beet, the maple tree, and certain palms. The sorghum cane is often very rich in sucrose, rivaling the tropical cane, but the manufacture of sugar from this plant has not been a commercial success, though large quantities of table sirup are annually made from it in many parts of the United States.

Sucrose crystallizes in the monoclinic system, forming hemihedral anhydrous transparent crystals. The specific gravity of the crystals at 17½°/17½° C. is 1.58046 (Gerlach). Sucrose is readily soluble in water and in dilute alcohol. It is practically insoluble in absolute alcohol, ether, chloroform, and anhydrous glycerin. Dry sucrose, free of raffinose or other impurity, may be heated to a temperature of 120°–125° C. without discoloration. The melting point of sucrose crystals has been variously reported from 160° to 180° C. Sandera and Mircev[1] found large variations in the melting point of different dry sugars. They found that pure sugar had a melting point around 174° C.; that small amounts of invert sugar, dextrose, and moisture reduced the melt-

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[1] *Intern. Sugar J.*, June, 1934, p. 237 (abs.).

ing point, whereas small proportions of sulfated ash increased the melting point. They suggested that the melting point might serve as a means of judging the normal or abnormal nature of the non-sugars which may be present.

Sucrose, in common with many other substances, has the property of rotating the plane of polarization of a ray of light. It rotates the plane to the right and is termed dextrorotatory. This property is utilized as will be shown (Sec. 442), in the construction of polariscopic apparatus, for the analysis of sugars.

433. Hydrolysis or Inversion of Sucrose. It has long been known that sucrose when warmed with acids changes its chemical and optical character. The chemical change is expressed by the reaction

$$\underset{\text{Sucrose}}{C_{12}H_{22}O_{11}} + \underset{\text{Water}}{H_2O} = \underset{\text{Dextrose}}{C_6H_{12}O_6} + \underset{\text{Levulose}}{C_6H_{12}O_6}$$

This adding on of water gives rise to the term hydrolysis. The optical result is a change from right rotation to left rotation and on account of this reversal of the optical properties the process is termed inversion and the product, which is a mixture of equal parts of dextrose and levulose, is called invert sugar. Certain ferments (the commonest being invertase, present in yeast and in the saliva) have the same property of hydrolyzing or inverting sucrose. Sucrose itself is not directly fermentable, but after the inversion of the invert sugar is readily fermented.

434. Inverting Power of Acids. The inverting power of acids differs widely, as is shown by the following table[2] where the inverting power of hydrochloric acid is taken as 100.

Acid	Inverting Power, HCl = 100	Acid	Inverting Power, HCl = 100
Hydrobromic	111.4	Phosphoric	6.21
Hydrochloric	100.0	Citric	1.72
Nitric	100.0	Formic	1.53
Methylsulfonic	100.0	Malic	1.27
Sulfuric	53.6	Lactic	1.07
Oxalic	18.57	Acetic	0.40

The speed of inversion is closely proportional to the electrical conductivity of the acid and its chemical affinity. As has been shown by many investigators the pH of a solution is a direct measure, or rather, directly proportional to its inverting power. Raising the temperature greatly increases the inverting power. F. G. Stadler[3] worked out the relationship between the amount of sucrose inverted per hour at different temperatures and pH's. King and Jison[4] reproduced these figures in graphic form which is shown in part in Fig. 108.

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[2] Browne, *Handbook of Sugar Analysis,* second edition, p. 663.

[3] *Intern. Sugar J.*, July, 1932, p. 273.

[4] *Sugar News,* 1933, **14**, No. 1, p. 24.

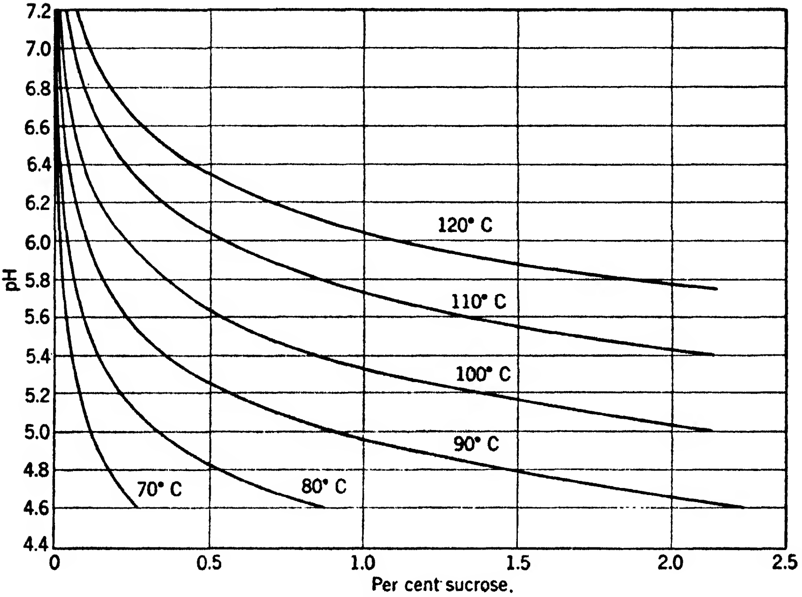

Fig. 108. Inversion of Sucrose per Hour at Different Temperatures and pH's.

435. Dextrose. This sugar is also called glucose or grape sugar and is widely distributed in the vegetable kingdom, where it is found in mixtures with other sugars. Its chemical formula is $C_6H_{12}O_6$ and it belongs to the monosaccharides (aldohexoses). Dextrose is always present in sugar cane, and when sucrose is inverted, it and levulose are formed in equal quantities.

Anhydrous dextrose forms rhombic crystals whose melting point is 144°–146° C., and the hydrate forms crusts or transparent crystals which melt at 80°–90° C. Dextrose is manufactured on a large scale and sold as corn sugar. It has a sweetness of about two-thirds that of sucrose.

Dextrose is readily soluble in water and in alcohol, the solubility in the latter varying with its dilution and temperature. It rotates the plane of polarization of light to the right, as the name would imply.

The chemical methods for the detection and estimation of dextrose, as well as other sugars, are based upon its property, in alkaline solution, of absorbing combined oxygen and reducing metallic oxides to lower oxides. The reaction utilized in analysis is the reduction of cupric copper in certain forms to insoluble cuprous oxide.

436. Levulose (Fructose). This sugar is usually associated with dextrose and sucrose in sugar cane. It is widely distributed and is also called fructose or fruit sugar. Levulose is a monosaccharide, hexose (ketohexose); it forms colorless, shining crystals of the rhombic system, which are hygroscopic and until recently were considered difficult to produce. In 1926

Jackson, Silsbee, and Profitt[5] published a method for producing it on a manufacturing scale from the Jerusalem artichoke (*Helianthus tuberosas*). Former investigators had reported levulose as having a needle-like crystal, but they found that when crystallized from a water solution, it always had the shape of a slightly tilted cube. Levulose is sweeter than sucrose, the relationship in sweetness being variously stated by different investigators from 103 per cent to 173 per cent that of sucrose. The crystals melt at 102°–104° C. The chemical formula of levulose is $C_6H_{12}O_6$. This sugar is very soluble in water and alcohol. It rotates the plane of polarization of light to the left, and is therefore termed levorotatory. In mature cane the quantity of levulose is small as compared with the dextrose. Both these sugars are sometimes present only in faint traces, and occasionally the levulose is absent.

Even though levulose is absent or present in very small quantity in the juice it always appears in large proportion in defecation-process molasses. This increase of levulose is not entirely due to inversion and may occur when no sucrose has been inverted. Such molasses will often have a low direct polarization and a very high true sucrose number.

The reappearance or increase of levulose is due to the action of alkalis and salts of alkalis with heat upon the dextrose. A part of the dextrose is converted into levulose. Likewise under similar conditions levulose may be isomerized and converted into dextrose. There are many references to this phenomenon in the chemical and technical journals.

437. Invert Sugar. When sucrose is acted upon by acids and certain other reagents it combines with water and is converted into a mixture of equal parts of dextrose and levulose. The sucrose is said to have been hydrolyzed or inverted. This mixture of sugars is called invert sugar.

The expressions invert sugar, reducing sugars, and glucose are frequently used synonymously in the cane sugar industry.

438. Abnormal Constituents of Sugar Cane Products.[6] There are a large number of compounds which, while not occurring normally in the juice of the cane, yet occasionally make their appearance in the cane and its numerous products, as the result of fermentation or of destructive influences during the process of manufacture. These abnormal products may be roughly classified as follows: first, sugars and closely related derivatives of the same; second, polysaccharides and related substances; third, acids; fourth, alcohols and esters; fifth, gaseous products. A few of these numerous compounds may be briefly mentioned:

Sugars and Closely Related Derivatives. Mannose, a fermentable hexose ($C_6H_{12}O_6$), has been reported by Pellet[7] in cane molasses from Egypt. Cane molasses also contains non-fermentable reducing substances that are

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[5] *Bur. Standards Sci. Paper 519.*

[6] This section originally prepared by Browne, at Spencer's request, has been checked and brought up to date by Dr. F. W. Zerban.

[7] *Rept. 5th Intern. Congr. Applied Chem.*, **3**, 383.

not precipitated by neutral lead acetate. These used to be considered identical with glutose, a hypothetical 3-ketohexose which has never been obtained in pure form or definitely characterized. (See Sec. 315.) The only nonfermentable sugar identified in molasses is *d*-allulose,[8] a 2-ketohexose closely related to levulose and allose. Mannose and allulose are produced by the action of alkalies upon dextrose and levulose, and consequently formed in the clarification of cane juice with excess lime.

Mannitol ($C_6H_{14}O_6$). This body is formed in considerable amount through the reduction of dextrose and levulose in certain fermentations of juices and sirups. Glycerol ($C_3H_8O_3$) and dimethylketol,[9] now generally known as acetylmethylcarbinol ($C_4H_8O_2$), have been reported in fermented molasses in small amounts.

Polysaccharides and Related Substances. Starch $(C_6H_{10}O_5)_n$. This substance, though practically absent in mature milling cane, occurs in small amounts in certain varieties, such as the Uba cane of Natal. It passes into the juice and leads to difficulties in clarification.

Dextran $(C_6H_{10}O_5)_n$. This gum, a most common and troublesome enemy of the sugar-maker, is the product of various fermentations, such as that produced by the Leuconostoc (Froschlaich), and is of frequent occurrence in canes injured by freezing or by insect ravages. The formation of this viscous gum in canes soon renders them worthless for milling. Its high specific rotation (+200), three times that of sucrose, introduces a serious error into the polarization of cane products, unless the gum is first removed by alcohol. Hydrolysis of dextran with acids gives dextrose.

Levan. A gum found by Smith[10] and Steel in sugar cane products in Australia and produced by the *Bac. levaniformans*. This organism was found to be very destructive in raw sugar; it causes a rapid inversion of sucrose and produces the slimy gum, levan, which has a specific rotation of −40. Hydrolysis of levan, with acids, yields levulose.

Cellulan. A gum found by Browne[11] in the tanks of a sugar house in Louisiana. It is formed in certain fermentations of cane juice and sirup, and consists of large leathery lumps insoluble in caustic alkali (distinction from dextran). The gum on treatment with boiling alkali yields a product giving all the reactions of cellulose (solubility in cuprammonium and blue coloration with zinc chloride and iodine), and on hydrolysis with acids is converted into dextrose.

Mannan. A gum found occasionally in the sedementary deposits of fermented juices and sirups. Hydrolysis of mannan, with acids, gives mannose.

Chitin $(4C_8H_{15}O_6N)_n$. This substance, which strictly speaking does not belong to the gums, was found by Browne[11] in large quantities in the scums of hot-room molasses in Louisiana. It is of fungoid origin and on hydrolysis with

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[8] Zerban and Sattler, *J. Am. Chem. Soc.*, July, 1942.

[9] Browne, *The Planter,* **34**, 237 (1905).

[10] *Intern. Sugar J.*, **4**, 430.

[11] *The Planter,* **34**, 238 (1905).

hydrochloric acid yields an amino sugar, glucosamine ($C_6H_{11}O_5NH_2$), and acetic acid.

Acids. Formic, acetic, proprionic, butyric, capric, and various other acids of the fatty series have all been found among the fermentation products of cane juices and sirups. In addition to the above, lactic acid should be mentioned as of very common occurrence in juices, sirups, and molasses. The latter acid may be formed either by the action of lime upon the sugars of the juice during clarification, or through the agency of various organisms, as *Oideum lactis* and *Bac. lacticus.*

Oxalic, glutaric, and other dibasic acids have also been reported as occurring in molasses in small amounts.

It has recently been shown by McCalip and Seibert[12] that aconitic acid is the most important acid constituent of cane products, constituting 80 or 90 per cent of the non-volatile acids.

Glucic acid has been reported by Browne[13] as formed in the alkaline clarification of cane juices. It is speedily broken down into formic acid and other decomposition products that are formed in the by-products of sugar manufacture, such as sirup and molasses. The formation of lead glucate and its rapid oxidation may possibly explain the reported occurrences of the spontaneous ignition of the precipitates obtained by clarifying sugar solutions with basic lead solutions.

Alcohol and Esters. Ethyl, amyl, butyl, and other alcohols have been found in the fermented products of the sugar cane. The combination of these alcohols with the various acids, acetic, butyric, etc., gives rise to numerous esters, the fruity odor of which is characteristic of many fermented cane products. Ethyl sulfite and sulfide have also been found in fermented juices which have undergone sulfitation, and the objectionable odor (tufo) of these compounds is frequently noticeable in the spirits from tropical distilleries.

Gaseous Products. Carbonic acid (CO_2) is nearly always given off by cane products undergoing fermentation. In many fermentations a reduction takes place and in such cases hydrogen may be evolved. The explosion of hydrogen generated from the sour water, left standing in vacuum pans, has occurred at times, and on one occasion with disastrous effects.[14]

Another product in the reducing fermentation of juices which have been sulfured is hydrogen sulfide (H_2S), and the odor of this gas from sulfured juice or sirup which has been left standing a long time is sometimes highly objectionable.

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[12] *Ind. Eng. Chem.*, May, 1941, p. 637.

[13] *Intern. Sugar J.*, February, 1938, p. 30.

[14] *Louisiana Planter,* 5, 243 (1890).

CHAPTER 18

OPTICAL METHODS IN SUGAR ANALYSIS

439. Polarized Light. Ordinary light such as sunlight or light from any radiant source vibrates in all directions at right angles to the direction of the beam of light. A beam of ordinary light is therefore made up of vibrations which proceed in an infinite number of planes. By means of various optical devices it is possible to cause a beam of light to vibrate in one plane only and such light is said to be polarized or more specifically plane polarized, and the plane in which it vibrates is called the plane of polarization.

Many substances, including solutions of sucrose and other sugars, have the power to rotate the plane of polarization. The angle through which the plane of polarization is rotated may be measured by suitable optical instruments and it is the measurement of the rotation which is employed for analyzing sugars by optical methods.

Confining our remarks to the three sugars of most interest to the cane sugar chemist, it should be noted that sucrose rotates the plane of polarization to the right and is therefore termed a right-hand, or dextrorotatory, sugar. Dextrose also rotates the plane to the right. Levulose rotates the plane to the left and is levorotatory, or a left-hand sugar. Quartz is of two kinds, right and left hand, and is used in certain types of polariscopes to compensate for the rotation due to sugar.

440. Specific Rotatory Power. The power of a sugar to rotate the plane of polarization differs for different sugars. The number expressing this is termed the specific rotation or specific rotatory power, and is the rotation in angular degrees which a calculated 100 per cent solution of the pure substance 100 mm. long gives to the plane of polarized light. The expression $[\alpha]$ is used to indicate specific rotation and as it depends upon the wavelength of light employed and the temperature at which it is taken these must be specified. The bright yellow line of sodium light, known as the D line, is the light almost universally used and the standard temperature is 20° C.; the symbol for specific rotatory power under these conditions being $[\alpha]^{20}_{D}$.

It will be seen that when a ray of polarized light is passed through a solution of any given sugar the amount of rotation varies with the strength of the solution, the length of the column, the nature of the light, and the temperature. By having a fixed length of column, standard temperature, and class of light the rotation then becomes a function of the concentration of the sugar in the solution.

These optical properties of sugars, Iceland spar, and quartz are utilized in the construction of the polariscope, and keeping them in view the descriptions of the various types of instruments, as given farther on, will be readily understood.

441. Nicol Prism. The polarization of the ray of light is accomplished by passing it into a Nicol prism. This prism is made from a rhombohedron cut from a transparent crystal of Iceland spar. The end surfaces of the prism, Fig. 109, are ground off so that each acute angle is 68°. The prism is then cut into two parts, *A* and *B*, through the obtuse angles, and the surfaces are polished and cemented together again, in their original positions, with Canada balsam. Other forms of the prism are used, but this description will answer for the present purposes.

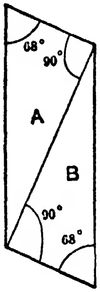


FIG. 109. Nicol Prism.

On passing a ray of light into this prism it is separated into an ordinary ray, which is reflected from the prism by the balsam cement, and an extraordinary or polarized ray, which passes through the polariscope and upon whose properties the construction of the instrument depends.

Every polariscope contains two Nicol prisms, the polarizer and the analyzer, which together make up the essential feature of the instrument.

APPARATUS AND MANIPULATIONS

442. The Polariscope. The instrument used in the quantitative estimation of sugars by the optical methods is usually called a polariscope. The names polarimeter and saccharimeter are also sometimes used.

Polariscopes were formerly of two classes, viz., shadow and transition tint instruments, but the tint polariscopes have long been obsolete. The shadow instruments may be divided into polariscopes using white light, as from an electric lamp, and those requiring a monochromatic light, the yellow ray from a sodium lamp.

Up to very recently all commercial instruments used an ordinary white-light source and the instruments first described below are of this class.

A brief description of the polariscopes in general use will be given in the following paragraphs and will be sufficient for the purposes of this book. The reader is referred to *Bureau of Standards Circular C 440,* "Polarimetry, Saccharimetry and the Sugars" (1942), for more detailed descriptions of the theory and construction of the instruments.

443. Half-Shadow Compensating Polariscope (Schmidt & Haensch). The optical parts of this instrument are indicated in Fig. 110. The polariscope shown is of the single-compensation type.

At *P* there is a slightly modified Jellet-Cornu Nicol prism, at *L* is a plate of levorotatory quartz, at *E* is a quartz-wedge movable by means of the screw

A, and at *D* is a quartz wedge, fixed in position, to which is attached the vernier. The scale upon which to note the distance the quartz wedge *E* has been moved, in compensating for the rotation of the plane of polarization due to interposing an optically-active body, is attached to the wedge. The scale is graduated, for technical work, to read percentages of cane sugar. These quartz wedges are of dextrorotatory quartz.

The parts *D, E,* and *L* constitute the compensating apparatus, i.e., that compensates for the deviation of the plane of polarization, as explained above. The substance to be examined, dissolved in a suitable solvent, is placed in the observation tube, as shown in the figure. At *H* is the analyzer, a Nicol prism; at *F* is the telescope used in observing the field, and at *M* are the telescope and reflector for reading the scale. The two lenses at the extreme right are for transmitting the rays of light from the lamp in parallel lines to the Nicol prism, forming the polarizer.

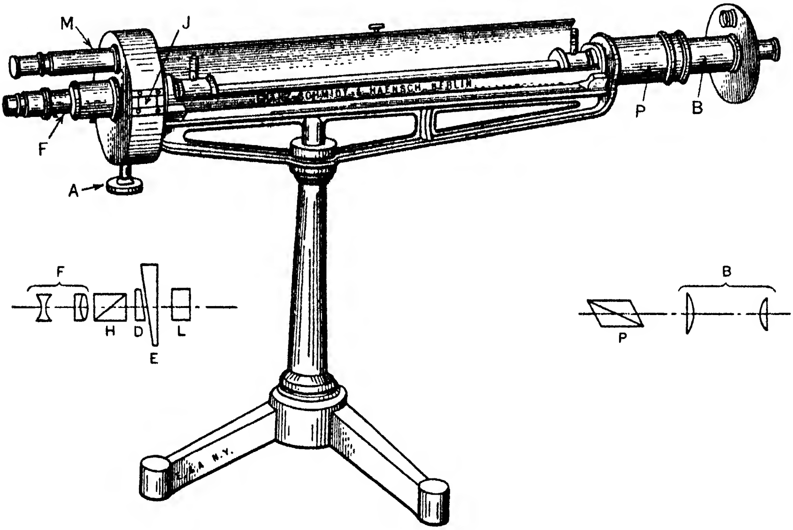
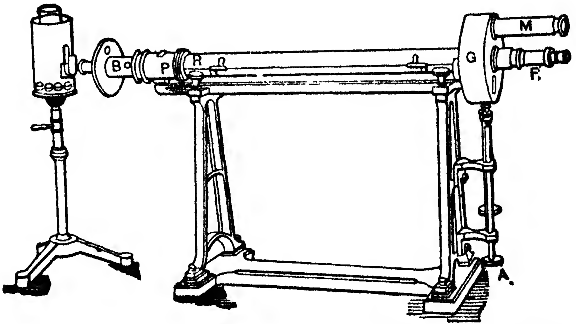


FIG. 110. Schmidt and Haensch Single-Compensating Polariscope.

The instrument described above is of the single-compensation type. A double-compensating instrument is shown in Fig. 111. The polariscope differs from that already described in having two sets of quartz wedges of opposite optical properties and two scales and verniers. The arrangement of double-compensating wedges is shown in the optical parts of the Bates-Frič instrument, Fig. 114.

The field of vision of the above instruments, when set at the neutral point, is a uniformly shaded disk. If the milled screw, controlling the compensating wedge, is slightly turned to the right or left, one-half of the disk will be shaded and the other light. It is from this half-shaded disk and the compensating wedges that this instrument takes its name.

The optical parts of all modern instruments are protected from exposure by the cap *G*, Fig. 111, and by plain glass plates. This protection of the optical parts is especially important in the tropics, where, owing to some peculiar climatic condition, the lenses often become coated with an opalescent film that can be removed only by polishing. These plates also prevent minute spiders and other insects in the tropics from damaging the instrument. The scale is lighted from the lamp, the light being reflected by means of a prism and mirrors. The telescope *F* is focused by a screw. In the more modern instruments the screws for adjusting the position of the quartz wedges have long stems so that the hand of the observer may rest upon the table. A glass cell filled with a 3 per cent solution of bichromate of potassium is placed at *B* and serves as a ray filter (see Sec. 448). This ray filter should always be used, except for dark-colored solutions. The heavy trestle support of Fig. 111 is preferable to the tripod form of Fig. 110 and is used in most modern polariscopes.



Schmidt and Haensch.

FIG. 111. Double-Compensating Polariscope.

444. Half-Shadow Polariscope with Glass Scales (Josef Frič and Jan Frič). The instrument shown in Fig. 112 has single-wedge compensation for use with white light. The optical parts are enclosed in a metal case for protection from dust. The scales and verniers are engraved on glass and are lighted by a part of the polarized rays which are reflected upon them.

Messrs. Frič also make a quartz-wedge polariscope with adjustable sensitivity (Fig. 113), designed for the U.S. Bureau of Standards by Frederick J. Bates. This instrument is double quartz wedge compensating and has a Lippich polarizing system. Readings may be accurately made to 0.01° sugar. The Frič instruments were among the first to be graduated according to the specification of the International Commission at 20° C. for use with the normal weight of 26 grams with the 100-true-cc. flask at 20° C. All makers now conform to these specifications. The optical system of the Bates-Frič

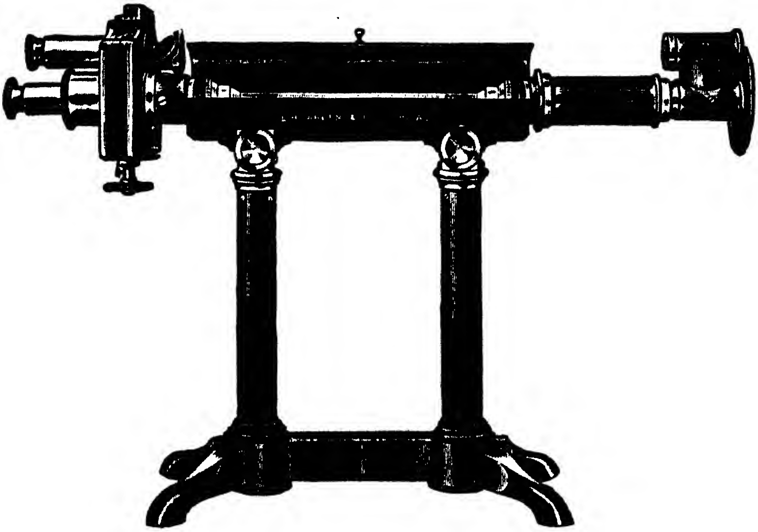


FIG. 112. Frič Single-Compensating Polariscope.

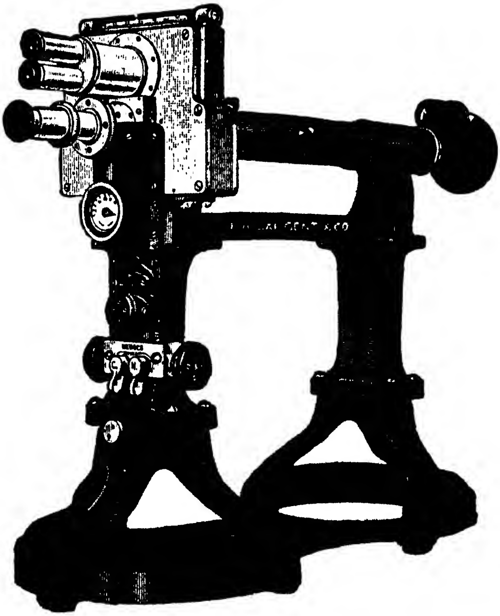


FIG. 113. Bates-Frič Polariscope.

is shown in Fig. 114 and illustrates the arrangement of the quartz wedges in a double-wedge compensating polariscope. A full description of the Bates-Frič instrument is given in *Bureau of Standards Circular C 440*, pp. 70–72.

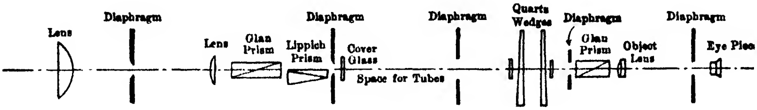


FIG. 114. Optical System of Bates-Frič Instrument.

445. Half-Shadow Polariscope (Julius Peters). This instrument, Fig. 115, is double compensating. It is similar in principle to the other compensation polariscopes. The optical parts are protected by glass plates from the action of the atmosphere. A dust cap, not shown in the figure, protects the optical parts at the front end of the instrument.

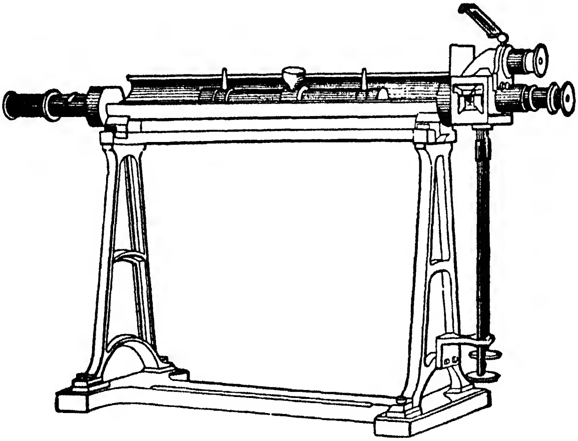


FIG. 115. Peters Double-Compensating Polariscope.

The quartz wedges are not mounted in brass as is usual, and it is claimed are not exposed to pressure or strain with the expansion of metal. As is well known, the rotation of quartz is changed by pressure.

446. Triple-Field Polariscope (Schmidt & Haensch). This instrument differs from those already described in having two small Nicol prisms placed in front of the polarizer, as shown in Fig. 116. The field of the instrument is divided into three parts, 1, 2, and 3 of the diagram. This figure shows the arrangement of the Nicol prisms (I, II, III), and a diagram of the field. When the instrument is set at the neutral point, the field is uniformly shaded; in other positions 1 is shaded and 2 and 3 are light or vice versa.

This arrangement permits a very high degree of accuracy in the adjustment of the field, in polariscopic observations, provided that the instrument is in perfect adjustment. It has been found, however, that minor differences in the two outer sections of the field are likely to develop and that the observer compares the central division with one of these sections only, making in effect only a double field. The triple-field type for this reason has not found general favor.

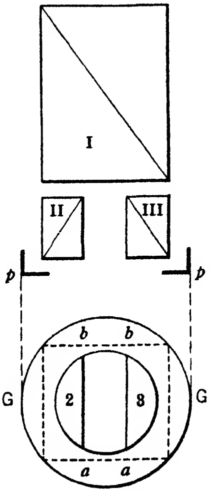


Fig. 116. Prisms in Triple-Field Polariscope.

447. Bausch and Lomb Polariscope. This instrument was designed after obtaining the opinions of many prominent sugar chemists as to the requirements for a high-class polariscope for industrial use, and it embodies several new features for convenience and cleanliness as well as for accuracy. It is single-wedge compensating with a scale range from −30° to 110° and is equipped with either the Lippich double prism polarizer or the Jellet single-prism type. The first system is very sensitive, and the dividing line is practically invisible, but it is fragile and subject to disintegration, while the Jellet polarizer is less sensitive but not so easily put out of adjustment. The Jellet single-prism system is advocated for industrial laboratories. The diagram of the optical parts with Jellet polarizer is shown in Fig. 117, this figure illustrating in considerable detail the construction of a single-wedge polariscope. The scale is of glass, with the vernier and scale lines overlapping, which facilitates exact reading and the estimation of fractions of tenths. The scale field and the polarized field are illuminated from the same light source, and the light to the scale passes through a filter similar to that used in the observation field, making both fields of the same color and intensity. In place of the bichromate cell is a ray filter of glass having the same optical properties, and which may be thrown out of the field by a push rod when dark-colored solutions are read.

448. Bichromate Light Filter. For all accurate work with polariscopes employing white light a bichromate filter must be used. This consists in most instruments of a solution of potassium bichromate of such a strength that the percentage of bichromate times the length of the cell equals 9; e.g., a 3-cm. cell should contain a 3 per cent solution. Some polariscopes employ a polished crystal of bichromate for this purpose but such crystals are not stable. A glass filter having the same optical properties as the standard bichromate cell is used in some instruments, and these glass filters may also be purchased from laboratory supply houses for use in those instruments which normally use the solution as a filter.

According to Schönrock the use of a bichromate ray filter makes a difference of 0.12° in polarization with a white light instrument. With the filter he obtained a reading of 100° and without it 100.12°.[1]

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[1] *Bur. Standards Circ. 44; Z. Ver. deut. Zuckerind.,* 1904, pp. 521–558.

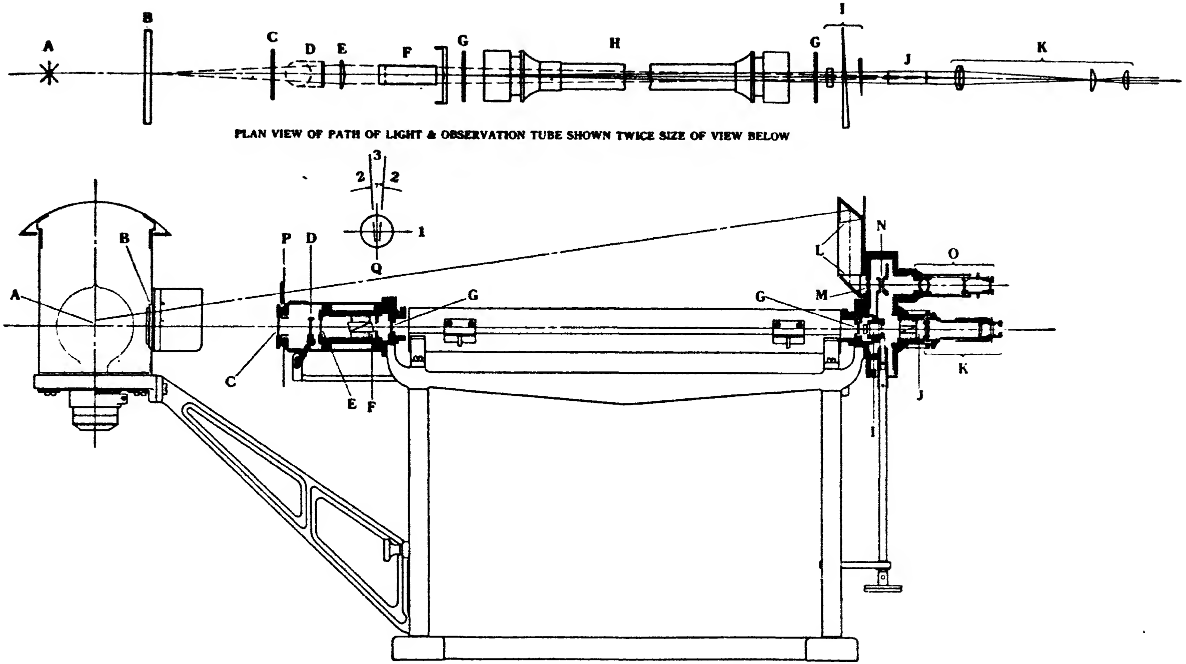

FIG. 117. Diagram of Bausch and Lomb Polariscope.

A, Light source; *B*, Ground glass lamp window; *C*, Cover glass; *D*, Glass filter replacing bichromate cell; *E*, Condensing lens; *F*, Polarizer; *G*, Splash glass; *H*, Observation tube; *I*, Quartz compensation; *J*, Analyzer; *K*, Observing telescope; *L*, Scale illuminating prism; *M*, Condensing lens; *N*, Scale and vernier plates; *O*, Scale reading eyepiece; *P*, Glass filter in scale-illuminating system; *Q*, Photometric field as seen from eyepoint; 1, Plane of polarization of analyzer; 2, Planes of polarization of semifields; 3, Half-shade angle.

449. Mutarotation. An important property of all optically active reducing sugars is that of mutarotation, or birotation. A freshly prepared solution of such a sugar undergoes a rapid change in polarizing power, the change becoming more gradual until finally after a few hours the polarization reaches an equilibrium. A dextrose solution possesses about twice the rotation when first prepared that it does after it reaches a constant reading. As this was the first sugar in which the change was observed, the phenomenon was termed birotation because of this 2:1 ratio. It has since been found to exist in different degrees in other sugars, so the modern term mutarotation was substituted. (In xylose, for example, the ratio of polarization of a freshly prepared solution to final constant polarization is 4.5 to 1.) Heating the solution will hasten the change to constant rotatory power.

This phenomenon is of considerable importance in the analysis of inverted sirups, honey, and other products high in reducing sugars and may have an effect in cane molasses, or even in deteriorated raw sugars. For this reason successive readings of such substances must be taken until constant readings are obtained.[2]

450. Photoelectric Polariscope. The use of photoelectric cells to measure the intensity of light in the two halves of the polariscopic field has been considered a possibility for many years and a number of arrangements have been suggested[3] but so far no commercial instrument has been developed using this device. An arrangement using two parallel polariscopes is outlined by Singh and Anantha Rao[4] in which distilled water is placed in the tube in one side of the instrument, and the solution to be polarized in the other side. The quartz-wedge compensator in the one containing the test solution is then moved until the light falling on the two photoelectric cells again is brought into balance by moving the appropriate compensating wedge. Scale readings are taken with the tube of test solution in each side of the instrument so that errors due to wall thickness of tubes, light source, etc., are eliminated.

451. The Electrical Sodium Lamp in Saccharimetry. The polariscopes so far described employ ordinary white light which in the past twenty-five years has been supplied by special stereoptican electric bulbs of the concentrated filament type. The use of monochromatic light greatly simplifies the instrument, as this eliminates the need for the quartz-wedge compensation system (Fig. 110). The sodium lamp with a gas flame has been used in polarimeters reading in circular degrees, but its use has been restricted to research work. An electric sodium lamp has recently been invented giving an excellent monochromatic source of light which is from twelve to thirty times more intense than that from a salt-burning sodium gas lamp. Landt[5]

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[2] See Browne-Zerban, *Sugar Analysis,* third edition, 1941, pp. 283–293, for a detailed study of mutarotation; also *Bur. Standards Circ. C 440* (1942), pp. 439–456.

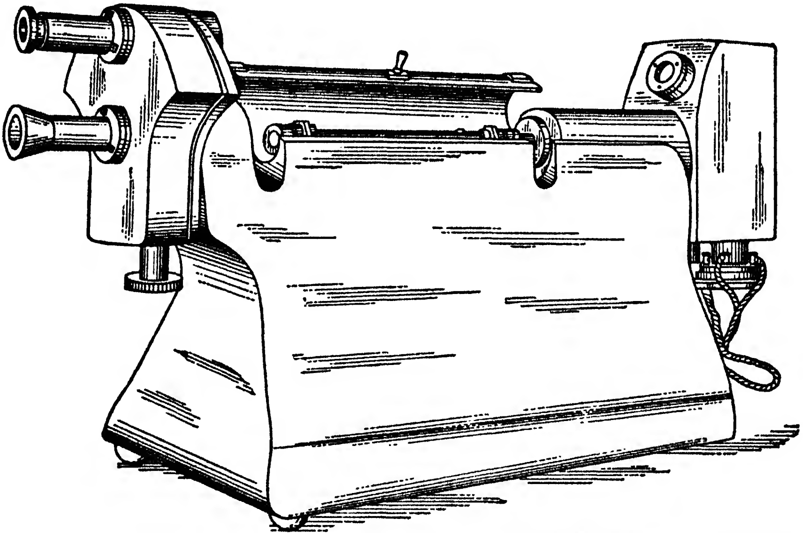
[3] *Intern. Sugar J.,* February, 1939, p. 67 (abs.).

[4] *Intern. Sugar J.,* February, 1939, p. 67 (abs.).

[5] *Intern. Sugar J.,* December, 1935, p. 474.

discusses the use of this lamp with a polarimeter and suggests that this will serve as a saccharimeter by engraving the circular scale in Ventzke degrees, corrected to 34.613 circular degrees for the 100° point, in accordance with the international scale (Sec. 455). The advantages outlined by Landt are: (1) greater accuracy than with the quartz-wedge compensating polariscope because a smaller half-shadow angle may be employed in the polarimeter; (2) the half-shadow angle is readily adjustable for sensitivity, a refinement that is obtainable in only the most expensive make of polariscope (see Sec. 444); (3) the influence of temperature is much smaller in the saccharimeter and adjustment to 20° may be done with water-jacketed tubes instead of with constant temperature rooms; (4) the polarimeter is optically much simpler.

452. Bellingham and Stanley Saccharimeter with Sodium Lamp. A polarimeter employing the new electric sodium lamp which is made especially for sugar work is illustrated in Fig. 118. It is purposely made to have the same general design and appearance as a quartz-wedge polariscope with the same type of scale, the same method of reading, and the same actuating mechanism.[6]



Courtesy Browne-Zerban, Sugar Analysis.

FIG. 118. Bellingham and Stanley Saccharimeter without Quartz-Wedge Compensation.

Improved accuracy, lower price, and greater optical simplicity indicate that instruments of this type may ultimately displace the quartz-wedge compensating type of polariscope.

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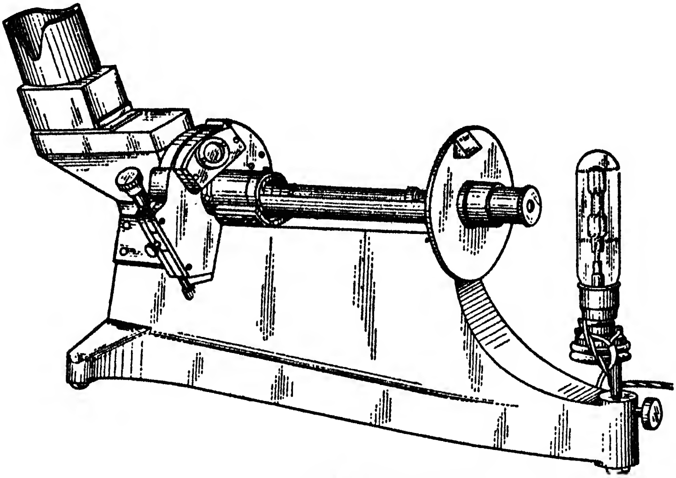
[6] Stanley, *Intern. Sugar J.*, September, 1939, p. 348.

453. Projection Saccharimeter. Among the several novel features which this instrument[7] embodies the principal one is that the scale reading is projected on a ground glass with sufficient magnification so that no telescope is needed (Fig. 119). By placing his eyes at the hood on the left-hand side of the instrument, the observer sees the usual half-shadow disk, and also in the hood the projection of the sugar and scale and vernier. No dark room or polariscope box is necessary. The observation end of the instrument is turned at a right angle to make manipulation of tubes more convenient. Like the saccharimeter previously described, made by the same firm, the use of the electric sodium lamp eliminates the quartz-wedge compensation feature.



Courtesy Browne-Zerban, Sugar Analysis.

FIG. 119. Bellingham and Stanley Projection Saccharimeter without Quartz-Wedge Compensation.

454. The Polariscopic Scale. Ventzke Scale. The Normal Weight. Polariscope scales are divided to read either circular degrees or percentages of cane sugar or both. Commercial instruments usually have only the Ventzke or cane sugar scale.

The Ventzke or cane sugar scale is so divided that, if a certain weight of the material is dissolved in water and the solution diluted to 100 cc. or ml. and observed under standard conditions in a 200-mm. tube, the reading will be in percentages of cane sugar, or if pure sucrose is used, the reading will be 100. (See reference to Bates-Jackson scale below.) The weight required under these conditions to give percentage readings is termed the normal weight of the instrument. In commercial work, especially in the polarization of sugars, the divisions of the cane sugar scale are usually termed degrees.

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[7] Bellingham, *Intern. Sugar J.*, 1938, p. 433.

The normal weight for most instruments now in use is 26 grams, but this is subject to modification as is shown below. The flask used with this normal weight must hold 100 true cc. (ml.)[8] of solution at 20° C. The solution must be prepared and the observation be made at 20° C. to secure the greatest accuracy, or correction for temperature must be made (Sec. 479).

The relationship between the older normal weight of 26.048 dissolved in 100 Mohr's ml. at 17½° C. and the present one of 26 grams in 100 ml. (true cubic centimeters) at 20° C. is extremely close. Exact mathematical correspondence would have been secured if 26.0082 grams[9] had been chosen instead of 26 grams, but this difference of less than 10 mg. is quite evidently negligible in polariscopic work.

455. International Sugar Scale. In 1916 Bates and Jackson[10] published a report of experiments with pure sucrose repeatedly recrystallized from water *in vacuo* showing that the polarization of 26 grams of sucrose in 100 ml. of water at 20° C. was 99.895 on the Ventzke scale. This new value has been adopted by the Bureau of Standards in the standardization of quartz plates and saccharimeters, and because of this, government polarizations, e.g., for duty purposes, are always about 0.1° higher than those made on the same sugars in commercial or industrial laboratories.

The designation "international" has been objected to[11] for this scale as it is by no means universally used and also because the term international was used as early as 1896 by Sidersky and Pellet for the 20-gram or bidecimal normal weight, and it is so referred to in much of the literature.

Browne and Zerban[12] corroborated the figures of Bates and Jackson in work done at the Bureau of Chemistry and the New York Sugar Trade Laboratory respectively, finding that "100° point" on the Ventzke scale is actually 99.90. They conclude that:

> . . . in working with refined sugars, high-grade sirups, and other products of high purity, where there are no counterbalancing plus errors due to the volume of lead precipitate and to other causes, it would be more accurate to employ a normal weight of 26.026 grams than the present weight of 26 grams. With products of low purity, however, where the counterbalancing plus errors may equal or exceed the minus error due to the graduation of the scale, the introduction of a corrected normal weight or of a correction factor will increase instead of diminish the errors of observation.

On the basis of these and other similar findings, the International Commission for Uniform Methods of Sugar Analysis at Amsterdam in 1932, ruled as follows:

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[8] The Bureau of Standards has requested that the true cubic centimeter be called a milliliter (ml.) and this is followed in this book wherever precise measurements are specified.

[9] Browne-Zerban, *Sugar Analysis*, third edition, 1941, p. 178.

[10] *Bur. Standards Sci. Paper 268.*

[11] Browne-Zerban, *Sugar Analysis*, third edition, 1941, p. 179.

[12] *J. Assoc. Official Agr. Chem.*, **11**, No. 1 (1928).

> In the case of existing instruments graduated on the Herzfeld-Schönrock scale (i.e., Ventzke Scale) it shall be permitted either to change the saccharimeter scale or to use a weight of 26.026 grams in 100 ml.

However, this ruling was still further modified by the Commission at the meeting in London in 1936. As will be explained in Sec. 473, the volume of the lead precipitate when using lead subacetate solution for clarification causes an increase in the polarization of a 96° raw sugar of about 0.1°. The two errors, that due to the difference between the older Herzfeld-Schönrock scale and the new international scale, and that due to the volume of the lead precipitate when using lead subacetate solution, therefore about offset each other. To correct one error without simultaneously correcting the other is to leave the result worse than before. Therefore the Commission in 1936 ruled as follows:

> If no change in the sugar scale is or has been made, clarification shall be effected with standard lead subacetate solution (Third Session of International Commission, Paris, 1900); but if a change from the Herzfeld-Schönrock scale to the International Sugar Scale is made, then clarification shall be effected with standard dry lead subacetate (Horne's Dry Lead, U.S. Patent).

To summarize the above: If dry lead subacetate is used for clarification of solutions for polarizations, use a normal weight of 26.026 grams *provided* the polariscope has the Ventzke scale. If lead subacetate solution is used for clarification the normal weight of 26.000 grams should be used with such an instrument.[13] On the other hand, if the instrument is equipped with the international scale, or if Bureau of Standards standard quartz plates calibrated to the international scale are used, then dry lead for clarification is mandatory and the normal weight of 26.000 grams must be used. In order to avoid confusion with the various normal weights, the old Mohr unit of 26.048 grams is made round; the 26.026 gram normal weight is hexagonal, and the 26.0 gram weight is square.[14]

A point to be remembered is that the above applies to raw sugars where the compensating error of the scale and the volume of the lead precipitate are both in effect. For high-test sugars requiring no clarification, the 26-gram normal weight gives an error of minus 0.1 with the Ventzke scale, i.e., a pure sugar polarized with the 26-gram normal weight, and the Ventzke scale will read 99.9° instead of 100°. Therefore for sugars requiring no clarification the normal weight should be 26.026 grams, always provided that the Ventzke scale is actually used and not converted to the international scale by the use of Bureau of Standards quartz plates. Because ultimately all polariscopes will be fitted with the international scale which requires the 26.000-gram normal weight, the simplest correct procedure would seem to be calibration of all instruments by means of Bureau of Standards quartz

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[13] This does not refer to the use of dry lead subacetate for routine purity determinations (see Sec. 660), but to its use for clarifying solutions for polarizations (see Sec. 475).

[14] Browne and Balch, *Ind. Eng. Chem., Anal. Ed.,* 1933, No. 9, p. 283.

plates;[15] the employment of the 26.000-gram weight and then the use of dry lead for clarification wherever lead is needed. In line with this procedure all tables in this book have been calculated to the 26.000-gram normal weight.

456. Twenty-Gram or Bidecimal Normal Weight. French Normal Weight. In 1896 Sidersky suggested 20 grams as the normal weight, and some French polariscopes and a few other makes were built with a scale for this normal weight. There are valid arguments for the use of a bidecimal normal weight and it is used to some extent in Australia, Fiji, Mauritius, and Egypt.[16]

The French scale as officially adopted in 1896 has a normal weight of 16.29 grams, but this has recently been corrected to 16.267 which brings the French scale and international scale into exact agreement.[17]

457. Manipulation of a Polariscope. Methods of preparing the solutions will be described in Sec. 462. Having dissolved and clarified the normal weight of the material to be polarized, fill an observation tube with a portion of the solution and place it in the trough of the instrument, and pass the light from a suitable lamp through it.

The observer with his eye at the small lower telescope of the instrument will notice that one-half of the disk is shaded more than the other, provided the instrument is not set at the neutral point. The vertical line separating the half-disks should be sharply defined, and if not, the ocular should be moved backwards or forwards until a sharp focus is obtained.

The observer should now turn the milled screw, which moves the quartz-wedge compensator, until the field appears uniformly shaded, then read the scale as directed in Sec. 458.

In making the observation the eye should be in the optical axis of the instrument, and should not be moved from side to side.

A little practice will enable the operator to detect very slight differences in the depth of the shadow and to attain great accuracy in this manipulation.

The manipulation of the triple-field polariscope is as described above, except that the field is in three sections.

The double compensating polariscopes are provided with two scales, in the older instruments, one graduated in black and the other frequently in red. The black scale is operated by the black screw and the red by the brass one. For ordinary work the red scale is set at zero, and the field is equalized with the black screw.

To make an invert reading, i.e., one with a levorotatory or left-hand sugar, the black, or right-hand, scale, should be set at zero and the other scale used.

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[15] The *Bur. Standards Circ. C 440,* 1942, p. 80, also recommends this procedure and calls attention to the fact that no inconvenience is experienced as most instruments require some correction anyway because of scale inaccuracies, etc.

[16] See Browne, *Intern. Sugar J.,* December, 1936, p. 467.

[17] See Bates, *Proc. 9th Session Intern. Commission Uniform Methods of Sugar Analysis,* London, 1936, p. 28 S.

The readings should be recorded with the algebraic sign minus. With the single-compensating instruments the direct and invert readings are made on the same scale, the graduations extending both sides of the zero.

Metal or glass scales are used in modern instruments. The overlapping glass scale of the Bausch and Lomb polariscope described previously is a comparatively recent refinement.

458. Reading the Scale. Having equalized the field of the polariscope as already described, the scale is to be read. The method of reading the scale is best shown by an example. Let the position of the scale and vernier be as shown in Fig. 120. The zero of the vernier is between 30 and 31 of the scale; record the lower number and note the point at which a line on the vernier corresponds with one on the scale, that is, if extended would coincide with the line on the scale, in this case at 7 of the vernier. Enter this number in the tenths place and the completed reading is 30.7. If the zero of the vernier falls directly opposite a line of the scale, the reading is a whole number. The divisions to the left of the zero of the vernier in single compensating instruments are for making invert readings.

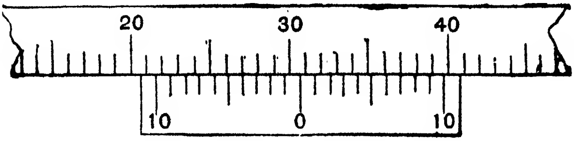

FIG. 120. Diagram of Vernier Scale.

If the normal weight of sucrose, or of a substance containing sucrose and no other optically active substance, has been dissolved in water and diluted to a volume of 100 ml., and a 200-mm. observation tube has been used, and the observation is made at the temperature at which the instrument was standardized (now generally 20° C.) the reading on the sugar scale is the percentage by weight of sucrose in the material. The readings must be corrected for other weights than the normal, for other volumes than 100 ml. and for other tube lengths than 200 mm.

459. Polariscope Lamps. Compensating half-shadow instruments require a strong white light.

The most efficient polariscope illumination in a factory having twenty-four-hour electric service is the nitrogen-filled concentrated-filament Mazda lamp of 100 watts capacity. This light is so very intense that a ground-glass plate should be interposed in polarizing light-colored solutions.

Considerable momentary variation may occur in the voltage of factory current which may cause appreciable change in the intensity of the light and, consequently, errors in the polariscope readings. This may be obviated by having a rheostat and voltmeter located in the lamp circuit and reducing the voltage to a fixed point with the rheostat, e.g., 100 volts for a 110-volt current. The voltmeter and rheostat should be located in full view of the operator (the upper, forward part of the polariscope compartment is convenient) in order that the voltage may be accurately adjusted for each reading.

Storage batteries and automobile headlight lamps are practicable where direct current is available for recharging. The Edison storage battery is simple to charge and requires little attention. The storage battery gives a fixed voltage and a good light, but the ordinary current with adjusted voltage as described above is preferable.

Acetylene lamps provide a strong and convenient source of light, probably next to the concentrated-filament lamp in efficiency. The Welsbach lamp, burning gas, alcohol, or gasoline, also gives a good light.

Messrs. Schmidt and Haensch equip many of their instruments with small electric lamps operated from small secondary batteries. The lamp is provided with a special socket attached directly to the instrument. This arrangement of the lamp is not entirely satisfactory in practical experience. Electric and acetylene lamps and alcohol or gasoline Welsbach lamps are easily obtainable on plantations and should be given preference in the order named over kerosene light.

It is quite essential that the position of the light with respect to the polariscope be fixed. The intensity of the light should be as constant as possible, and if changed, the zero reading observation should be verified. It is advisable to follow the maker's directions as to the position of the lamp with regard to the instrument and arrange the latter so that it cannot be jarred out of place. The distance of the lamp from the instrument is usually 15 to 20 cm. The position the lamp should occupy should also be marked, that it may be properly replaced if disturbed, and the intensity of the light should not be changed after adjusting the instrument until the observation has been made. A change in the position or intensity of the light, with certain instruments, will sometimes cause an error of 0.5° or more. The use of the electric sodium lamp with certain recently introduced instruments has been described (Sec. 451).

460. Adjustment of the Polariscope. The scale of the instrument is the only part likely to get out of adjustment. To test this adjustment, place a polariscope tube filled with water in the trough of the instrument and make an observation. This observation may be made without the tube, but the adjustment is more readily made with the water. If the scale is properly adjusted the reading should be zero.

The method of adjusting is the same for all modern compensating polariscopes. A micrometer screw, generally placed at the right-hand side of the dust cap enclosing the forward optical parts of the instrument and turned by means of a key, is arranged to move the vernier a short distance. The field is equalized as usual, but with water in the observation tube, and the vernier is moved by the micrometer screw until the zero lines of the scale and vernier coincide with one another. The scale is now moved through several degrees by the milled screw, and the field again is equalized as before, and if the zero lines do not coincide the vernier is to be again adjusted. These manipulations are repeated until the zeros coincide in several successive observations. These adjustments are very fatiguing to the eye, which should be rested a short time before making the final observations.

It is advisable to have quartz control plates for checking the adjustment of the instrument and the correctness of the scale. Standardized plates of the highest accuracy may be obtained from the makers of polariscopes, and these take the place of the observation tube and a standard solution of pure sucrose. One plate should read approximately 96° and a second about 60°, as these parts of the scale are used in the most important polarizations.

It is well to have the quartz plates standardized by the Bureau of Standards but it should be noted that the Bureau uses the Bates-Jackson scale (see Sec. 455) which is about 0.10° *higher* than the Ventzke scale, i.e., a plate marked 98.50° on the Ventzke scale will be certified as close to 98.60° by the Bureau of Standards.

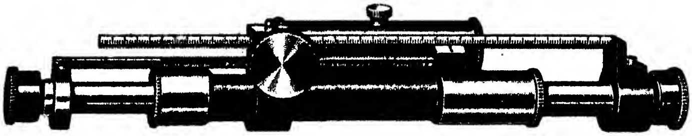

FIG. 121. Control Tube.

The scale may be tested with a control tube made by Schmidt and Haensch, a recent form of which is shown in Fig. 121. It is telescopic in construction and the column of solution can be adjusted to any length from 210 to 420 mm. The tube length may be read by means of a vernier scale to 0.1 mm. A full description of the use and value of the control tube is given by Browne.[18]

The committee of the Fourth International Congress of Applied Chemistry also recommends the use of pure cane sugar for testing the polariscope. The preparation of pure sucrose is too exacting a process for the average laboratory.[19]

The analyzer and polarizer should not be removed from the instrument or adjusted except by an experienced workman, and in the event of an accident to the polariscope it should be returned to the dealer for repairs.

All parts of the polariscope should be kept very clean, especially the exposed parts of the lenses; these should be occasionally cleansed with alcohol and wiped with old linen or lens paper.

461. Observation Tubes. The older-type tubes of glass are shown in Fig. 122. The upper tube has screw caps and the lower the Landolt slip cap. The slip cap is designed to prevent undue pressure upon the cover glass. The tubes of the French Laurent instrument are supplied with slip caps that have

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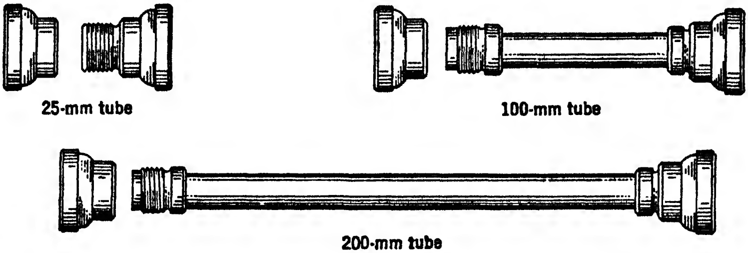
[18] *J. Ind. Eng. Chem.*, **12**, No. 8 (1920). See also Browne-Zerban, *Sugar Analysis*, third edition, 1941, p. 190.

[19] See *Bur. Standards Circ. C 440*, "Polarimetry, Saccharimetry and the Sugars" (1942), pp. 392–396.

bayonet catches. A coiled spring is arranged inside the cap to bear upon the cover glass and hold it in position, without unnecessary pressure.

Tubes of the type shown in the illustration, Fig. 122, must be completely filled with the solution to be polarized. The cover glass should be slipped sidewise onto the tube, pushing off the surplus liquid. The glass body of the filled tube should not be touched with the hands, since the warmth will cause striations to form in the liquid. These prevent an immediate observation. In the event of striations, the tube should be left in the trough of the polariscope until the field clears.


25-mm tube

100-mm tube

200-mm tube

Fig. 122. Plain Glass Polariscope Tubes.

Observation Tube with Enlarged End. This tube is shown in Fig. 123. In using this tube, it is nearly filled with the solution to be polarized, leaving room for a small bubble of air, which rises to upper part of the enlargement. This arrangement obviates the necessity of excluding air bubbles, and facilitates filling the tube.

Fig. 123. Polariscope Tube with Enlarged End.

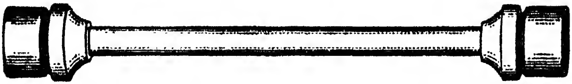


Fig. 124. Bates Metal Tube.

Bates' Observation Tube.[20]

In order to overcome the prevalent defects in the theoretical design, as well as secure a tube suitable for severe usage, this Bureau (U.S. Bureau of Standards) has brought out the Bates type of tube shown in Fig. 124. It will be observed that the weight is carried upon two shoulders, which are integral parts of the tube and not upon the caps, thereby eliminating all danger from

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[20] Copied from *Bur. Standards Circ. 44,* "Polarimetry," p. 39.

turning while in the trough of the instrument. The bore is 9 mm., permitting the utilization of the full aperture of the polarizing system. This also reduces to a minimum the light depolarized by reflection from the walls of the tube. The field of the instrument thus appears for the first time as a bright, sharply defined circle with no overlying haziness, and the reading can be made with increased accuracy. Both ends are enlarged with all the attendant advantages, yet but one size of cover glass and washer is required. The walls are unusually heavy, eliminating all danger from bending.

Observation Tube with Side Tubule. The tube shown in Fig. 125 is very convenient in general sugar analysis. The cover glasses need rarely be removed. This arrangement reduces the risk of error by compression of the cover glasses. The tubes should be frequently cleaned with diluted acetic acid.

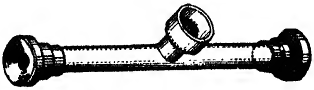

FIG. 125. Polariscope Tube with Side Tubule.

Pellet's Continuous Tube. This tube, Fig. 126, is designed for very rapid polarization and is especially adapted to the use of laboratories for beet-seed selection and the purchase of raw material on a basis of its sucrose content. The Pellet tube is also convenient in the control of the char filters in sugar refineries.

The Pellet tube provides for the rapid change of solutions without removing it from the trough of the polariscope. The tube is so arranged that it may form a part of a syphon by connecting rubber tubing to the tubules. The short leg of the syphon terminates in a glass tube which is dipped into the new solution and a pinch cock on the long leg is then opened. The incoming liquid displaces the previous solution. This arrangement should be used only with solutions of approximately the same polarization and not when scientific accuracy is desired.

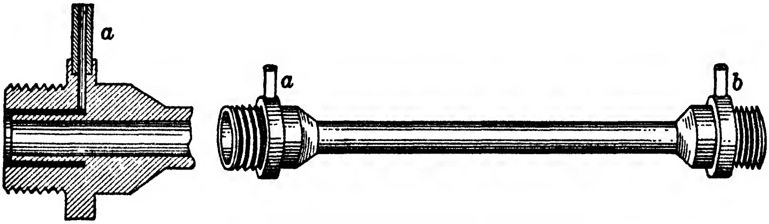

FIG. 126. Pellet Continuous Tube.

Landolt's Inversion Tube: Wiley's Modification. This tube, Fig. 127, is arranged for the control of the temperature of the solution under observation, especially in the Clerget double-polarization method. The glass observation tube is enclosed in a metal jacket through which water is circulated while polarizing. A side tubule, enlarged to funnel shape, is provided for use in filling the tube and in taking the temperature of the solution. A centigrade thermometer graduated to fifths of a degree should be used.

An improved form of inversion tube (Fig. 128) has baffle plates to insure proper circulation of the cooling water; the thermometer is offset to avoid interference with the light to the polariscope scale; the stopper is of ground glass with the thermometer cemented into it, the capillary *k* allowing for expansion of the liquid.

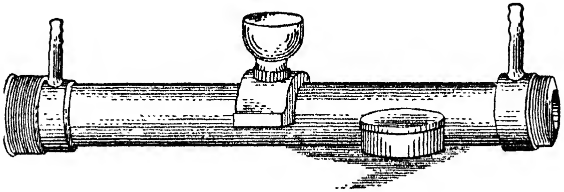

Fig. 127. Landolt's Inversion Tube.

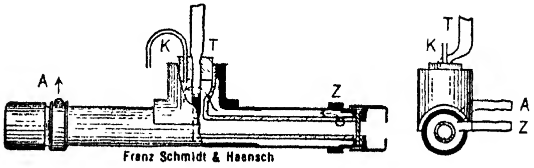

Fig. 128. Improved Inversion Tube.

462. Preparation of Solutions. A solution of a definite concentration, sufficiently free of color and clear to transmit light, must be prepared. Though solutions of various concentrations may be used, it is general practice to simplify the calculations by using a multiple or convenient fraction of the normal weight dissolved in 100 ml. of solution or a multiple of this volume number.

Dissolve the normal weight (see Sec. 454) of the material contained in a 100-ml. flask in water and add sufficient lead subacetate solution to give a solution that may be read easily in the polariscope. Complete the volume of the solution to the 100-ml. mark of the flask. Mix the contents of the flask thoroughly and pour upon paper for filtration. The entire contents of the flask should be poured upon the paper at once. Reject the first portions of the filtrate or all of it until a perfectly clear solution is obtained. These portions should be used in rinsing the receiving vessel. Air-dried filter paper is now specified by the International Commission for Uniform Methods (1932). Not less than 25 ml. of the filtrate should be discarded when polarizing raw sugars. The funnel should be covered during filtration to prevent evaporation. No portion of the filtrate should be poured back through the filter in careful work.

The above description covers the preparation of solutions in a general way. Detailed descriptions will be given in connection with the analysis of the various materials, especially as regards the use of the clarifying agents. The quantity of subacetate of lead to be used in clarifying solutions can be given only approximately on account of the variations in the materials themselves and in the illumination of the polariscope. In general the minimum quantity of the lead salt that will yield a clear and sufficiently light-colored solution should be used. Excessive use of lead introduces important errors into the analysis. The following numbers refer to milliliters of the lead solution of 54.3° Brix per one normal weight of the material: Raw juice, 0.75 to 2.0 ml.; sirup, 7 to 10 ml.; molasses of 20° Brix (vacuum-pan control), 5 to 10 ml.; first molasses, 7 to 10 ml.; final molasses, 20 to 30 ml.; 96° raw sugar, 1 to 3 ml.; 98° raw sugar, 1 to 2 ml.; 80 to 89° second sugar, 5 to 7 ml.; filter press cake, 6 ml.

Horne's dry lead subacetate may replace the solution of the salt. (Sec. 475.) About one-third of a gram of the dry salt is equivalent to 1 ml. of the 54.3 Brix.

Alumina cream should be used in addition to the lead subacetate in clarifying a sugar solution. The alumina cream alone is usually sufficient in the analysis of high-grade sugars.

With material of unknown composition add a little of the lead reagent to the sugar solution, mix thoroughly, await the subsidence of the precipitate, and then test the supernatant liquid with a drop of the reagent to ascertain whether more lead is required. An experienced operator can readily judge by the appearance of the precipitate whether the lead has been used in sufficient quantity. The reagent should be measured in routine work, and in so far as is possible the same quantity should always be used with similar materials. The need for avoiding an excess of lead will be understood from a study of Sections 473 and 477.

463. Weighing Dishes. The materials in sugar analysis are most conveniently weighed in a nickel or German-silver capsule, made especially for this purpose (shown on scale pan, Fig. 134). The solutions do not adhere to the polished surfaces of the capsule, and the shape of this is such that the material may be very readily washed into the flask. The capsules lose weight gradually through use. The counterpoise should not be filed to correct for this loss, but the stem or plug should be unscrewed and sufficient lead be removed from the cavity to compensate.

If the sugar flask has a narrow neck, as is prescribed in careful work, it requires skill to wash sugar or other material into it. This operation is facilitated by inserting the stem of a small German-silver funnel, made for the purpose, into the neck of the flask. This should reach to just below the neck, thus keeping the sugar from contact with the latter. The solution adhering to the capsule and funnel should be washed into the flask with a jet of water. Sufficient room must be left in the flask—about 20 ml.—to permit giving the liquid a rotary motion, for dissolving the material.

464. Dissolving the Material. The flask should be held by its neck to prevent the hand from warming the solution. From time to time it should be examined from below to note whether undissolved material remains. Many mechanical devices for dissolving sugars are in use, the one designed by Multer, Fig. 129, being a convenient form. It consists of a motor-driven inclined holder, for a single flask, which rotates slowly, allowing the sugar grains to fall through the solution as the flask turns. In practice it has been found that the device dissolves the normal weight of raw sugar in about the time that an operator takes to weigh out the succeeding sample and wash it into the flask, so one dissolver is ample for the needs of one analyst.

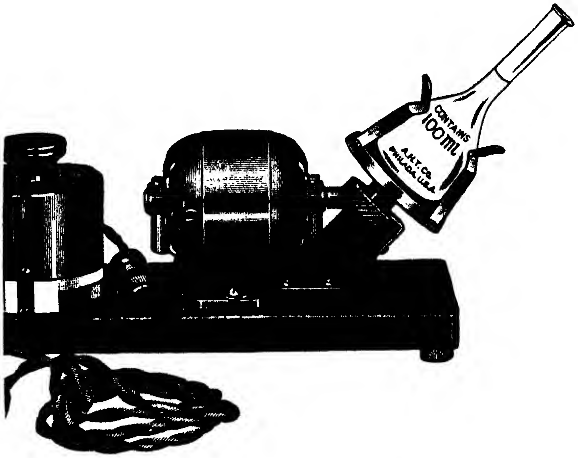

Fig. 129. Multer Sugar Dissolver.

465. Mixing and Filtering. After solution of the material and its clarification with lead subacetate as has been described, the volume should be completed to the mark with distilled water. If drops of water adhere to the neck of the flask they should be absorbed with strips of filter paper. A drop of ether or fusel oil may be poured on the surface of the liquid if foam is present when the solution is close to the mark. In filling to the mark the flask should be held on a level with the eye and the water added drop by drop until the lowest line of the meniscus appears to touch the line on the flask.

The contents of the flask should be thoroughly mixed by shaking and be poured immediately upon the filter. The filter should be of paper suitable for rapid filtrations, and should be fluted or "star" folded or a ribbed funnel should be used. The funnel and paper should be large enough, especially in analyzing sugars, to receive the entire contents of the flask. The filter paper should never be so large as to project above the edges of the funnel. The first few cubic centimeters of the filtrate should be used in rinsing the filtering cylinder and should then be rejected. It is always necessary in analyzing sugars and other materials requiring considerable accuracy to reject portions of the filtrate until it runs clear. If the filtrate does not clear, it is best to prepare a new solution, changing the quantity of lead. The polarization should never be attempted except with perfectly clear solutions.

It may sometimes be difficult to obtain clear solutions with the juice and products of unsound cane. The addition of a little common salt or sodium phosphate followed by refiltration will often remedy this, or both the salt and kieselguhr may be used. Occasionally the difficulty may be due to insufficient subacetate. If so, a little of Horne's dry subacetate may be added but for accurate work the test should be repeated.

Filtering Devices. A convenient filtering arrangement is illustrated in Fig. 130. *A* is a stemless funnel, *B* a quarter-pint precipitating jar, and *C* a small cylinder. A plain cylinder is preferred by many chemists to the lip cylinder, as the funnel makes a closer joint with the edge. *E* is a tumbler beaker of heavy glass which is particularly easy to wash and dry because of its open form. *D* is a copper funnel 4 in. in diameter, fluted, which is convenient and avoids breakage. The metal stemless funnels and heavy glass precipitating-jars, cylinders or tumblers may be washed and dried readily. The glass vessel serves as a convenient support for the funnels.

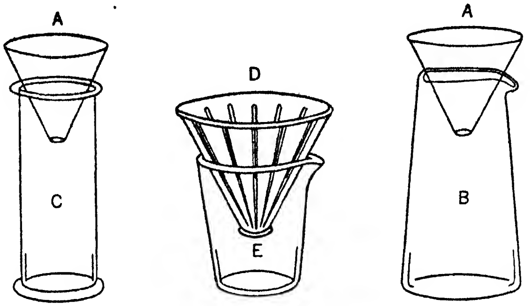

Fig. 130. Filtering Devices.

466. Sugar Flasks. The flasks used in sugar work are usually graduated to hold 50 ml., 100 ml., or multiples of 100 ml. They are frequently graduated with two marks, viz., 50–55 ml., 100–110 ml., etc., and are then called sugar flasks by the dealers.

Orders to dealers for flasks and other precision ware should be very specific in stating the system of graduation. It is important that all such ware in the laboratory be of the same system of graduation. Mohr's units, used with the old normal weight of 26.048 grams, have been almost entirely superseded by the metric or true cubic centimeters at 20° C. (ml.) with the normal weight of 26 grams (or 26.026 grams, see Sec. 454) adopted by the International Commission for Uniform Methods.

Flasks should be made from glass tubing of uniform bore and circular cross section. The shape of the body of the flask should approximate that of the diagrams in Fig. 131. A flask of this form gives little trouble from air bubbles.

Flasks for commercial work often have necks of larger internal diameter than those used in the U.S. Customs laboratories or in research work. This is unnecessary and the diameters specified below should be adhered to. The maximum internal diameters of the neck and limits of tolerance of error in the capacity of flasks as specified by the U.S. Bureau of Standards are given in the table.

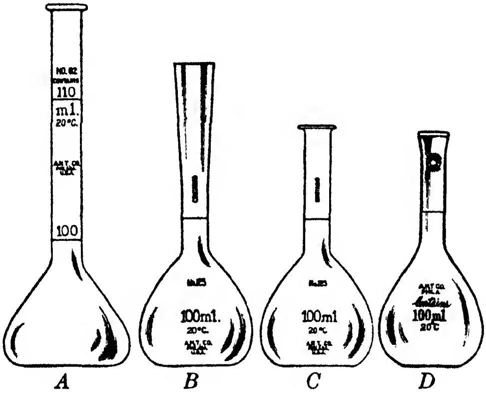

Fig. 131. Types of Sugar Flasks.

Capacity of the Flask, cc. (or ml.)	Maximum Internal Diameter of the Neck, mm.	Tolerance of Error, cc. (or ml.)
50	10	0.05
100	12	0.08
200	13	0.10
250	15	0.12
500	18	0.15
1000	20	0.30

This limit of tolerance is too large for flasks of such small neck diameter. Of several hundred flasks purchased from a prominent dealer for the laboratories under the direction of Dr. Spencer and bearing the maker's certificate of calibration, all were well within the tolerance limits given in the table. A maximum error of 0.04 ml. should be the requirement for a 100-ml. flask to be used in accurate polarization work. (See further, Sec. 467, "Calibration of Sugar Flasks.")

The 100-ml. flasks specified for use in the U.S. Custom House laboratories are like *B* of Fig. 131. These have a height of 130 mm. The neck is 70 mm. in length and its internal diameter must be not less than 11.5 mm. and not more than 12.5 mm. The graduation marks shall be not less than 30 mm. from the upper end and 15 mm. from the lower end of the neck.

All the flasks shown in Fig. 131 conform in shape of the body with the U.S. Customs regulations. They should be distinctly marked with their capacity and the system of graduation, e.g., "Contains 100 cc., 17.5/17.5° C." for the Mohr flask and "Contains 100 ml. 20° C." for the metric or true cubic-centimeter flasks. The graduation mark should completely encircle the neck of the flask.

The flasks *A* and *B* of various capacities fill most of the requirements of the sugar house laboratory. The Stift, *C*, and Kohlrausch, *D*, flasks are used in the analysis of filter press cake. The flask *C* if narrow at the graduation may be used in all classes of work.

The flasks should be frequently and thoroughly cleaned. C. A. Browne[21] recommends cleaning with a warm solution of sodium hydroxide and Rochelle salts, such as is used in preparing Fehling's solution. This removes the film of lead carbonate that deposits upon the walls of the flask. Strong commercial muriatic acid is usually used for this cleaning in sugar house laboratories. Treatment with nitric acid, followed by washing and then by a strong solution of chromic acid in concentrated sulfuric acid, is good preparation of flasks for calibration. It is advisable to use the chromic acid solution frequently in cleansing flasks. After this treatment the drops of water will drain from the neck of the flask properly instead of adhering to it.

467. Calibration of Sugar Flasks. No flask should be used in important work without its marked capacity having first been verified. There was formerly much confusion on the part of manufacturers between the true and Mohr's cubic centimeter, flasks of the one system being sometimes marked as having been graduated to the other.

Cleanse the flask as has been described above and thoroughly dry it in an oven. On cooling the flask note whether moisture has condensed upon the inside walls and if so return the flask to the oven.

The weighing should be by substitution to eliminate errors of the balance itself. Cool the flask to room temperature in the balance case, but do not wipe it again, and then accurately counterpoise it, placing it upon the left-hand balance pan. Pieces of metal or weights may be used in counterpoising. Remove the flask from the balance and counterpoise the weight on the right-hand pan with accurate weights substituted for the flask. This gives the weight of the flask to the limits of accuracy of the analytical weights. Fill the flask to the mark with recently boiled distilled water of room temperature. A large pipet should be used in running the water into the flask to avoid so far as is possible wetting the neck. With a roll of filter paper remove water that may adhere to the neck of the flask. Verify the filling of the flask by holding it by the upper part of the neck with the graduation at the level of the eye. If the lower part of the curve of the meniscus is not in line with the graduation mark, add or remove water by means of a small pipet and bring it into line.

Place the filled flask upon the balance pan as before and counterpoise it with pieces of metal or weights. Remove the flask and note the temperature of

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[21] Browne-Zerban, *Sugar Analysis*, third edition, 1941, p. 262.

the water with a thermometer reading to tenths of a degree. Counterpoise the metal with the analytical weights and record this weight as that of the flask and water. Deducting the weight of the flask gives the correct weight of the water whether the balance is in proper adjustment or not.

Reference is now had to the table, p. 697, showing corrections for weight according to temperature. For example, the flask is found to contain 99.680 grams of water at 22.3° C. By adding the correction 0.325, the capacity of the flask at 20° C. is 100.005.

468. Spencer Flask Calibrating Device. This instrument was designed by Dr. Spencer to permit of accurate graduation of flasks at the Central Control Laboratory of the Cuban-American Sugar Company at Cardenas, Cuba. Primarily, it is a method of recording by electrical contact the exact surface of the water in a flask and at the same time marking this surface level accurately on the outside of the flask. The drawing, Fig. 132, shows the construction of the device, and a description of the method of calibrating a flask will make the operation plain. Flasks of the right form and size required to hold the desired volume are purchased in "blank" with no graduation mark. In addition to the calibrating device a series of Morse calibrating burets (shown at *A*, Fig. 132), covering the volumes of flasks to be marked, are re-

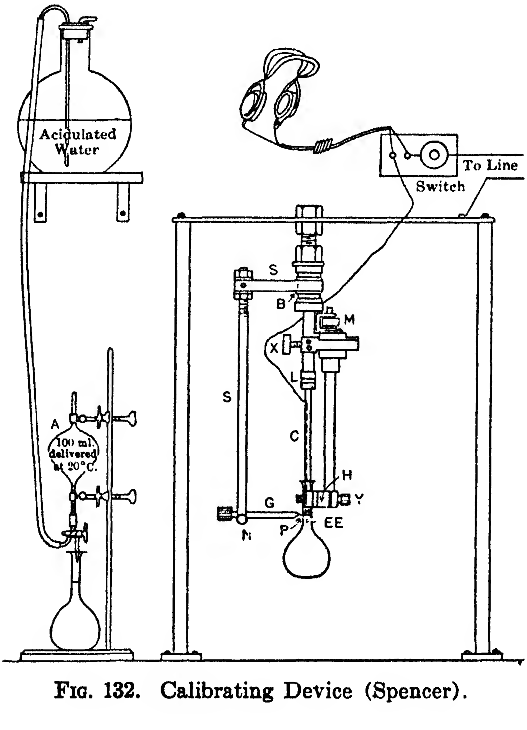

FIG. 132. Calibrating Device (Spencer).

quired. These burets should be standardized by the Bureau of Standards at Washington.

The first step is to adjust the graver point, *P*, and the platinum electrodes, *E*, so that they are exactly opposite each other. This is done, with no flask in the instrument, by loosening the lock nut, *L*, and raising or lowering the electrode carrier, *C*, at the same time that the nut, *N*, holding graver *G*, is loosened. The graver is pushed forward until it just touches the higher of the two points of the electrodes. The adjustment need be made only once for a series of flasks. The lock nut, *L*, is tightened, and the graver is drawn back, after which the flask holder, *H*, is lowered as far as it will go by turning the milled head, *M*, and the flask is put in place for centering only. By means of the screws *X* and *Y*, the adjustment of the flask is made so that the electrode holder will be approximately in the middle of the neck of the flask when the flask is raised.

The flask is now removed from the holder without disturbing this adjustment and the outside of the neck is coated with a film of molten beeswax and rosin which is allowed to cool. The flask, which has been dried previously, is now ready to be filled. From the proper Morse buret the exact volume of acidulated water ($H_2SO_4 - 1:1000$) is run into the flask which is promptly placed in the flask holder of the calibrating device and raised with the milled head until the electrode points approach the surface of the water. The current is now turned on, the headphones are adjusted to the ears, and the flask is raised very cautiously by means of the milled head until contact is made by the platinum points touching the surface of the water. This is apparent by the hiss in the earphones. It is now evident that the surface of the liquid in the flask is exactly on a level with the graver point on the outside. The graver is moved forward until the point penetrates the wax. (There is a spring behind the point which permits of definite contact with the glass and takes up any minor inequalities in the shape of the neck.) The whole marking sweep, *S*, is then swung completely around the flask on the bearing, *B*, so that the wax is cut away by the sharp point in a complete circle exactly at the liquid level. By means of Diamond Ink, or some similar hydrofluoric acid preparation, the flask is etched at this circle in the wax, after the flask has been removed from the instrument.

It is obvious that the work must be done in a place free from drafts and vibration. Between the time that the flask is filled and the mark made on the wax care should be taken to prevent any change in temperature.

Flasks marked in this manner and rechecked by the weighing method given in the previous section were found to be extremely accurate; over 93 per cent of the flasks so checked showed an error of less than 0.025 ml., while not one had an error amounting to 0.05 ml. The Bureau of Standards has adopted the Spencer device with certain modifications to suit its purposes.

469. Balances. Convenient types of balances for sugar work are shown in Figs. 133, 134, 135, and 136. In addition, a good analytical balance and a set of high-grade weights are required. The analytical balance is used in ash determinations, glucose tests, calibration of sugar flasks, and in the many special investigations. A sugar balance of good accuracy and a capacity of about 300 grams is needed in weighing samples for polarizations. For use in tropical and subtropical countries this balance should be fitted with agate bearing and knife edges. The balance shown in Fig. 133 is suitable.

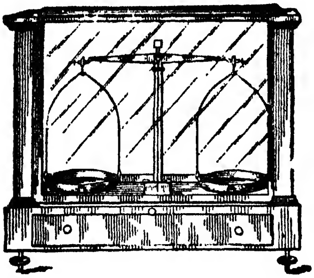

FIG. 133. Sugar Balance.

The Bates balance, Fig. 134, especially designed for polarization work, has several convenient features. The customary bow has been replaced by a single bow at the back, giving free access to the pans and reducing spilling of sugar to a minimum. The scoop or weighing dish balances either of the pans and is interchangeable with them, thereby avoiding the use of a counterpoise weight. Two sets of sugar weights, normal and half normal, should be provided. One set of these weights should be kept in reserve for verifying and checking the weight of the others. The balances should at least be sensitive to 2 mg. with a full load in the pans, though weights of sugar materials for polarization to within 5 mg. are usually sufficiently accurate. The errors that may be introduced through evaporation or absorption of moisture in slow weighing are of more importance than weights to within 2 mg.

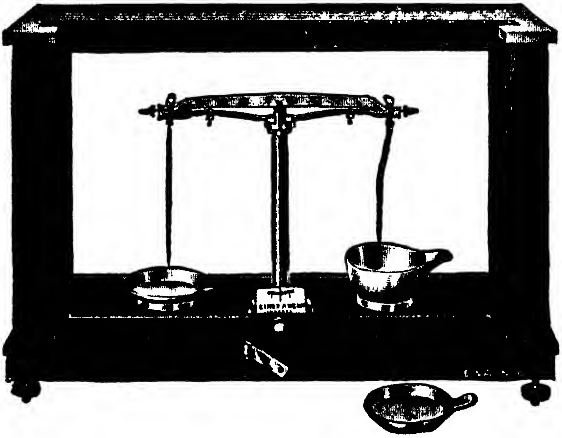

FIG. 134. Bates Sugar Balance.

The bullion type of balance shown in Fig. 135 is very convenient for the so-called rough weighings in which large capacity and speed of manipulation are essentials. This balance should be placed in a glass-framed hood to protect it from dust and currents of air. This instrument is made in several capacities. That of 5 kg., sensitive to 100 mg., is a suitable size. It should have agate bearings and knife edges for tropical work. This scale is suitable for use in bagasse analysis and in determining the degree Brix of massecuites and molasses. An ordinary trip balance is also useful in this type of work (Fig. 136).

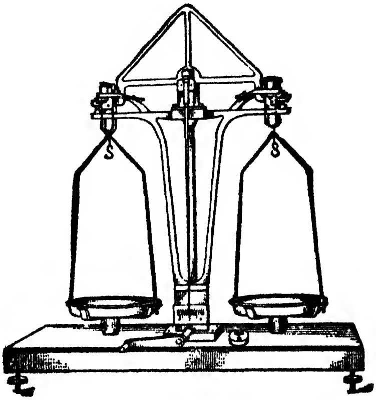

FIG. 135. Balance for Rough Weighings.

470. Wash Bottles. Stock Bottles. The water and the lead solution for use in sugar analysis should be kept in large bottles on a shelf above the work table. The water bottle should have a glass syphon tube with rubber connections, a pinch cock, and glass nozzle, forming a convenient arrangement for washing samples into the sugar flask and for diluting the solutions to the graduations on the necks of the flasks. This is a very satisfactory form of wash bottle, and several of these should be distributed about the laboratory. A double nozzle connection, one having a glass tip of large size and the other a fine glass tip is an added convenience, the first to permit of rapid filling and the second of fine adjustments to the mark of flasks, etc. The lead subacetate bottle should be connected with a reservoir buret. The buret has a three-way cock, one opening connecting with the stock bottle by means of a glass syphon and a rubber tube. The air inlet to the lead subacetate bottle should be provided with a small wash bottle containing caustic soda solution, to absorb the carbonic acid and prevent precipitation of the lead. A small precipitation is not objectionable, but where very large stock bottles are used the air-washing arrangement should be employed.

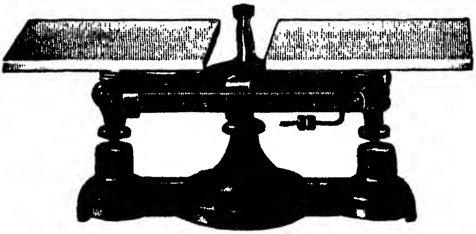

FIG. 136. Trip Balance.

Where lead subacetate solution is used in storing samples of juice a stock bottle containing the concentrated solution should also be provided.

471. Heating Devices. The electric hot plate is the most convenient heating device for laboratories having a twenty-four-hour electric service. Where the generators are operated only at night the hot plates must be supplemented by stoves. Various types of alcohol and gasoline stoves are on the market that are satisfactory for heating in inversions, extractions, etc. Where they are available, natural gas and illuminating gas afford excellent sources of heat for which many laboratory burners have been devised.

472. Notes on Polariscopic Manipulations. The screw caps of the observation tubes should not bear heavily upon the cover glasses, since glass is double refracting under these conditions and does not quickly recover its normal condition. A large error may be introduced through excessive pressure on the glasses. The cover glasses should be of the best quality of glass, perfectly clean and with parallel sides. A glass may be tested with regard to the parallelism of its surfaces by holding it in front of a window and looking through it at a window bar; on revolving the glass slowly between the thumb and a finger, if the bar appears to move, the surfaces are not parallel, and the glass should be rejected. Old glasses which have become slightly scratched should not be used. Glasses and observation tubes should be frequently washed with acetic acid.

The planes of the ends of the observation tubes should be perpendicular to the axis of the tube. This may be tested by placing a tube filled with a sugar solution in the trough of the polariscope and making an observation; on revolving the tube in the trough and making observations at different positions, if the readings vary, the ends of the tube have not been properly ground.

The manufacturers of polariscopes and their accessories have attained such accuracy in their methods that faulty apparatus rarely leaves their workshops, but the scales and accessories should be checked to verify graduations and tube length.

The polariscope should be used in a well-ventilated room. It should be protected from the heat of the lamp and, so far as practicable, from light from other sources. A convenient arrangement is to place the instrument in a box, leaving one end open. The lamp is placed outside the box, opposite the open end, and lights the instrument through a small opening. In making a reading, the observer stands at the open end of the box, his body cutting off the greater part of the extraneous light. A black curtain is also frequently employed. The inside of the box should be painted black. The polariscope should be fastened to the bottom of the box or table, so that it may not readily be jarred out of position.

The illumination of the scale is effected by reflecting light from the polariscope lamp upon it, except in the old models of instruments.

The zero-setting of the polariscope scale should be frequently verified, using standardized quartz plates. The bichromate filter cell (see Sec. 448) must always be used in this verification and in adjusting the vernier. In fact this ray filter should never be removed from the polariscope except for refilling or on account of the dark color of the solution to be tested.

473. Influence of the Volume of the Lead Precipitate. The lead precipitate introduces errors into the polariscopic analysis, some of which are offset by compensating errors, notably in the analysis of low-grade products. The errors due to the volume occupied by the precipitate will be considered in this paragraph, and those due to influence of the lead upon the sugars and other optically active bodies will be discussed farther on.

It is evident that if a part of the volume of the flask is occupied by a solid, and there is no compensation for the volume so occupied, the polariscopic reading will be too high. Numerous chemists studied this matter especially in connection with the beet sugar and refining industries in the early days of this century and before, and more recently numerous investigators studied the effect of the precipitate on the polarization of raw cane sugars.

The earlier investigations by Scheibler, Sachs, Pellet, and others were for the purpose of establishing the fact that the volume of the precipitate does actually raise the polarization. Most of these studies were on beet juice or beet molasses, cane juice or cane molasses. It is not important for us to review here the methods used or the varying results found, but in general, molasses and low-grade products were found to have compensating errors to offset the effect of the volume of the precipitate, whereas with high-test materials there was a perceptible error due to the precipitate. There was considerable controversy among the chemists named and other contemporaries as to whether the volume error actually existed, but it is now accepted as a fact and the investigations in recent years have been for the purpose of determining the extent that the volume of the precipitate increases the polarization of various grades of raw sugars. This work is closely bound up with the use of Horne's dry lead method described in Sec. 476 and will be reviewed following the description of that method.

474. Correction for Volume of Precipitate. The method of Sachs will illustrate methods employed for determining the volume of the lead precipitate in the earlier investigations.

Clarify 100 ml. of juice with subacetate of lead as usual, using a tall cylinder instead of a flask. Wash the precipitate by decantation, first with cold water and finally with hot water, until all of the sucrose is removed. Transfer the precipitate to a 100-ml. flask and add one-half the normal weight of cane sugar of known polarization, dissolve the sugar, and dilute the solution to 100 ml., mix, filter, and polarize, using a 400-mm. observation tube. The results are calculated as follows.

Let P = per cent of sucrose in the sugar;

P' = the polarization of the solution, made up in the presence of the precipitate;

x = the volume of the lead precipitate.

Then

$$x = \frac{100P' - 100P}{P'}$$

Scheibler's so-called double-dilution method[22] and Horne's method for direct weighing of the precipitate in a pycnometer have also been used.

475. Horne's Dry Lead Method. In 1903 W. D. Horne[23] recommended the use of dry lead subacetate to eliminate the volume of precipitate error. The finely powdered lead salt is added to the solution after dilution to volume. Thorough mixing and filtration follow. If the lead salt has been added in just sufficient quantity for the clarification, there is no dilution of the solution and no precipitate error. All the precautions necessary in the use of the lead in solution as regards precipitation of reducing sugar, influence upon specific rotation, etc., must be observed with the dry salt. The anhydrous lead subacetate employed should be only that especially prepared for sugar analysis and should contain 72.8 per cent of lead. One-third of a gram of the salt is equal to 1 ml. of the regular 54° Brix lead solution.

It is surprising that for twenty-five years following Horne's recommendation, the use of dry lead for raw sugar polarization was not authorized by any of the official government or scientific agencies. The reagent lay dormant so far as its original purpose was concerned, although sporadic attempts were made to introduce it. It early proved of great value in sugar analysis, however, and is almost universally used in routine purity determinations and other control tests in beet houses, cane factories, and refineries. Rapidity and accuracy are gained by its use in these tests since the necessity of a dilution factor is avoided. The dry lead is also very valuable as a preservative in the storage of juice samples, and its use for this purpose is general. Extensive experiments by Spencer proved that raw juices may be stored in hot climates for a period of several days without appreciable deterioration using no more of the lead salt than is necessary for the clarification of the juice. (See Sec. 611.)

Within the past fifteen years the dry lead method as originally recommended by Horne has been given general recognition. Its use in polarizations was made optional by the Association of Official Agricultural Chemists about 1926 and was declared "permissible" by the International Commission for Uniform Methods of Sugar Analysis in Amsterdam in 1932. The volume error was studied extensively at the recommendation of the Commission as will be shown later, and in 1933 the Central Association of the Czecho-Slovakian Sugar Industry made the use of dry lead mandatory in conjunction with the change in normal weight from 26.00 to 26.026 grams (see Sec. 455). At the 1936 meeting of the Commission in London the use of dry lead was made official in all cases where the international scale has been adopted. (See Secs. 454 and 455.) This means that the use of the dry reagent is the only *correct* method of clarification for polarization of raw sugar, since the use of the wet method is sanctioned only where the incorrect scale is used, i.e., the wet method is for the purpose of setting up a compensating error to offset a known error in the Ventzke scale.

[22] *Zeit. Rübenzucker-Industrie,* **25**, 1054 (1875).

[23] *J. Am. Chem. Soc.,* **26**, p. 186.

476. The Volume Error in Raw Sugars. The extent to which the volume of the lead precipitate affects the polarization of raw sugars has been studied in various countries. An extensive investigation was made by Zerban and Gamble[24] who reviewed previous work and noted that, besides the volume error, lead subacetate produces other effects, two of which have been known for a long time. These are the precipitation of levulose by the alkaline lead salt, and the other is the decrease of the levorotation of the levulose in the presence of lead subacetate. Both these effects increase the direct polarization of the sugar (see p. 393) as does the volume of the precipitate, but they cannot be corrected by the use of the dry lead subacetate. Zerban and Gamble were the first to study this total effect of lead clarification on the polarization of raw cane sugar, although many had previously investigated the increase due to the volume of the precipitate. In working with artificial mixtures of sucrose, dextrose, levulose, ash, and sodium aspartate they found that the total average increase in the absence of sodium aspartate is only 0.05, but when the aspartate is added in various quantities the increase is much greater, from 0.23 to 0.58. This is of great importance as Zerban, Ambler,[25] and others have found that the principal amino compound in cane products is aspartic acid, which in the presence of lead subacetate has about the same specific dextrorotation as sucrose.

Zerban and Gamble, in the study referred to above, found that for Cuban raws of about 96 test the volume error caused by the use of lead subacetate solution was equal to 0.14°. The additional error caused by the effect of the lead subacetate on the rotation, because of the presence of the levulose and amino compounds, persists even when Horne's method is used and is equal to 0.16°. They suggested no method for correcting this second error but stated that since the actual sucrose content of raw sugars, as determined by double polarization methods (Chapter 19), is usually higher than the direct polarization, this error caused by the presence of levulose and amino compounds tends to give a direct polarization more nearly approximating the true sucrose provided the volume error is corrected.

In a later study, covering 1525 samples of raw sugar taken at random, Zerban[26] found an average difference between the polarizations with lead solution and with dry lead of 0.138. The average differences ranged from 0.08 to 0.16, depending on country of origin, Louisiana sugars showing the lowest difference and Cubans the highest.

It should be mentioned that some few samples showed a *plus* difference for the dry lead polarization whereas a relatively large number (most of which were from the Philippines) gave differences less than minus 0.05. At least 20 per cent of the Cuban sugars had minus differences greater than 0.25 and a

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[24] *Facts About Sugar*, April, 1933, p. 180.

[25] *Proc. VIII Intern. Congr. Applied Chem.* (1912), p. 103; *Intern. Sugar J.*, 1927, p. 437.

[26] *Facts About Sugar*, August, 1934, p. 277.

few were above minus 0.40. These figures show that the *average* volume error may be very misleading for individual samples. In general the higher the polarization the less the difference, as would be expected, but polarization is not the only factor. For example, Louisiana sugars, with almost exactly the same polarization as Cuban sugars, showed only half the difference due to the volume of the precipitate. These average figures for the volume error with raw cane sugars corroborate the early investigations of Horne and others.

Sandera in Czecho-Slovakia and many other European investigators found the volume error in raw beet sugars to be about 0.1° V. Both the cane and beet investigators all agree that the volume error is appreciable and may be avoided by the use of Horne's dry lead subacetate. The only dissenting voice concerning the use of dry lead is the British Committee of the International Commission who argue [27] that the direct polarization of raw sugars is a conventional figure; that the volume error has always been present; that there are other errors which cannot be corrected by the use of dry lead; that some of these other errors are opposite to that caused by the volume error. They therefore conclude that lead subacetate solution should be continued as the clarifying agent for raw sugars. These arguments may have some weight but against them may be given the fact that the volume error is widely variable in different sugars and that its elimination by the simple expedient of using the dry reagent seems reasonable even in a commercial test which is admittedly only a convenient approximation.

477. Influence of Lead Salts and Other Substances upon the Sugars and Optically Active Non-Sugars in Cane Products. *Sucrose.* In the polariscopic methods the importance of the effect of lead subacetate and other substances on the specific rotation of the sugars present is obvious. The rotatory power of sucrose is not modified by lead subacetate under the usual analytical conditions. In the presence of a large excess of the salt there is a slight diminution of the rotatory power; there is a decided diminution in alcoholic solution containing the lead salt.

The investigations of Bates and Blake, U.S. Bureau of Standards,[28] show that errors of importance in rotation are caused by excessive amounts of subacetate of lead. An excess of 0.5 cc. causes a diminution of 0.1°; 1 cc. of 0.12°; 2 cc. of 0.11° and 3 cc. a diminution of 0.09° on the cane sugar scale. The rotation reaches a minimum value when an excess of 1 cc. is present and returns to the initial value with 6 cc. in excess and continues to increase with the amount of lead solution added. A sugar polarizing 99.9° was used in these experiments.

Numerous investigations during the past sixty years have shown that the chlorides, nitrates, sulfates, phosphates, and acetates of sodium and potassium, and the chlorides of alkaline earths, as well as many other salts all decrease

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[27] *Intern. Sugar J.*, May, 1935, p. 180.

[28] *Bur. Standards Bull. 3* (1907), p. 105.

the specific rotation of sucrose. The decrease generally varies inversely with the molecular weight of the salt.[29]

Small amounts of the chlorides of barium, calcium, and strontium decrease the specific rotation, but the decrease reaches a maximum as the concentration increases and then further additions of the salt cause an increase which finally exceeds that of the pure sugar solution. It is to be noted that the depression of the rotatory power is largely due to the relationship between the salt and the water, not of the sugar and the salt. This is of practical importance as the evaporation of a sugar solution high in mineral salts may appear to cause a loss in sucrose because of the increased concentration of the salts due to the evaporation.

In their studies of the Clerget method (see Sec. 488) Jackson and Gillis found [30] that when a known amount of either sodium, calcium, or ammonium chlorides, or potassium oxalate has been added to a normal weight solution of pure sucrose the depression of the polarization may be expressed by a simple formula in which the weight of salt is the variable. Browne [31] showed that the depressing effect is proportional to the concentration of sucrose as well as the salt concentration. This fact is now taken into account in the calculation of the Clerget divisor.

Dextrose. The rotatory power of dextrose is not modified, or, if at all, but very slightly, by either the subacetate or neutral acetate of lead, under the analytical conditions. See also Invert Sugar.

Levulose. The rotatory power of levulose is very greatly diminished by the presence of lead subacetate. Acetic acid added to acidity restores the rotation of the levulose (Gill in 1871, Spencer in 1885, Pellet). Levulose is partly precipitated as a lead salt in the presence of certain chlorides, in quantities greater or less, according to the relative proportions of the salts, lead, and levulose (Pellet, Edson). Basic lead acetate precipitates levulose in part, when salts occur in the same solution with constituents of which basic lead acetate forms insoluble combinations (Prinsen-Geerligs).

Invert Sugar (Dextrose and Levulose). In the presence of the salts formed in the decomposition of lead subacetate, dextrose and levulose are precipitated in part (Pellet, Edson). The influence of the basic lead salt on the rotatory power of levulose (see "Levulose") or the formation of levulosate of lead of little optical activity gives undue prominence to the dextrose, which is not affected and results in a plus error. Increasing amounts of lead subacetate added to invert sugar solutions decrease the left rotation, and finally the rotation is to the right on account of that of the dextrose. C. H. Gill called attention to this error in the *Journal of the Chemical Society,* April, 1871, and in the early editions of this book, Spencer advised the use of acetic acid to restore the rotatory power of the levulose.

[29] Farnsteiner, *Ber.,* **23,** 3570 (1890).

[30] *Bur. Standards Bull. 375.*

[31] *Ind. Eng. Chem.,* 17–39 (1925).

In the work already cited, Jackson and Gillis showed that many salts increase the levorotation of invert sugar by amounts that may be expressed as a simple formula; for example, with m grams of NaCl in the presence of an inverted half normal weight of sucrose, referred to normal basis, the reading in degrees S at 20° C. $= 32.00 - 0.540m$.

Acetic acid slightly lowers the rotatory power of invert sugar; sodium acetate has an opposite effect. Sulfuric and hydrochloric acids increase the rotation; oxalic acid has no effect. The rotation increases as the quantity of the mineral acid is increased. The action of inorganic acids is less pronounced and may be disregarded in polariscopic analysis.

Malic Acid. This acid is levorotatory. The artificial malic acid is optically inactive. Malic acid is precipitated by lead subacetate.

Pectine and Parapectine. These substances are dextrorotatory and are both precipitated by lead subacetate, and the second by normal lead acetate.

Asparagine. This is not precipitable by lead subacetate but is rendered dextrorotatory instead of levorotatory by the lead salt. In water solution and alkaline solution asparagine is levorotatory; in acid solution, dextrorotatory.[32] Asparagine is insoluble in alcohol, and in the presence of acetic acid is inactive.[33] In neutral and alkaline solution asparagine is levorotatory; in presence of a mineral acid, dextrorotatory; in the presence of acetic acid the rotation is diminished and with 10 molecules of the acid becomes 0°, and with additional acid is dextrorotatory (Degener). Asparagine is present in cane juice, especially that from immature cane; it is changed to aspartic acid by the action of lime, and as a lime salt is found in molasses.[34]

Aspartic Acid. This is formed from asparagine by the action of lime; the lime salt is soluble.

Zerban and Gamble[35] report that, although aspartic acid in neutral solution is slightly levorotatory, in the presence of lead subacetate it has about the same specific dextrorotation as sucrose. They showed that as little as 0.1 per cent sodium aspartate together with other impurities may raise the polarization as much as 0.23° when 1 ml. of lead solution is used for clarification (see Sec. 476). Previous editions of this handbook stated that aspartic acid was precipitated by lead subacetate, but Zerban and Gamble's findings do not bear this out.

478. Boneblack Error. Boneblack or animal charcoal exercises an adsorbent action on sugars and the impurities present in sugar products. For this reason its use in analytical work has been discontinued, and most official methods definitely exclude its employment. The same objection holds for vegetable carbons. Modern high-power electric lights in polarizing have made it possible to dispense with these decolorants.

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[32] Landolt, *Optical Rotation of Organic Substances,* Dr. Long's English edition, p. 541.

[33] Champion and Pellet, *Compt. Rend.,* **82,** 819.

[34] W. Maxwell, *Louisiana Expt. Station Bull. 38,* second series, p. 1380.

[35] *Facts About Sugar,* April, 1933, p. 181.

479. Influence of Temperature upon Polarizations. Corrections for Temperature. The compensating type of polariscope can only give correct readings at the temperature at which it was standardized, which for modern instruments is 20° C.

The rotation due to the quartz wedges increases with rise of temperature and that of sucrose decreases, giving a combined effect of a decrease in the reading of a normal weight of pure sucrose of 0.03° Ventzke for each 1° C. increase in temperature. The U.S. Treasury Department was the first to use temperature corrections in polarizations, these being based on tables prepared by Dr. H. W. Wiley.

C. A. Browne[36] made a full study of the influence of temperature in the polarization of raw sugar, and stated that the formula

$$P^{20} = P^{t}[1 + 0.0003(t - 20)] \qquad (1)$$

in which P^{20} is the corrected polarization, P^{t} the observed polarization, and t the temperature of the observation might be applied without serious error to cane sugars polarizing above 96°. (In actual practice, many laboratories simplify the calculation by merely adding 0.03 V. for each degree above 20° C. This is the equivalent of the correction added by the U.S. Customs Laboratories for the purpose of assessing duties.)

The formula is based on the effect of temperature on the rotation of *pure sucrose,* and as the impurities increase (particularly levulose) the formula loses its validity. Browne has shown that at about 80° V. the effect of temperature upon the rotation of levulose and other impurities counterbalances the decrease in the rotation of the sucrose and quartz wedges, so that at this point no temperature correction at all is applied. Below 80° V. the temperature coefficient of sucrose is generally more than overbalanced so that the polarization *increases* with a rise in temperature. The following formula is applicable to cane products other than high test products:

$$P^{20} = P^{t} + 0.015(P - 80)(t - 20) \qquad (2)$$

It is to be understood that this formula is for materials of average composition, and will fail to give accurate results in individual cases where the composition is unusual, particularly as to levulose content.

The Association of Official Agricultural Chemists[37] recommends that the formula (1) be used only for refined sugars testing 99° or above and that the formula (2) given above be used for all raw cane sugars irrespective of polarization.

The following table of corrections for different polarizations and temperatures is calculated according to formula (2). As has already been explained the formulas are merely approximations for materials of average composi-

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[36] *J. Ind. Eng. Chem.,* 1, 567.

[37] *Official Methods,* fifth edition, 1940, p. 491.

Temperature °C.

Pol.	21°	22°	23°	24°	25°	26°	27°	28°	29°	30°
90	0.02	0.03	0.05	0.06	0.08	0.09	0.11	0.12	0.14	0.15
92	0.02	0.04	0.05	0.07	0.09	0.11	0.13	0.14	0.16	0.18
94	0.02	0.04	0.06	0.08	0.11	0.13	0.15	0.17	0.19	0.21
96	0.02	0.05	0.07	0.10	0.12	0.14	0.17	0.19	0.22	0.24
98	0.03	0.05	0.08	0.11	0.14	0.16	0.19	0.22	0.24	0.27
100	0.03	0.06	0.09	0.12	0.15	0.18	0.21	0.24	0.27	0.30

tion. Bryan[88] has shown that whereas the average of 4117 samples of raw sugars corrected by the formula (2) was within 0.01° S. of the polarization of the same sugars done at 20°, less than half the individual samples checked within 0.05° by the two methods. The practical accuracy of the correction formula (2) has been demonstrated year after year by comparisons of refinery control polarizations corrected by the formula, with polarizations of the same sugars in the New York Sugar Trade Laboratory where the standard temperature room at 20° is employed. Average polarizations by the two methods have always been extremely close.

480. Constant Temperature Room. Dr. Browne concluded that the only strictly accurate method was to polarize at 20° C. and he equipped the New York Sugar Trade Laboratory (of which he was then in charge) with a constant temperature room in which all sugar solutions are prepared and polarized at 20° C. This has recently been redesigned and rebuilt as described by Zerban.[89] Although it is recognized that this is the only means of securing uniformity of results for all classes of products and should be used by arbitration laboratories, research laboratories, and the like, few industrial sugar laboratories could justify the expense of constant-temperature rooms, particularly in the tropics. The practical accuracy of the correction formulas for average results make their use in control work entirely permissible.

Obviously certain precautions can and should be observed to reduce the effect of temperature. The laboratory and polariscope room should be well ventilated and of the same temperature. The polariscope should not be in the vicinity of a heated wall or the laboratory ovens. The solutions should

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[88] *J. Assoc. Off. Agr. Chem.*, **4**, 328 (1921).

[89] Browne-Zerban, *Sugar Analysis*, 1941, p. 397.

be prepared and polarized at room temperature. Composite samples of sugar, used in part as a basis of the technical reports (run reports), should be polarized at a time of low temperature in tropical laboratories rather than in the heat of the afternoon.

481. Limits of Accuracy in Saccharimetric Analysis.[40] Dr. C. A. Browne notes twelve errors that may enter into sugar analysis. Though these refer specifically to sugar testing, they apply also to a considerable extent in all other analyses.

1. Loss of moisture during mixing.
2. Loss of moisture during weighing.
3. Error in normal weights.
4. Volume of precipitate in clarification.
5. Precipitation of levulose.
6. Error in capacity of flasks.
7. Imperfect mixing of contents of flask.
8. Evaporation during filtering.
9. Error in length of polariscope tubes.
10. Omission of bichromate cell.
11. Variations in temperature.
12. Defects in scales of saccharimeters.

The errors 3, 6, 9, and 12 are in general mutually compensating and with careful management do not appear in the results. Since flasks and tubes may be selected that vary but slightly on each side of the correct numbers, the errors disappear in the averages of duplicate tests. The weights may easily be kept within the limits of accuracy of the balance. The scales of the polariscope may be checked near the important points with accurate standard quartz plates, and corrections may be made. Other points and in fact all parts of the scale may be checked with the Schmidt and Haensch control tube, Fig. 121. The final residual error in careful work should not exceed +0.12 as follows:

CAUSE OF ERROR	SUGAR DEGREE
1. Evaporation in mixing	+0.010
2. Evaporation in weighing	+0.005
4. Volume of precipitate	+0.090
5. Precipitation of levulose	+0.015
Total error	+0.120

There are other sources of error that have already been considered in the previous pages of this work, such as those introduced by pressure upon the cover glasses, pressure upon or the warming of the standardized quartz plates, and preparation of the solution at a different temperature from that of the observation. Such errors are entirely avoidable.

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[40] See *Louisiana Planter 54,* **26** (1915), for Browne's full discussion of the subject.

The use of alumina cream or the normal lead acetate in the clarification instead of the basic acetate, when the material will permit, practically eliminates the levulose error. Horne's dry-lead method removes, or nearly so, the volume of precipitate error.

The temperature error is one of the most important with which the tropical chemist must deal. This may amount to −0.5° in extreme cases. Since a constant-temperature laboratory is an improbable addition to a sugar house, the only defense against this error lies in making important tests early in the morning during the low-temperature period, and in the use of the temperature correction formulas given in Sec. 479.

CHAPTER 19

DOUBLE POLARIZATION METHODS

CLERGET SUCROSE

482. Theoretical Considerations. It is evident that the simple polarization or direct polarization as described in the previous chapter can give correct results only when no optically active substance except sucrose is present. In order to eliminate the influence of the other optically active sugars generally present with sucrose in cane products Clerget, in 1846, standardized the method of double polarization which bears his name.

Cane sugar products usually contain the three sugars, sucrose (+), dextrose (+) and levulose (−). The direct polarization is therefore the resultant of the polarizations of these three sugars. The principle of the Clerget method is to supplement this direct polarization with a second polarization after complete inversion of the sucrose by suitable means. The rotation of the sugars other than sucrose remains constant, and the change in polariscopic reading before and after the inversion is due to the inversion of the sucrose. The sucrose is calculated by dividing this change in the polariscopic reading, due to inversion, by the Clerget constant; this constant represents the algebraic difference in the polariscopic readings of pure sucrose, before and after inversion, under the conditions of the method used. Suitable correction is made for the temperature at which the polariscope is read. In theory the Clerget is simple, but practically it requires the most exacting and rigorous adherence to correct technic, and the many modifications and changes which are constantly advocated are evidence of the troublesomeness of the method. The inverting agents employed are invertase and hydrochloric acid. The invertase method is the more exact but it has many attendant difficulties in the preparation of the substance and the time required for the inversion.

"For the purely practical considerations of the analysis time and the expense of the enzymes these methods are confined largely to research projects."[1]

The acid inversion is generally used in factory laboratories. It is recognized that, in common with most sugar analytical methods, the results are more or less close approximations chiefly of value from a comparative standpoint.[2]

483. Comparison of Methods. In a series of investigations Zerban and his collaborators have made a critical study of double-polarization methods under the auspices of the Association of Official Agricultural Chemists, and

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[1] *Bur. Standards Circ. C 440* (1942), p. 152.

[2] See also Browne-Zerban, *Sugar Analysis*, 1941, p. 440.

those results pertinent to cane sugar control work may be summarized as follows:

1. The solution used for the direct polarization must have the same dry-substance concentration as the solution used for inversion, and the Clerget divisor must be based on this concentration and not on the difference between the direct and invert polarizations.
2. Wherever possible it is preferable to carry out the inversion at room temperatures because at high temperatures slight variations in the time used may have an effect on the destruction of invert sugar, the hydrolysis of reversion products, and on the interaction between invert sugar and amino compounds.
3. The invertase method is the only one that may be depended upon to give reliable results under all conditions, provided the inversion is carried out at room temperature.
4. The sucrose result by the Jackson-Gillis method IV is *increased* by the hydrolysis of reversion products. Asparagine or aspartic acid, on the other hand, *lowers* the sucrose result because of the large difference in H-ion concentration between the solution for direct reading and the acid solution for invert reading. These amino compounds are generally present in all cane products, and for this reason the method IV gives correct results for these materials only through a compensation of errors.
5. The plain acid method (Herzfeld) may give any kind of a result, high, low, or correct, depending on the relative proportions of the constituents in the material analyzed.[8]

INVERSION WITH HYDROCHLORIC ACID

484. Modifications of the Method. There have been many modifications of the acid inversion method since it was first formulated by Clerget, and these have been devised with a view to simplifying the procedure and reducing the error through decomposition of levulose. These modifications deal largely with the temperature and time of heating and the amount and strength of the acid. All these factors, together with the concentration of the sugar solution after inversion, affect the Clerget divisor. It should be emphasized again that the prescribed technic must be rigidly adhered to in all Clerget procedures and that without the greatest care and precaution on the part of the analyst the methods cannot give accurate or even reproducible results.

485. Herzfeld-Schrefeld Modification of Clerget's Method. The Herzfeld inversion procedure is still widely used although it has been proved

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[8] The student is referred to Zerban's "Sucrose Determination by Double-Polarization, and the Clerget Divisor," prepared for the International Commission of Uniform Methods of Sugar Analysis, 1941, for a detailed study of the above factors and other considerations in this chapter. Also, "Polarimetry, Saccharimetry and the Sugars," *Bur. Standards Circ. C 440* (1942), pp. 126–164.

by Zerban and others to be unreliable in the ordinary cane sugar products such as are met with in factory control, particularly final molasses. The method as given below is adapted from the Official Methods of the Association of Official Agricultural Chemists, fifth edition, 1940, p. 494, but the Jackson-Gillis method IV given in Sec. 488 is recommended for raw sugar factory control work.

Dissolve 52 grams of substance in water in a 200-ml. flask; add lead subacetate for clarification, then 1–2 ml. alumina cream, dilute to mark, mix well, and filter. (For low-grade massecuites or molasses or other dark-colored material weigh 26 grams to 200 ml. or 300 ml., calculating results to the basis of the normal weight per 100 ml.) Delead by adding powdered sodium oxalate, a little at a time, avoiding an excess. (Sodium carbonate for deleading is objectionable as it may cause destruction of levulose and darkens the solution.) Filter again using dry kieselguhr to aid filtration. Add 50 ml. of this lead-free filtrate to a 100-ml. flask, make to mark, mix well, and polarize in a 200-mm. tube. The result times 2 is the direct reading *P*.

To a second 50-ml. portion in a 100-ml. flask add 25 ml. of water; then little by little, while rotating the flask, 10 ml. of hydrochloric acid 1.1029 sp. gr. 20°/4° C. (24.85° Brix at 20° C.). Heat a water bath to 70° C., place the flask, with the thermometer inserted, in the bath and agitate constantly until the contents of the flask reach 67° C., which should require from two and a half to three minutes. Then continue the heating of the solution for exactly five minutes from the moment the thermometer reaches 67°, during which time the temperature of the contents of the flask should rise to about 69° C. At the end of the five minutes plunge the flask into cold water and cool to about room temperature, leaving the flask in the cooling bath at least thirty minutes longer.

Wash the solution from the thermometer into the flask and complete the volume to 100 ml. at the temperature of the direct polarization. If the invert sugar solution is too dark colored it should be treated with successive small portions of metallic zinc dust after completion of the volume. The coloring matter is destroyed by the nascent hydrogen. No more zinc than is necessary should be used.

Make the invert polarization, using an inversion tube, Fig. 128. Circulate water of the temperature of the direct polarization through the water jacket of the tube during the operation. Note the polariscope reading (minus) and ascertain the temperature by inserting an accurate thermometer, graduated to ⅒° C., into the solution through the side tubule. If there is a possibility of mutarotation (see Sec. 449), the tube of solution should be allowed to stand several hours before polarizing. Reduce the polariscope reading to terms of a normal solution observed in a 200-mm. tube. (In the case as here given, reading times 2 equals invert reading *I*.) Calculate the sucrose by the following formula:

$$S = \frac{100(P - I)}{133 + 0.0676(m - 13) - 0.5(T - 20)}$$

in which S = sucrose per cent;
P = direct reading calculated to a basis of normal solution;
I = invert reading calculated to a basis of normal solution;
T = temperature at which readings are made;
M = grams of solids in 100 ml. of invert solution as read (solids by refractometer multiplied by specific gravity of solution).

It will be noted that in this formula m denotes total solids and not sucrose because it has been found by Browne, Zerban, and others that *water* concentration and not sucrose concentration regulates the Clerget divisor. For routine laboratory procedure the solids will be known with sufficient exactness from the composition of the material, if a refractometer is not at hand.

Recent work of Jackson and McDonald indicates that the concentration coefficient may be much higher than 0.0676.[4]

486. Inversion at Room Temperature. If time permits, the inversion should be carried out at room temperature. The Association of Official Agricultural Chemists recommends twenty-four hours if the inversion is not below 20° C. and ten hours if it is above 25°, but for low-purity products such as molasses and even for raw sugars a few hours longer at these temperatures is needed to insure complete inversion. As the twenty-four-hour period is usually the most convenient, inversion may be assured in this period by placing the flask in a warm place (28° C. See p. 407). When inversion is at room temperature the Clerget constant in the above calculation is 133.2 instead of 133.0.

487. Clerget's Method as Modified by Steuerwald.[5] The inversion is conducted at room temperature with increased acid strength.

Prepare the solution as described in the preceding paragraph. Measure 50 ml. of the filtrate into a 100-ml. flask and add 30 ml. of hydrochloric acid of 1.1 sp. gr. (acid of 1.188 sp. gr. diluted with an equal volume of water). Set aside three hours if the temperature is between 20° and 25° C. or two hours if above 25° C. Dilute the solution to 100 ml. and polarize, observing the same temperature conditions as have been described in the preceding paragraph. A different constant must be used in connection with the Herzfeld formula. The readings should be reduced to terms of the normal weight of the material in 100 ml. of solution before making the calculations by the following formula in which the letters have the same values as above.

$$S = \frac{100(P - I)}{135.53 + 0.0683(m - 13) - 0.53(T - 20)}$$

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[4] *J. Assoc. Off. Agr. Chem.*, **22**, 580 (1939).

[5] *Archief*, **21**, 831 (1913); *Intern. Sugar J.*, **15**, 489 (1913).

488. Jackson-Gillis Clerget Modification (Method IV). Jackson and Gillis of the U.S. Bureau of Standards made an extensive study of acid inversion methods[6] which resulted in the formulation of several modified methods using neutral salts in the direct polarization to offset the effect of the acid on the invert sugar in the invert polarization. This method has been shown by Zerban and collaborators to be correct only in those products which are free of amino compounds and since Ambler[7] has shown that practically all cane products contain appreciable quantities of amino compounds, it would appear that the method is objectionable for cane products. However, method IV has been adopted by the U.S. Customs Service and by practically every governing sugar association throughout the world, including the International Society of Sugar Cane Technologists whose "System of Factory Control" (p. 54) says, "Method IV comes closest to the result obtained by the invertase method although it averages somewhat higher. . . . Taking all the different factors into consideration the Committee has decided to adopt the Jackson-Gillis Method IV for sucrose determinations."

The general method as given by the authors is quoted below, but details of the amounts of substance to be taken and the preparation of solutions for juices, sugars, molasses, etc., will be given in the chapters devoted to the analysis of these materials.

Prepare a normal solution or a solution of such fractional normality as the nature of the substance and the sensibility of the saccharimeter requires. Make the solution to volume at the temperature at which the polariscopic observations are to be made. Clarify with the minimum quantity of dry basic lead acetate. Products of higher purity may be clarified by adding alumina cream before making to volume. Filter.

(If desired, the excess of lead may be removed at this point. Add pulverized potassium or sodium oxalate to complete precipitation of lead. The deleading reagent should be added to the whole filtrate. If the deleading is omitted, the lead is satisfactorily removed by the chlorides subsequently added.)

Pipette two 50 ml. portions of the clear filtrate into two 100 ml. flasks and add 20 ml. of water to each flask. To one portion add 10 ml. of a solution of sodium chloride containing 231.5 grams per liter; make to volume at the temperature at which the observations are to be made and polarize in a water-jacketed tube. (Reading calculated to normal weight basis = P.) To the other portion add hydrochloric acid and invert by the method of Herbert S. Walker as follows: Insert a thermometer in a flask and heat by immersing in a hot-water bath until the temperature is exactly 65° C. Remove the flask from the bath, add 10 ml. of hydrochloric acid (1.1029 sp. gr. at 20°/4° C. = 24.85° Brix), mix by rotating and set aside for thirty minutes. (Longer standing does not affect the results.) Cool and make to volume at the temperature at which the observations are to be made. Polarize in a water-jacketed tube with temperature carefully regulated to the same as that for direct polarization. (Reading calculated to normal weight basis = $-P'$).

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[6] *Bur. Standards Bull.* **376** (1920).

[7] *Intern. Sugar J.*, **29**, 437 (1927).

The Walker method of inversion described above may be insufficient to cause complete inversion in low-purity products, particularly in cool weather when cooling may be rapid. For final molasses it is recommended that 1 to 2 ml. of acid be added to the solution before heating to neutralize any excess of lead subacetate that may be present.[8] (See p. 407.)

489. Inversion at 60° C.[9] The U.S. Customs Regulations prescribe inversion as follows:

> Immerse in a water-bath which is maintained at exactly 60° C. Agitate the solution continually for 3 minutes and allow it to remain in the bath for a total time of 10 minutes. Cool quickly and make to volume at the temperature at which the observations are to be made. Shake and filter as rapidly as possible. Polarize. (See p. 407.)

490. Inversion at Room Temperature. Inversion at room temperature, as described in Section 486, is preferable if time permits. Jackson and Gillis have found thirty-one hours necessary for complete inversion at 20° C. and fifteen hours at 25° C. As stated previously the convenient twenty-four-hour period will insure complete inversion if the solution is allowed to stand in a warm place in the laboratory. (See p. 407.)

491. Calculations of the Sucrose by Jackson-Gillis Method IV. The Jackson-Gillis method IV as adopted by the U.S. Treasury Department and the International Commission for Uniform Methods (London, 1936) employs a table for the values of the Clerget divisor (as given on p. 796) based on the value $P - P'$, i.e., the sucrose concentration. Browne and others have shown that it is not the sucrose concentration, but the water concentration or what amounts to the same thing, the dry substance concentration, which determines the Clerget divisor, so the use of the table is incorrect. Nevertheless, it continues to be used by the Treasury Department for the U.S. Customs purposes and by most other laboratories because it is prescribed by the International Commission. The correct method of calculation is as follows:

For Walker method of inversion. $$S = \frac{100(P - P')}{132.63 + 0.0794(m - 13) - 0.53(t - 20)}$$

For 60° inversion the basic value is 132.56 instead of 132.63, and for room temperature 132.66. The rest of the formula is the same. The concentration coefficient 0.0794 is the value found by Jackson and McDonald[10] instead of the coefficient 0.0676 formerly used. The calculation may be greatly simplified by the use of simple tables which will be given under the specific directions for molasses, juice, and sugar analysis.

The use of the Jackson-Gillis table on p. 796 is as follows. Find opposite the algebraic sum of $P - P'$ (column 1), the value of the Clerget divisor (column 2). Apply the temperature correction. If the original solution was

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[8] *Bur. Standards Circ. C 440* (1942), p. 153.

[9] *Bur. Standards Circ. C 440,* p. 794.

[10] *J. Assoc. Off. Agr. Chem.,* **22,** 580 (1939).

of fractional normality, $P - P'$ must be multiplied by this fraction before selecting the value from the table. Divide the factor corrected for temperature into $P - P'$ to obtain the Clerget sucrose.

CLERGET METHOD USING INVERTASE[11]

Commercial invertase preparations are available on the market.[12] If it is desired to prepare the solution in the laboratory, the procedure described in Sec. 492 may be used. The laboratory preparation may be further purified and concentrated by the ultrafiltration method described in Sec. 493(*a*).

492. Preparation of Crude Invertase Solution. Mix yeast with water in the proportion of 10 lb. of compressed baker's yeast with 5 liters of water. Add 2 liters of toluene and stir thoroughly at frequent intervals during the first twenty-four hours. Allow to stand for seven days with occasional stirring and filter by gravity through large fluted papers. Mix the residue with 2 liters of water, filter, and combine the filtrates. Purify[13] by adding 15 grams of neutral lead acetate to each liter of extract and filtering on paper after all lead acetate has been dissolved. Complete the purification immediately by dialysis or by washing on the ultrafilter as directed in Sec. 493(*a*).

493. Preparation of a Collodion Ultrafilter. Dissolve 6 grams of pyroxylin or nitrocellulose in a mixture of 50 ml. of absolute alcohol and 50 ml. of absolute ether by first adding the alcohol to the cotton, allowing the mixture to stand in a stoppered flask for ten minutes, adding the ether, and shaking. Allow the solution to stand overnight, pour about 100 ml. into a 2000-ml. cylinder, and coat the entire inside surface of the cylinder with the collodion. Drain and dry for ten minutes. Fill with water, let stand ten to fifteen minutes, pour out the water, and remove the collodion sack. Test for leaks by filling with water. Slit open longitudinally and cut out a circular piece about 7–8 in. in diameter. Cut the bottom from a 2-liter bottle or Erlenmeyer flask and grind the edge smooth. Place it upon the still moist collodion disk, fold the edge of the disk up around the bottle, and cement it thereto with collodion that contains an increased percentage of ether. Place three or four thicknesses of wet filter paper in an 8-in. Buchner funnel. Place the bottle with the collodion membrane upon the filter paper. Pour melted petrolatum, to the depth of an inch, between the bottle and inside of the funnel. Provide the bottle with a small mechanical stirring device.

(*a*) *Washing and Concentration of Invertase Solution by Ultrafiltration.* Filter 4 liters of the partially purified solution through the ultrafilter, stirring continuously, until about 1 liter remains. Wash with distilled water introduced by means of a constant level device until the filtrate is colorless, 3 or 4 liters of wash water being required. During the entire process the invertase solution must be preserved with toluene.

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[11] *Assoc. Off. Agr. Chem., Official Methods,* fifth edition, 1940.

[12] Wallerstein Laboratories, 180 Madison Ave., New York City.

[13] *J. Ind. Eng. Chem.,* **16,** 562 (1924).

(*b*) *Determining the Activity of the Invertase Solution.* It is generally sufficient to test the activity of the invertase solution as follows. Dilute 1 ml. of the invertase preparation to 200 ml. Transfer 10 grams of sucrose (granulated sugar) to a sugar flask graduated at 100 ml. and 110 ml., dissolve in about 75 ml. of water, add 2 drops of glacial acetic acid, and dilute to the 100-ml. mark. To the 100 ml. of sugar solution add 10 ml. of the dilute invertase solution and mix thoroughly and rapidly, noting the exact time at which the solutions are mixed. At the termination of exactly sixty minutes make a portion of the solution just distinctly alkaline to litmus paper with anhydrous sodium carbonate and polarize in a 200-mm. tube at 20° C. If the invertase solution is sufficiently active, the alkaline solution will polarize approximately 31° V. without correcting for the dilution to 110 ml. and the optical activity of the invertase solution.

494. Determination with Invertase. (*a*) *Direct Reading.* Dissolve the double normal weight (52 grams) of the substance in water in a 200-ml. flask; add basic lead acetate solution carefully, avoiding any excess, then 1 to 2 ml. alumina cream; shake; dilute to the mark with water; mix well; and filter, rejecting at least the first 25 ml. of the filtrate. Cover the funnel with a watch glass. When sufficient filtrate has collected, remove the lead from the solution by adding anhydrous sodium carbonate,[14] a little at a time, avoiding any excess; mix well; and filter again, rejecting at least the first 25 ml. of the filtrate. (Instead of weighing 52 grams into a 200-ml. flask, two 26-gram portions may be diluted to 100 ml. each and treated exactly as described. Depending on the color of the product, multiples or fractions of the normal weight may be used, and the results reduced by calculation to the basis of 26 grams in 100 ml.). Pipet one 50-ml. portion of the lead-free filtrate into a 100-ml. flask, dilute with water to the mark, mix well, and polarize in a 200-mm. tube. The result, multiplied by 2, is the direct reading (*P* of formula given below) or polarization before inversion. (If a 400-mm. tube is used, the reading equals *P*.)

(*b*) *Invert Reading.* First determine the quantity of acetic acid necessary to render 50 ml. of the lead-free filtrate distinctly acid to methyl red indicator; then to another 50 ml. of the lead-free solution in a 100-ml. volumetric flask add the requisite quantity of acid and 5 ml. of the invertase preparation. Fill the flask with water nearly to 100 ml. and let stand overnight (preferably at a temperature not less than 20° C.). Cool, and dilute to 100 ml. at 20° C. Mix well and polarize at 20° C. in a 200-mm. tube. If the analyst is in doubt as to the completion of the hydrolysis, allow a portion of the solution to remain for several hours and again polarize. If there is no change from the previous reading, the inversion is complete, and the reading and temperature of the solution should be carefully noted. If it

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[14] The use of sodium carbonate is objectionable as it may destroy levulose and also tends to darken the solution. Dry potassium oxalate is preferable or dry ammonium dihydrogen phosphate (*Intern. Soc. Sugar Cane Tech.*, "System of Factory Control," 1942, p. 54).

is necessary to work at a temperature other than 20° C., which is permissible within narrow limits, the volumes must be completed and both direct and invert readings must be made at the same temperature. Correct the invert reading for the optical activity of the invertase solution and multiply by 2. Calculate the percentage of sucrose by the following formula:[15]

$$S = \frac{100(P - I)}{132 + 0.073(m - 13) - 0.5(T - 20)}$$

in which S = percentage of sucrose;
P = direct reading, normal solution;
I = invert reading, normal solution;
T = temperature at which readings are made;
m = grams of total solids in 100 ml. of the invert solution read in the polariscope. (Solids by refractometer multiplied by specific gravity of solution.) See p. 402.

COMPLETE ACID INVERSION OF FINAL MOLASSES

In the directions for acid inversion (Secs. 486, 488, 489, 490) some doubt is cast on the time required for complete inversion of low grade products, particularly final molasses. Quite recently Zerban, Mull, and Martin[16] studied the special conditions required for complete acid inversion in Cuban and Puerto Rican blackstraps and reported the following conclusions: (1) For complete inversion in 24 hours the average blackstrap must be kept at 28° C. (2) Heating at 60° for ten minutes (U.S. Customs Regulations, see Sec. 489) is not sufficient and heating time must be extended to 15–20 minutes. (3) Heating to 70° C. for 8 minutes (modification of Herzfeld method, p. 401) gives results checking inversion for 24 hours at 28° C.

This study casts doubt on the Walker method of inversion recommended by Jackson and Gillis (Sec. 488), even with the extra acid suggested on p. 404. Overheating, even too high room temperature (e.g., 35° C.), is to be avoided or destruction of invert sugar will result. For tropical laboratories the 24-hour inversion should therefore be carried out in a relatively cool place.

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[15] The concentration factor 0.073 is considerably lower than the 0.082 factor found by Jackson and McDonald. (See *Bur. Standards Bull. C 440,* p. 134.)

[16] *J. Assoc. Off. Agr. Chem.,* February, 1944.

CHAPTER 20

CHEMICAL METHODS IN SUGAR ANALYSIS

DETERMINATION OF REDUCING SUGARS

495. General Introduction. The chemical methods of sugar analysis depend upon the property that dextrose, levulose, and other so-called reducing sugars have, in alkaline solutions, of reducing copper in the cupric state to cuprous oxide. Under specified conditions, the amount of copper reduced is in proportion to the quantity of the reducing sugars present.

Methods and modifications of methods show a wide diversity. Smolenski in the Report of the Proceedings of the 9th Session of the International Commission for Uniform Methods at London (1936) [1] gives a chart showing twenty-nine different methods "used or recently proposed." In this chapter the methods cited are those which have had wide use and acceptance in the cane sugar industry. Space does not permit the inclusion of alternative methods or ones whose usefulness has proved doubtful. In the chemical methods of determining sugars (frequently called glucose [2] determinations), it is essential to comply strictly with the directions for the analysis since a change in the conditions renders the tables for the calculations useless. In selecting the reduction method it must be taken into account that there is a slight reduction of copper by sucrose in the presence of invert sugar, so consideration must be given to the relative amounts of sucrose and reducing sugars in the material to be analyzed.

496. Analytical Methods in General. The successive steps involved after the selection of the method suitable for the material to be analyzed are: the preparation of the solution; the precipitation of the copper by boiling a measured portion of the prepared solution with a fixed amount of Soxhlet's modification of Fehling's alkaline copper tartrate solution (for some special cases other copper solutions are used); a means of determining the amount of copper precipitated in the gravimetric methods or of determining when the reduction is complete in the volumetric methods; and finally the calcu-

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[1] Supplement *Intern. Sugar J.*, January, 1937.

[2] *Note.* It is customary in the cane sugar industry to use the word "glucose" as synonymous with reducing sugars. Refiners use the term "invert" or "invert sugar" to mean the same thing. The term "reducing sugars" is used generally throughout this book in accordance with the terminology recommended by the International Society of Sugar Cane Technologists. The results of the determinations are expressed as invert sugar (equal parts of dextrose and levulose) although these two sugars may be present in other proportions.

lation of the quantity of invert sugar present by means of tables or formulas suitable to the method employed.

To determine sucrose by chemical methods it is first necessary to change it into invert sugar by hydrolysis with acids or with invertase. If reducing sugars are already present these must be determined before the inversion of the sucrose and deducted from the total found after inversion, the sucrose being calculated from this difference. (See p. 430.)

Specific directions as to which of the methods described later is to be employed with juices, sirups, molasses, sugars, etc., will be given in the special chapters devoted to the analysis of these materials.

497. Preparing Solutions for Determinations. The proper preparation of the solution for the determination of reducing sugars is of prime importance no matter what method is selected. Unfortunately, there is still considerable difference of opinion regarding the need for defecation in the various cane sugar products, and it would seem that there should be more investigation on the subject. It has long been known that lead subacetate cannot be used as a defecant because of the precipitation of a part of the reducing sugars, largely levulose, by this basic salt. The use of neutral lead acetate as a clarifying agent is more acceptable but extensive studies by Meade and Harris [3, 4] at the Central Control Laboratory (Cardenas, Cuba) of the Cuban-American Sugar Company showed that the directions as to the quantity of lead to be used and the deleading agents to be employed allowed wide latitude and gave discordant results. Such directions as add "sufficient lead to clarify, and delead with sodium sulfate, carbonate or oxalate" were all that were given in standard textbooks at the time these investigations were made. Within the limits of such directions differences of 1 per cent of the reducing sugars present in a Cuban molasses could be found by the same method of determination. The investigations at Cardenas brought out the following points for work done on Cuban molasses and Cuban raw sugars.

1. The amount of normal lead acetate solution must be accurately measured and must be the minimum amount required to obtain a clear filtrate. Increasing the lead decreases the amount of reducing sugars found.

2. Sodium sulfate and sodium carbonate should never be used as deleading agents as they give too low results.

3. Potassium oxalate and sodium phosphate in solution, and dry sodium oxalate, used as deleading agents give results checking each other though slightly higher than results using no lead whatever.

4. Where lead and deleading agents are used, if the lead precipitate is filtered off and the filtrate deleaded it will give lower results than if the deleading agent is added to the leaded solution, and the combined precipitates are filtered out together.

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[3] Meade and Harris, *J. Ind. Eng. Chem.*, **8**, 504 (1916)

[4] Harris, *J. Ind. Eng. Chem.*, **13**, 925 (1921).

5. Sodium phosphate and phosphoric acid are the only deleading agents that completely remove the lead. A small amount of lead left in solution does not disturb the precipitation of the copper; a large excess gives low results.

6. Clarification without lead, using kieselguhr only, gives consistent results and is a practical and simple method.

Clarification with kieselguhr without the use of lead has been employed for over twenty years in the various laboratories of the Cuban-American Sugar Company with constant cross checking showing excellent results. It must be remembered in using this method that the thin solution without lead or other preservative will deteriorate quickly, so determinations must be made immediately after the material is dissolved and filtered.

The only objection to this method is that there may be present copper-reducing non-sugars precipitable by lead. In the work cited molasses treated with normal lead acetate and deleaded with sodium or potassium oxalate invariably gave results a little *higher* (1 to 2 mg. of copper) than are obtained with the no-lead and kieselguhr clarification; while the same molasses treated with the *oxalate only* and kieselguhr gives the same high result as the lead and oxalate, indicating that the disturbing influence is the oxalate and not the lead.

Eynon and Lane [5] proved that the presence of even small quantities of calcium salts is sufficient to cause a considerable decrease in the reducing power of reducing sugars, particularly in the volumetric method bearing their name (Sec. 506). They recommend the removal of the calcium salts with sodium or potassium oxalate. Their findings show the reason for the higher results by Meade and Harris where oxalate was used. Since all cane products, particularly low-purity materials, contain some lime salts, it seems advisable where no lead is used to add some dry sodium oxalate with the kieselguhr before filtering. One-tenth of a gram per gram of molasses is sufficient, or if preferred 1 ml. of 10 per cent potassium oxalate solution per gram of molasses may be added before making to the mark. A moderate excess of oxalate does not affect the copper-reducing powers. Eynon and Lane also found no indication of copper-reducing non-sugars precipitable by lead in the molasses and sugars they investigated, as evidenced by the fact that the use of lead for clarification and deleading with oxalates gave higher results than with no clarification at all (except kieselguhr). Oxalate alone checked their results with lead plus oxalate, proving that the lead had no effect on the reduction but that the oxalates removed the disturbing calcium salts.

W. A. Davis of the Distillers Company, Ltd., Epsom, England,[6] recommends that no defecation whatever, either with lead acetate solution or oxalate for decalcification, be used for molasses. He quotes figures from Eynon and Lane on molasses from various countries to show that this method gives results in close agreement with those when defecation with lead and

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[5] *Intern. Sugar J.*, **25**, 305 (1923).

[6] *Methods of Analysis of Molasses Used in the Fermentation Industries*, 1938.

oxalate solution are used. Results by Lever comparing the Eynon and Lane method with the Munson and Walker[7] and other methods is of interest because he used lead and oxalate defecation with Eynon and Lane method, and filtration with kieselguhr with the Munson and Walker and other methods. With eleven samples of final molasses from eight different countries he found six gave higher results with Eynon and Lane and lead defecation than with the Munson and Walker method without defecation (+0.12 to +0.64 average +0.35). Two were in almost exact agreement, and three gave lower results (−0.18 to −0.32 average −0.23). If the Eynon and Lane method is assumed to give comparable results with the Munson and Walker method (as many investigators have found) Lever's results indicate that there is no reason for the lead defecation.

In contradistinction to these findings and those brought out at Cardenas, Cook and McAllep[8] working with Hawaiian molasses report that they found:

> . . . decidedly lower results after treatment with lead acetate. That this was due to removal of non-sugar reducing substances was proved as follows: lead precipitate was absolutely freed from sugar by washing, decomposing by H_2S, and reprecipitating with neutral lead acetate after boiling out excess of H_2S, this process being repeated several times. The sugar-free precipitates were finally deleaded in several different ways, filtered, the filtrate added to Soxhlet solution, and heated under the conditions of the glucose determination. Copper was reduced in all cases. The amounts of these reducing non-sugars are sufficient to affect the results, hence lead clarification is necessary.

They corroborated the facts that lead is completely removed by disodium phosphate whereas lime is not, and that the oxalates remove all the lime but not all the lead. They recommend the use of a mixture of these salts in the proportion of 7 grams of disodium phosphate and 3 grams of potassium oxalate to 100 ml., using 1 ml. of this mixture for each gram of molasses. By this mixed solution all lead and all lime are completely removed.

498. Recommendations for Preparation of Solutions. The practical arguments for omitting the lead seem to this writer to be many. (1) The preparation is simpler to carry out. (2) The omission of the lead leaves no option (as to quantity of reagents) to the operator. (3) It has been shown that, in general, the difference with or without lead is not great.

The only argument for the use of lead is the presence of lead precipitable reducing substances. It cannot be denied that many responsible investigators have found indications of such substances, although the work of Meade and Harris, Eynon and Lane, and Lever quoted above tend to show that they are negligible in many types of molasses. But the question arises as to why a "minimum of lead" is always specified by the advocates of lead defecation. Meade and Harris (and unpublished work by Meade) show that increasing the amount of lead (within reasonable limits) frequently gives lower results, the decrease being much more marked with some samples of molasses than

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[7] *Intern. Sugar J.*, May, 1936, p. 181.

[8] *Facts About Sugar*, **13**, 298 (1928).

with others. If the purpose of the lead is to remove precipitable matter it would seem that the lead should be added in sufficient quantity to give the *lowest* possible result, i.e., until all precipitable matter is removed. If this is not done then the results by different analysts using a discretionary amount of lead may differ.

Another point to be emphasized is that, if lead is used, two filtrations should be employed, one after leading and the other after deleading. The easier practice is to add the lead and then the deleading agent without an intermediate filtration, but this will give higher results than if the two-filtration method is used.

No matter what method of preparation of the solution is selected, it should be rigidly adhered to in all classes of determinations, otherwise comparable results cannot be obtained for use in calculating the "glucose balance" of the factory or refinery. Some authorities advocate treatment with kieselguhr only without lead for raw sugars and defecation with lead for molasses, e.g., the Committee on Uniformity of the International Society of Sugar Cane Technologists says a minimum of lead may be used with molasses but that no clarification is required for raw sugars. The use of an oxalate to remove lime salts is imperative in all cases whether lead is used or not. The difference with raw sugars, using lead or no lead, may appear small but in percentage of the invert sugar present it is about as great as with molasses. For deleading and decalcifying when lead is used, the phosphate-oxalate solution of Cook and McAllep (see above) should be used.

499. Copper Reagent. All the methods to be described here (with the exception of the Luff-Schorl and Kraisy methods, p. 427) use the Soxhlet modification of Fehling's solution. This will be understood whether referred to as "Fehling's," "Soxhlet," or merely "the copper solution." It consists of two separate solutions which are mixed in equal volumes just before the analysis.

a. Copper Sulfate Solution. Dissolve 34.639 grams of copper sulfate ($CuSO_4 \cdot 5H_2O$) in water, dilute to 500 ml., and filter through prepared asbestos.

b. Alkaline Tartrate Solution. Dissolve 173 grams of Rochelle salts (sodium potassium tartrate) and 50 grams of sodium hydroxide in water, dilute to 500 ml., allow to stand two days, and filter through prepared asbestos.

If pure chemicals are used in the preparation of these solutions, filtration is not necessary.

GRAVIMETRIC METHODS FOR REDUCING SUGARS

500. Introductory. In the gravimetric methods the precipitated cuprous oxide is filtered out of the solution after the reduction (an excess of copper always remaining in the filtrate) and the copper is determined by any one of the methods to be enumerated later. From the amount of copper found the weight of reducing sugars is determined from tables which have been calculated for the method employed. The amount of precipitated copper may be arrived at volumetrically, but this does not make the method a volumetric glucose as generally understood. The volumetric methods depend upon determining the amount of the solution required to effect complete reduction in a fixed amount of Soxhlet solution.

501. Herzfeld Method. (*Using Meissl-Hiller Factors.*) Extended experience has proved this to be an excellent method for all classes of cane sugar products, and its use is recommended for general routine work except for high-test sugars containing little invert sugar. The first step in the analysis after the preparation of the sample is the estimation of a suitable quantity of the material for the test. This need be done only for materials in which the approximate amount of reducing sugars is not known, which is not generally the case in sugar house routine work. Prepare a series of large test tubes by adding successively 1, 2, 3, 4, and 5 ml. of the solution prepared as described above. Add 5 ml. of mixed Soxhlet's solution to the contents of each tube and heat to boiling during about two minutes. Allow the precipitates to settle. Compare the color of the supernatant liquid in each tube and note that which has the lightest tint, but is distinctly blue. Measure 20 times the volume of the prepared solution that this tube contained into a 100-ml. flask and dilute it to the mark with water. For example, tube 3 is selected; 3 × 20 = 60 ml. of the original solution to be made up to 100 ml. Use 50 ml. of this solution for the determination.

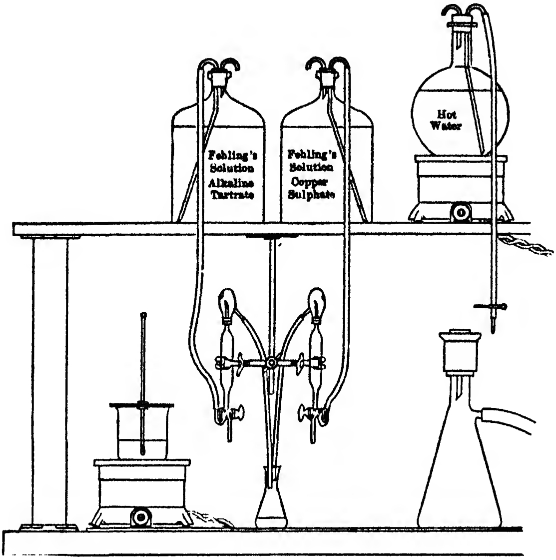

FIG. 137. Arrangement for Reducing Sugar Determinations.

Reduction to Cuprous Oxide. Measure 50 ml. of mixed Soxhlet solution (Sec. 499), i.e., 25 ml. of the copper solution and 25 ml. of the alkali solution into a 400-ml. Pyrex or other alkali-resistant beaker and add 50 ml. of the properly prepared sugar solution. Heat the contents of the beaker to the boiling point, taking approximately four minutes to reach this temperature, and continue the boiling with gentle ebullition for exactly two minutes. At the conclusion of the heating period add 100 ml. of *cold recently boiled* distilled water, and immediately filter and collect the cuprous oxide, using one of the methods described farther on.

The use of a thermometer to determine the beginning of the boiling period was recommended by Harris [9] and has been found to increase accuracy as well as to aid in the regulation of the pre-heating to the required four minutes. The thermometer is thrust through a rubber stopper which is held in the aperture of a perforated watch glass of suitable size to cover the 400-ml. beaker. The point of the thermometer should reach to within 5 mm. of the bottom of the beaker when the cover glass is in place. (See Fig. 137.) The moment when the thermometer registers 100° C. is taken as the beginning of the two-minute boiling period, although local ebullition sometimes starts before this temperature is reached and in other instances there is no evidence of boiling when the thermometer reaches 100°. An interval timer or accurate sand glass may well be used for timing the two-minute interval.

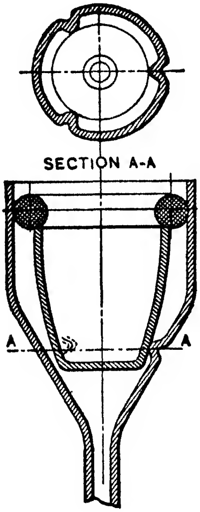

FIG. 138. Spencer Crucible Holder.

All the details of the method of preparing the solution and conducting the reduction must be strictly adhered to in order that the results may be comparable. The beakers should be of Pyrex, or similar glass, and all of one size, preferably not larger than 400 ml., and of uniform thickness and diameter. The boiling should not be violent but only just apparent. The addition of the cold-distilled water at the completion of the two-minute boiling period should be prompt, and the filtration should be done immediately.

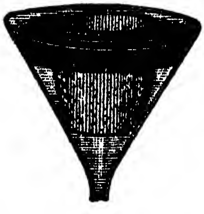

FIG. 139. Sargent Crucible Holder.

The Filtration of the Cuprous Oxide. The invention of the alundum filtering crucible and Spencer's method of making the joint at the rim of the crucible instead of the bottom as with the Gooch crucible have greatly simplified this stage of the analysis. Other methods of filtration than with alundum are given for use in the absence of alundum ware.

a. Provide a Spencer funnel (Fig. 138) or Sargent's alundum crucible holder (Fig. 139). The funnel and holder must be of the proper size, otherwise the

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[9] Meade and Harris, *J. Ind. Eng. Chem.*, 8 (1916), p. 504.

crucible itself will trap the washings and prevent prompt filtration. A brass funnel for use with the Sargent holder which permits very free filtration is shown in Fig. 140. A brass Spencer funnel may be readily made by sugar factory mechanics, and is entirely satisfactory. The upper walls of the funnel must be parallel. The rubber rings are carried in stock by the dealers. These should be of pure soft rubber and of about ¼-in. cross section. The funnel is placed in a suction-filtering heavy Erlenmeyer flask with side tubule. The suction is obtained by a filter pump or preferably through a pipe communicating with the vapor pipe of the multiple-effect evaporator. The alundum[10] crucibles require a very efficient filter pump on account of their large filtering area.

The 25-ml. alundum crucible, porosity R A 360, has been found most satisfactory. New crucibles should be soaked in dilute nitric acid, thoroughly washed with hot water, then soaked in hot mixed Soxhlet's solution, and again thoroughly washed with hot water. Reverse washing is advisable both for new crucibles and for used crucibles between tests. The funnel shown in Fig. 140 is convenient for this purpose. Remove the Sargent holder and the spring brass cross in the bottom of the funnel and place the crucible upside down on the rubber stopper. When suction is applied and hot water sprayed on the outside of the crucible the water is drawn through in the reverse direction from the filtration, freeing the pores of the alundum of all foreign matter. The crucibles can be freed of contained copper by soaking in hot dilute nitric followed by thorough washing. All crucibles after washing should be dried in an oven heated over a flame or preferably in an electric muffle and stored in a desiccator before use.

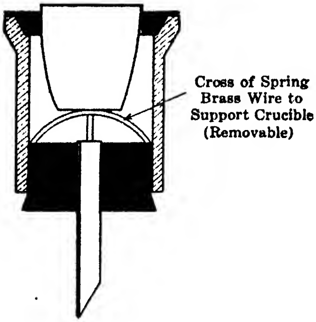

Fig. 140. Brass Funnel for Use with Sargent Holder.

A Gooch crucible with asbestos mat may be used in place of alundum. The asbestos should first be digested for several days in dilute (1 : 3) hydrochloric acid washed free of acid, then digested for the same length of time with 10 per cent NaOH solution after which it is treated for several hours with hot alkaline tartrate solution. Then digest in nitric acid (1 : 3), wash free of acid with water, and shake fiber up in water. About ¼-in. asbestos mat is recommended in the crucible. The asbestos fiber improves with use. The copper adhering to the fiber is dissolved with strong nitric acid after which the fiber is thoroughly washed with hot water. A convenient form of laboratory layout for gravimetric determinations is shown in Fig. 137.

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[10] Alundum crucibles may be substituted for platinum in most of the analytical work of the factory and agricultural laboratories. These lose weight very slowly in glucose work, owing to the action of the alkali and the acid used for washing but remain practically at constant weight for the duration of the test.

Immediately after the reduction to cuprous oxide, this precipitate is collected in the alundum crucible and is thoroughly washed with hot water. The crucible should be filled only about half full during filtration. The sub-oxide distributes itself on the walls of the crucible. The washing may be followed by moistening the oxide and crucible with pure alcohol to expedite the drying. Dry the crucible in an oven or cautiously over the flame of a lamp, according to the form in which the copper is ultimately to be weighed. Proceed by one of the following methods:

502. Determination of the Copper. *Wedderburn's Method of Reduction to Metallic Copper.*[11] This is the simplest of the methods involving reduction to metallic copper, and its results are nearly as accurate as those by electrolysis and equal to reduction in hydrogen. Bend the wires of a pipe stem or silica triangle to form a tripod support for the alundum crucible. Place the tripod in a metal beaker or other convenient metallic vessel. Cover the bottom of the beaker to a depth of about 1 cm. with alcohol. Denatured alcohol will serve. Place the beaker, covered with a watch glass, on a hot plate and warm the alcohol until its vapors condense on the under side of the cover glass. Heat the crucible to full redness to burn off organic matter that may have been carried down with the cuprous oxide; remove it from the flame and let it cool until the redness almost disappears; remove the cover from the beaker and place the crucible on the tripod and replace the cover. The oxide of copper is almost instantly reduced to metallic copper in the atmosphere of alcoholic vapor and adheres firmly to the walls of the crucible. The object of cooling the crucible to very faint redness is to prevent setting fire to the alcohol. If the alcohol should take fire the flames are readily extinguished by blowing upon them after covering the beaker. The beaker should be removed from the hot plate a moment after introducing the crucible. It is necessary to let the crucible cool for three or four minutes in the vapor of alcohol, after the reduction to avoid reoxidation of the copper. Should the crucible become quite cold, it should be moistened with pure alcohol and this be burned off. After cooling in a desiccator the crucible is weighed and the weight of copper is ascertained by difference. The whole operation consumes but five or six minutes, and the copper plating is as good as that obtained by electrolysis.

Wedderburn's method may be conducted with a Gooch crucible, but the alundum ware is more convenient. An error may enter from occluded ash from the test solution or from carbon from the alcohol, but tests show this error to be very small and usually negligible. The calculation of the reducing sugars is given farther on. This method has been adopted as official by the U.S. Customs Service.[12]

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[11] *J. Ind. Eng. Chem.*, **7**, 610. Original method, Vladimir Stanek, *Z. Zuckerind. Boehmen*, **32**, 497; Votoček and Laxa, Abstract in *Chem. Zeit. Chem. Repertorium*, **21**, 324.

[12] *Bur. Standards Circ. C 440*, p. 179.

Electrolytic Method in Nitric Solution. Determining the copper electrolytically is never practiced in routine control tests. The practical accuracy of the various indirect methods has been demonstrated by numerous investigators so that electrolysis is resorted to only for checking methods and for research work.

Collect the cuprous oxide in an alundum crucible as described in Wedderburn's method, except that a glass funnel must be used and the crucible need not be tared. After washing the oxide very thoroughly, change the receiving vessel for the filtrate. Let 4 ml. of concentrated nitric acid fall drop by drop upon the oxide, being careful that all parts of the latter are wetted by the acid. Follow the acid with a jet of hot water from a wash bottle and wash the walls of the crucible thoroughly. Should any of the red oxide remain, repass the acid filtrate through the crucible. The oxide may conveniently be reduced to metallic copper as in Wedderburn's method, and this may be dissolved instead of the oxide. Transfer the filtrate to a 250-ml. beaker and dilute it to approximately 100 ml.

If a Gooch crucible is used for the collection of the cuprous oxide, the asbestos mat and adhering oxide should be transferred from the crucible to the beaker by means of a glass rod. Dissolve any remaining oxide in the crucible with about 2 ml. of strong nitric acid from a pipet and rinse thoroughly with hot water, allowing the acid and rinsings to flow into the beaker with the asbestos and dissolved copper. Heat the contents of the beaker until all copper is dissolved, filter through another prepared Gooch and wash thoroughly, diluting the filtrate to about 100 ml. as in the case of the alundum.

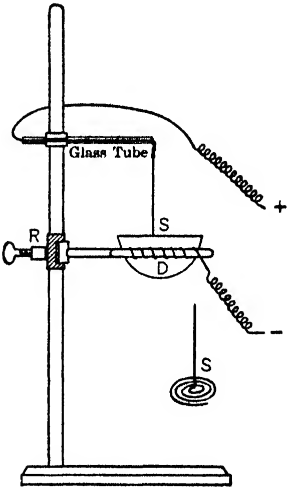

FIG. 141. Apparatus for Electrolytic Copper Determinations.

A convenient electrolytic apparatus may be made up in most laboratories with a large platinum dish (125–150 ml.), a heavy platinum wire bent in a flat spiral *S* as shown in Fig. 141, and a storage battery, either of the automobile or radio type in conjunction with a suitable rheostat. Connect the dish, *D*, containing the dissolved copper with the negative pole of the battery by placing it on a ring wrapped with the bare copper lead-wire. Insulate the ring from the support by means of a piece of rubber tubing, *R*. When the spiral anode, *S*, is connected with the positive pole, the copper will deposit on the inside of the dish. A current of 0.5 to 1.0 ampere is sufficient with stationary anode, the deposition of copper being hastened and made more even by having the solution heated to about 60° C. at the beginning of the electrolysis. Under these conditions the determination requires from one to three hours, depending on the amount of copper present. Rotation of the anode will greatly increase the rapidity of the operation, and permits the use of 3 to 5 amperes current. If direct current from a lighting circuit is available it is the most convenient source for electrolytic work, using an ordinary rheostat or a bank of incandescent lamps to regulate the amount. Toward the end of the electrolysis, which becomes evident by the disappearance of the color, the solution should be tested for copper from time to time by withdrawing a drop and adding it to a little ammonia to neutralize the acid, then acetic acid to acidity, and finally a drop of potassium ferrocyanide solution, using a white porcelain plate to hold the solutions. When this solution no longer reacts for copper, i.e., does not turn brown when the ferrocyanide is added, without cutting off the electric current, withdraw the acid solution with a large pipet or by syphoning, at the same time replacing it with water. Repeat this operation until all the acid has been removed, then break the current, remove and dip the cylinder in pure alcohol and then in ether, and dry it in an oven for a few minutes. The current must not be discontinued so long as a trace of acid remains. The dish is now weighed, its increase of weight being due to the metallic copper deposited.

Weighing the Copper as Cuprous Oxide. The red oxide collected on the alundum or Gooch crucible is completely washed with hot water as before and is then moistened with strong alcohol followed by a little ether, after which the crucible is heated in an oven at approximately 100° C. and dried thirty minutes, cooled, and weighed. The weight of Cu_2O times 0.888 equals the weight of copper. This method has little to recommend it and should never be used, although some authorities permit it for materials of high purity. Work with 96° raw sugar[13] showed that results calculated from cuprous oxide were 10 per cent higher than those calculated from cupric oxide or metallic copper. The results obtained by weighing as cuprous oxide with raw sugars were not much more accurate, based on percentage of the invert sugar content, than when working with final molasses.

Weighing the Copper as Cupric Oxide. The crucible containing the red oxide is thoroughly washed with hot water as before. It is then dried and heated to full redness for fifteen minutes in a muffle furnace to oxidize the cuprous oxide to cupric. It is cooled in a desiccator and weighed as quickly as possible as the black oxide is very hygroscopic. The weight of CuO times 0.8 equals the weight of copper reduced.

This is an excellent method for routine work and long experience with it has proved its accuracy. It is good practice to determine the copper as cupric oxide first and then to reduce the oxide to metallic copper by the Votoček-Wedderburn method and compare the two results, each method thus checking any possible error in the other. The metallic copper generally shows a fraction of a milligram higher, even in the most accurate work, owing to occluded carbon from the alcohol.

Low's Volumetric Thiosulfate Method. This is a highly accurate method for determining the quantity of copper precipitated which is used in investi-

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[13] Meade and Harris, *loc. cit.*

gations to check other methods. It is used in some industries for routine work, and in the hands of a trained operator it is rapid as well as accurate.

1. Standardization of the thiosulfate solution.[14] Prepare a solution of sodium thiosulfate containing about 19 grams of the pure crystals to the liter. Standardize as follows: Weigh accurately about 0.2 gram of pure copper foil and place in a flask of about 250 ml. capacity. Dissolve by warming with 5 ml. of a mixture of equal volumes of strong nitric acid (sp. gr. 1.42) and water and then dilute to about 50 ml. Boil for a few minutes to expel most of the red fumes and then add 5 ml. of strong bromine water and boil until the bromine is thoroughly expelled. The bromine is to insure the complete destruction or removal of the red fumes. Remove from the heat and add a slight excess of strong ammonia water. Ordinarily it suffices to add 7 ml. of ammonia water of 0.90 sp. gr. Again boil until the excess of ammonia is expelled, as shown by a change of color of the liquid and a partial precipitation of the copper as hydroxide or oxide. Now add strong acetic acid in slight excess, perhaps 3 or 4 ml. of the 80 per cent acid in all, and again boil for a moment if necessary to redissolve the copper precipitate. Cool to room temperature and add about 3 grams of potassium iodide or 6 ml. of a solution of the salt containing 50 grams in 100 ml. Cuprous iodide will be precipitated and iodine liberated according to the reaction,

$$2Cu(C_2H_3O_2)_2 + 4KI = Cu_2I_2 + 4KC_2H_3O_2 + 2I$$

The free iodine colors the mixture brown. Titrate at once with the thiosulfate solution until the brown tinge has become weak and then add sufficient starch liquor to produce a marked blue coloration. Continue the titration cautiously until the color due to free iodine has entirely vanished. The blue color changes toward the end to a faint lilac. If at this point the thiosulfate is added drop by drop and a little time is allowed for complete reaction after each addition, there is no difficulty in determining the end point within a single drop. One milliliter of the thiosulfate solution will be found to correspond to about 0.005 gram of copper. The reaction between the thiosulfate and the iodine is:

$$2Na_2S_2O_3 + 2I = 2NaI + Na_2S_4O_6$$

The starch liquor may be made by boiling about 0.5 gram of starch with a little water and diluting with hot water to about 250 ml. The liquor should be homogeneous and free from lumps. It should be used cold and must be prepared frequently.

2. Analysis. After washing the precipitated cuprous oxide, cover the Gooch crucible with a watch glass and dissolve the oxide by means of 5 ml. of warm nitric acid (1:1) poured under the watch glass with a pipet. Catch the filtrate in a flask of 250 ml. capacity and wash watch glass and Gooch crucible free of copper; 50 ml. of water will be sufficient. Boil to expel red fumes, add 5 ml. of bromine water, boil off the bromine, and proceed as in standardizing the thiosulfate.

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[14] *Bur. Standards Circ. 44*, p. 88.

Calculation of the Percentage of Invert Sugar for Herzfeld's Method by Meissl-Hiller Factors. If the material analyzed contains more than 1.5 per cent glucose and less than 98.5 sucrose in the contained solids calculate the percentage of glucose by the formula and factors given below. Tables have been expanded for products of known composition by E. W. Rice and others from these factors and formula, so that the tedious calculation is avoided for most routine work. (See Tables 42, 43, 44 on pp. 785–791.)

Let Cu = the weight of copper obtained;
P = the polarization of the sample;
W = the weight of the sample in the 50 ml. of the solution used for the determination;
F = the factor obtained from the table for the conversion of copper to invert sugar;
$\frac{Cu}{2}$ = approximate absolute weight of invert sugar = Z;
$Z \times \frac{100}{W}$ = approximate per cent of invert sugar = y;
$\frac{100P}{P + y}$ = R, relative number for sucrose;
$100 - R$ = I, relative number for invert sugar;
$\frac{CuF}{W}$ = per cent of invert sugar.

Z facilitates reading the vertical columns; and the ratio of R to I, the horizontal columns of the table, for the purpose of finding the factor F for the calculation of copper to invert sugar.

Example. The polarization of a sugar is 86.4 and 3.256 grams of it (W) is equivalent to 0.290 gram of copper. Then:

$$\frac{Cu}{2} = \frac{0.290}{2} = 0.145 = Z$$

$$Z \times \frac{100}{W} = 0.145 \times \frac{100}{3.256} = 4.45 = y$$

$$\frac{100P}{P + y} = \frac{8640}{86.4 + 4.45} = 95.1 = R$$

$$100 - R = 100 - 95.1 = I = 4.9$$

$$R : I = 95.1 : 4.9$$

By consulting the table it will be seen that the vertical column headed 150 is nearest to Z, 145, and the horizontal column headed 95 : 5 is nearest to the ratio of R to I, 95.1 : 4.9. Where these columns meet we find the factor 51.2 which enters into the final calculations:

$$\frac{CuF}{W} = \frac{0.290 \times 51.2}{3.256} = 4.56 \text{ per cent of invert sugar}$$

MEISSL AND HILLER'S[15] FACTORS FOR THE DETERMINATION OF MORE THAN 1 PER CENT OF INVERT SUGAR

Ratio of Sucrose to Invert Sugar = $R : I$	Approximate Absolute Weight of Invert Sugar = Z						
	200 mg. per cent	175 mg. per cent	150 mg. per cent	125 mg. per cent	100 mg. per cent	75 mg. per cent	50 mg. per cent
0 : 10	56.4	55.4	54.5	53.8	53.2	53.0	53.0
10 : 90	56.3	55.3	54.4	53.8	53.2	52.9	52.9
20 : 80	56.2	55.2	54.3	53.7	53.2	52.7	52.7
30 : 70	56.1	55.1	54.2	53.7	53.2	52.6	52.6
40 : 60	55.9	55.0	54.1	53.6	53.1	52.5	52.4
50 : 50	55.7	54.9	54.0	53.5	53.1	52.3	52.2
60 : 40	55.6	54.7	53.8	53.2	52.8	52.1	51.9
70 : 30	55.5	54.5	53.5	52.9	52.5	51.9	51.6
80 : 20	55.4	54.3	53.3	52.7	52.2	51.7	51.3
90 : 10	54.6	53.6	53.1	52.6	52.1	51.6	51.2
91 : 9	54.1	53.6	52.6	52.1	51.6	51.2	50.7
92 : 8	53.6	53.1	52.1	51.6	51.2	50.7	50.3
93 : 7	53.6	53.1	52.1	51.2	50.7	50.3	49.8
94 : 6	53.1	52.6	51.6	50.7	50.3	49.8	48.9
95 : 5	52.6	52.1	51.2	50.3	49.4	48.9	48.5
96 : 4	52.1	51.2	50.7	49.8	48.9	47.7	46.9
97 : 3	50.7	50.3	49.8	48.9	47.7	46.2	45.1
98 : 2	49.9	48.9	48.5	47.3	45.8	43.3	40.0
99 : 1	47.7	47.3	46.5	45.1	43.3	41.2	38.1

503. Herzfeld's Method for Materials of High Purity. (Containing less than 1.5 per cent glucose.) Prepare a solution of the material so that it contains 20 grams in 100 ml. and clarify by filtering through kieselguhr. Using 50 ml. of this filtrate conduct the test exactly as given in Sec. 501, determining the amount of copper by any one of the methods given. From the copper found obtain the corresponding percentage of invert sugar from the table (p. 792).

Although generally described as being limited to materials of over 98.5 per cent sucrose, this is the accepted method for raw sugars and other materials of high purity having a low percentage of invert sugar. For such materials as refinery soft sugars the expanded tables of Rice (p. 785) may be used for

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[15] *Zeitschrift,* **1889,** p. 735.

the calculations, after selecting the proper amount of material for analysis to bring the copper precipitate within the range of the tables.

504. Munson and Walker Unified Method. This is of wide application, the complete tables giving amounts of dextrose, invert sugar, invert sugar and sucrose, lactose, lactose and sucrose, and maltose corresponding to quantities of copper reduced when these various sugars are present. For our purposes invert sugar alone and invert sugar in the presence of sucrose are the only mixtures to be considered and the tables (p. 793) are abridged accordingly.

> If the composition of the mixture is known approximately, the weight of substance required can be judged without a previous assay. If less than 10 per cent of reducing sugar is present, a weight of 2 grams of total sugar should be taken in 50 ml. If more than 10 per cent, 0.4 gram in 50 ml. is the required amount. For unknown mixtures it is best to make an analysis with either one of the amounts of total sugar. This will serve either as a preliminary or final assay according to the amount present.[16]

The analysis is conducted as in the two Herzfeld methods in so far as preparation of the solution, precipitation of the copper, and collection and washing of the precipitate are concerned, *except that no water is added at the end of the boiling period;* also water at *60° C. is specified for washing the precipitate.* This method is of great value for thin solutions of low purity such as press waters, char filter wash waters, and the like.

The method is also valuable for high-test molasses, and a revised table published by Hammond in 1940 is given (p. 793) which includes 0.3 gram total sugars. Without this table it was necessary to add pure sucrose to the solution in order to bring the sucrose-invert relationship within the scope of the old table.

VOLUMETRIC METHODS FOR REDUCING SUGARS

505. Introductory. As opposed to the gravimetric procedure of determining the copper precipitated by a fixed amount of the sugar solution, the volumetric methods determine the volume of test solutions required to precipitate completely all the copper in a certain amount of Fehling's solution. Until the introduction in 1923 of the Eynon-Lane method described below, volumetric methods were not considered accurate. The Eynon-Lane method dispenses with the necessity of testing for the copper with ferrocyanide as in the old Soxhlet method which has now become obsolete. Eynon and Lane and many other investigators have found the newer method as accurate as the gravimetric procedures, whereas the simplicity and rapidity are evident. It has been adopted as standard procedure by many governing bodies, including the Committee on Uniformity of the International Society of Sugar Cane Technologists, the Association of Official Agricultural Chemists, and others.

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[16] *Bur. Standards Circ. 44,* pp. 84, 85.

506. Eynon-Lane Volumetric Method. (Using methylene blue as internal indicator.)[17] The sugar solution is prepared of such a strength that it will contain from 0.25 to 0.8 gram reducing sugars per 100 ml. if the sucrose is proportionately low (as in molasses) or from 0.1 to 0.3 gram if the sucrose is in greater proportion as in juices, sirups, and sugars. (See chapters relating to analysis of these products for specific instructions as to amount of sample.) Dry sodium oxalate is added to remove lime salts (see p. 410), and filtration is effected with kieselguhr; or normal lead acetate, deleading and deliming with mixed potassium oxalate and disodium phosphate as recommended by Cook and McAllep may be employed for clarification. After filtration the test is carried out as follows.

Solutions. The Fehling's solution used is Soxhlet's modification and is prepared as in Sec. 499. The indicator is prepared by dissolving 1 gram of methylene blue in distilled water and making up to 100 ml. This solution will keep for months without change.

Standard Method of Procedure. Ten or 25 ml. of mixed Fehling's solution is measured into an Erlenmeyer flask of 300 to 400 ml. capacity and treated cold with almost the whole of the sugar solution required to effect reduction of all the copper, so that if possible no more than 1 ml. is required later to complete the titration. (The approximate volume of sugar solution required is ascertained by a preliminary incremental titration which will be described farther on.) The flask containing the cold mixture is heated over a wire gauze; after the liquid has begun to boil it is kept in moderate ebullition for two minutes, and then, without removal of the flame, three to five drops of the methylene blue indicator are added, and the titration is completed in one minute further, so that the reaction liquid boils together for three minutes without interruption.

The indicator is so sensitive that the end point can be determined to within one drop of the sugar solution in most cases. The complete decolorization of the methylene blue is usually sufficiently well indicated by the whole reaction liquid, in which the cuprous oxide is constantly churned up, becoming bright red or orange in color; but if doubtful the flask may be removed from the wire gauze for a second or two and held against a sheet of white paper on the bench, when the edge of the liquid will appear bluish if the indicator is not completely decolorized. It is inadvisable to interrupt the boiling for more than a few seconds, as the indicator undergoes back-oxidation rather rapidly when the air is allowed free access to the flask, but there is no danger of this so long as there is a continuous stream of steam issuing from the mouth of the flask.

Incremental Method of Titration. The method as outlined above is the one which permits the greatest precision and is least affected by personal factors. Since, however, the volume of sugar solution required must be known approximately in order that the whole of it may be added at one time before boiling,

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[17] *Intern. Sugar J.*, **25**, 143 (1923).

a preliminary titration is usually necessary. The method of carrying out this titration is as follows: 10 or 25 ml. of Fehling's solution, in a 300- to 400-ml. Erlenmeyer flask, is treated cold with 15 ml. of the sugar solution, and without further dilution is heated to boiling over a wire gauze. After the liquid has been boiling for about fifteen seconds, it will be possible to judge if all the copper is reduced by the bright red color imparted the boiling liquid by the suspended cuprous oxide. If it is judged that nearly all the copper is reduced, a few drops of the methylene blue indicator are added, boiling is continued for one to two minutes from the commencement of ebullition, and then the sugar solution is added in small quantities, say 1 ml. or less at a time, the liquid being allowed to boil between successive additions for about ten seconds, until the color of the indicator is completely discharged. If after the mixture of Fehling's solution with 15 ml. of sugar solution has been boiling for about a quarter of a minute there still appears to be much unreduced copper, a further 10 ml. of sugar solution is added, and the whole is allowed to boil for a quarter of a minute, and so on until it is considered unsafe to add a further large increment of sugar solution; boiling is then continued for one to two minutes, after which the indicator is added and the titration is completed by small additions of the sugar solution.

Having thus determined the amount of sugar solution required to reduce all the copper by this incremental method, a second determination may be made for accurate work by the standard method described above. (The incremental method will give results within 1 per cent of the invert sugar present.)

It is not advisable to add the indicator until the neighborhood of the end point has been reached, for the same reason that in the titration of iodine with thiosulfate solution the addition of the starch indicator is best postponed until as late a stage as possible, viz., because the indicator retains its full color until the end point is reached and gives no warning to the operator to go slowly.

The flask should remain on the wire gauze over the Bunsen flame throughout the entire titration, except when it may be removed for a few seconds to ascertain if the end point is reached. In adding the sugar solution to the reaction mixture the buret should be held in the hand and brought over the flask. The buret is fitted with a small outlet tube bent twice at right angles, so the body of the buret can be kept out of the steam while the jet is held over the flask. Burets with glass stopcocks are unsuitable for this work, as the cocks become heated by the steam and are very likely to jam.

507. Standardization of Soxhlet Solution. The authors recommend that the copper solution be standardized against a pure invert solution, which may be prepared as follows. Exactly 9.5 grams of dry refined sugar (medium granulated or tablet sugar) is dissolved in 100 ml. of water and treated with 5 ml. of conc. HCl (sp. gr. 1.19). Allow to stand three days at not less than 20° C. then make to 1 liter without neutralizing. This acidified 1 per cent invert sugar solution is very stable and will keep for months.

For the standardization, carefully neutralize 50 ml. of the standard invert sugar solution in a 250-ml. flask and make to the mark with water. Then proceed to run the titration as described above with this solution, using 10 ml. of combined Soxhlet solution. The factor of the standard solution as determined by the titration is:

$$\frac{\text{Titer} \times \text{Mg. of sugar per 100 ml.}}{100} = F'$$

Reference is then made to Table 47, where correct factors are tabulated. These factors, determined by Lane and Eynon represent the weight of invert sugar required to reduce the given amount of copper completely. Small deviations may arise from variations in individual procedure or from the composition of the copper solution. The deviation from the tabulated factor may be taken care of by applying a correction to the factors in the table or by correcting the strength of the copper solution. The alkali solution need not be corrected.

Since the accuracy of the result of the volumetric methods depends on the amount of copper taken, great care must be exercised in measuring this reagent for the test. Accurately calibrated pipets or an accurate buret should be used for the copper solution.

508. Calculation of Invert Sugar by Eynon-Lane Method. Use Table 47 if 10 ml. of copper solution has been used; Table 48 for 25 ml. Under the proper heading for sucrose present in 100 ml. of the solution tested and opposite the milliliters of solution required to precipitate the copper as found by the titration (titer) is the invert sugar factor. The amount of invert sugar in 100 ml. of the test solution is the factor times 100 titer, which is calculated in the column alongside the factor.

Example. A solution containing 5 grams of sucrose per 100 ml. took 28 ml. to neutralize 10 ml. of Soxhlet solution. The factor (Table 47) is 47.7

$$47.7 \times \frac{100}{28} = 170.3$$

This is the number of milligrams of invert sugar in 100 ml. of the test solution which is the amount shown corresponding to the factor, i.e., the calculations have been carried out in computing the table. Fractions of a milliliter may be interpolated.

509. Electrometric Modification. The end point may be determined electrometrically by a method devised by Tryller[18] and in use in Queensland[19] mills. The method as described by Zerban[20] is as follows:

The apparatus, Fig. 142, consists of a round, flat-bottom, wide-mouth flask (Soxhlet extraction flask) of 250 ml. capacity with a cork stopper one-third

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[18] *Intern. Sugar J.*, September, 1932, p. 353.

[19] *Laboratory Manual,* 1939, p. 83.

[20] *Sugar Analysis,* 1941, p. 756.

of which has been cut away. The sugar solution enters from the buret through tube *T*. The electrodes are of pure copper wire about 1 mm. thick, and preferably are cut from the same coil. The cell *C* in the center is made of Pyrex tubing 8 mm. in internal diameter and 16 cm. long. At its lower end it is filled with a plug, about 8 mm. high, made from a slurry of plaster of Paris of the consistency of thick cream. After the plug has hardened the cell is filled with a mixture of 5 ml. Soxhlet solution *B* (alkaline tartrate), 5 ml. of a solution containing 39.415 grams anhydrous sodium sulfate per liter, and 20 ml. of distilled water. The cell must then be allowed to stand for several hours, preferably overnight, to saturate the plug. A number of cells may be prepared at one time, partially filled with sodium sulfate solution, and kept in a stoppered jar containing the same solution, until required.

One electrode is immersed in the cell liquid, the other in the test liquid, and the two are connected through a sensitive galvanometer with a central 0 point and a tapping key. A sensitivity of 2 microamperes per division is satisfactory.

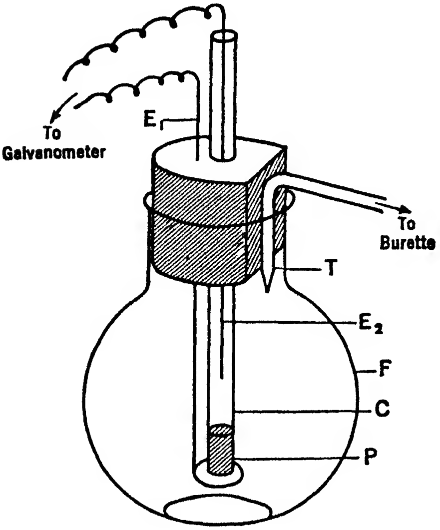

FIG. 142. Apparatus for Electrometric Modification of Eynon-Lane Method.

The determination is carried out in the usual manner with 10 ml. of mixed Fehling's solution, up to the point where the methylene blue would be added. The tapping key is pressed down, and the needle will be found to swing to one side. As the sugar solution is added drop by drop, the swings of the needle become smaller and smaller, and when the end point is reached the needle swings to the other side. Toward the end it is well to wait about 5 seconds between additions of sugar solution, because there is a slight lag in obtaining equilibrium. Duplicated tests usually agree within about 0.05 ml., the best results being obtained when the titration values are between 15 and 30 ml. If the titration values lie between 30 and 50 ml., the solution in the reference cell should be made up with 40 instead of 20 ml. of distilled water in order that the concentration will approximate more closely that of the liquid in the flask. After each titration the exposed end of the plaster plug should be cleaned with a stiff brush to remove any deposit which may have formed; the tube is then rinsed with distilled water. The copper electrodes should also be kept clean by polishing them occasionally with fine emergy paper.

The cell liquid should be renewed after every four determinations, and the plugs replaced by new ones after about forty determinations.

510. Small Amounts of Invert Sugar in the Presence of Sucrose. The Soxhlet-Fehling solution used in all the methods so far described has the objection that sucrose is rendered copper reducing because of the large amount of free alkali present. Other solutions have been employed substituting carbonate or other salts for the caustic alkali, generally for the determination of small quantities of invert sugar in the presence of large amounts of sucrose, e.g., in refined sugars, high-test raws, and beet sugars. One of these methods is here described.

Luff-Schoorl Method for Invert in Cane Sugars.[21, 22] The copper reagent employed is Luff's solution prepared as follows: 17.3 grams of $CuSO_4 \cdot 5H_2O$ and 115 grams of citric acid crystals are dissolved in about 200 ml. of water by gentle heating, and to this solution is added, while shaking, a solution of 185.3 grams of anhydrous Na_2CO_3 in about 500 ml. of water. It is important that the second solution should be added to the first, and not the first to the second. After cooling, the mixture is made up to 1 liter, shaken with 2 grams of washed and ignited kieselguhr, and filtered under suction. The alkalinity of the solution should be 1.78 N (phenolphthalein indicator) and should be controlled.

For all raw cane sugars the *Java Handbook* prescribes defecation with neutral lead acetate and removal of excess of lead by means of phosphate-oxalate mixture. According to Kekker, Klokkers, and Goslings this is unnecessary except for low-grade products such as molasses sugars. [See p. 409 regarding preparation of solutions.]

The quantity of sample used for a determination is 5 grams, or such smaller quantity as contains not more than 45 mg. of invert sugar; 25 ml. of sugar solution containing the requisite quantity of the sample is mixed with 25 ml. of the copper reagent in a 300-ml. Erlenmeyer flask, and, after addition of a few fragments of pumice, the flask is fitted with a vertical condenser and placed on a wire gauze, resting on an asbestos card and having a central hole

Milligrams of Invert Sugar in Reaction Mixtures

0.1 N Thiosulfate	No Sucrose	1.25 Grams Sucrose	2.5 Grams Sucrose	5 Grams Sucrose
0.0	0.00	.....	.....	.....
0.4	1.40	1.00	0.60	0.15
0.5	1.75	1.30	0.09	0.45
1.0	3.50	2.90	2.50	2.00
2.0	6.55	5.90	5.50	5.00
4.0	12.55	11.90	11.50	11.00
6.0	18.60	18.00	17.50	17.00
8.0	24.70	24.10	23.55	23.00
10.0	30.85	30.25	29.75	29.05
12.0	37.10	36.55	36.15	35.30
14.0	43.50	42.95	42.50	41.70
16.0	50.20	49.65	49.15	48.30

[21] *Intern. Comm. Uniform Methods Sugar Analysis*, London, 1936.

[22] *Intern. Sugar J.*, Supplement, January, 1937.

6.5 cm. in diameter. The liquid is heated to boiling in three minutes, and boiling is continued for exactly five minutes, after which the flask is at once cooled, without shaking, in tap water. The cooled liquid is treated with 15 ml. of 20 per cent iodate-free KI solution, and then with 15 ml. of 25 per cent H_2SO_4, added slowly to avoid loss by effervescence. With 1 ml. of 1 per cent starch solution used as indicator, the liberated iodine is titrated with 0.1 *N* thiosulfate solution. A blank determination is made, using 25 ml. of water in place of sugar solution. The difference between the volumes of 0.1 *N* thiosulfate required by the blank and the actual test is referred to a table (of which a skeleton is given above) to ascertain the corresponding quantity of invert sugar.

511. Kraisy's Method for Determining Invert Sugar in Refined Sugars. Zerban has used this method in the New York Sugar Trade Laboratory with good results. He describes it as follows: [23]

> Kraisy employed for this purpose the following copper reagent: solution I, containing 7.86 g. crystallized copper sulfate (= 2 g. copper) in 1 liter; solution II, containing 3.292 g. anhydrous sodium carbonate and 20 g. Rochelle salt in 1 liter. Twenty-five milliliters each of these solutions is transferred to a 250-ml. Erlenmeyer flask. In another similar flask a solution of 10 g. of the refined sugar in a total volume of 50 ml. is mixed with 5 ml. *N*/10 potassium bicarbonate solution (10.01 g. per liter). Both solutions are heated to boiling, each over a large burner, on a wire gauze covered with a piece of asbestos board having a hole about 6 cm. in diameter, the flames being so regulated that the sugar solution begins to boil when the copper solution is already boiling. It should take 2¼ to 2¾ minutes to bring the sugar solution to boiling. At this moment the boiling copper solution is poured into the sugar solution, and the boiling is continued for exactly 10 minutes, with a small flame. Then 50 ml. of freshly boiled and cooled water is added carefully, without drawing air bubbles into the solution, and without agitating the flask, which is then cooled for 5 minutes longer in cold water. The solution is next acidified with 1 to 1.25 ml. of 4 *N* hydrochloric acid (1 volume concentrated acid plus 2 volumes water), and immediately an excess of *N*/63.57 iodine solution (1 ml. = 1 mg. copper) is added. The cuprous oxide must dissolve completely, and an excess of about 5 ml. of iodine solution should be used. This is then titrated back with *N*/63.57 thiosulfate solution, using starch as indicator. The addition of thiosulfate is continued until the blue color of the iodine-starch does not return for at least 10 minutes. The difference between the milliliters of iodine added and the milliliters of thiosulfate used in the back titration is equal to the number of milligrams of copper reduced. A blank titration is run with 50 ml. of water instead of sugar solution, and the result applied as a correction to that of the actual test. Each milligram of copper corresponds to 0.435 mg. invert sugar, or 0.00435 per cent when 10 g. of sugar is used for the analysis. This proportionality holds up to 0.05 per cent invert sugar, which is well beyond the limit for refined sugars.

Kraisy recommends a correction for the reducing effect of sucrose, but Zerban finds that this may be omitted and that the results check those by Main's pot method.

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[23] Browne-Zerban, *Sugar Analysis*, 1941, p. 842.

512. DeWhalley's Method for Refined Sugars. The method consists[24] in finely grinding the sample of cubes, granulated or crystals, dissolving in cold water in a glass test tube, adding methylene blue and caustic soda solutions, heating in a boiling water bath for a very short time, and comparing the colors of the resulting solutions with standard test solutions.

The test is made in test tubes of white glass 6 in. by ¾ in. of uniform weight about 9.4 to 9.6 grams, around the top of which large rubber rings are fitted, so that they can be supported in the water bath. This bath is of sheet copper, 7-in. cube, with three holes of 1-in. diameter; the central hole is used for the test and the other two as steam vents. A constant water level is arranged 2 in. below the top of the bath. A manometer on the gas supply insures pressure of 3.5 to 3.75 in. of water, and constant pressure may be maintained by use of a gas-pressure regulator.

Then 7 grams of the ground sample are weighed out correctly to within 0.05 gram, poured into a clean, drained test tube, and to it is added 6 ml. of distilled water; 1 ml. 0.20 per cent methylene blue solution, measured by a microburet; and 1 ml. of 3 *N* caustic soda solution, also measured by a microburet.

The mixture in the tube is stoppered and shaken vigorously for fifteen seconds, the cork is removed, and the tube is immersed in the boiling-water bath for a hundred and twenty seconds. It is then removed and compared with the row of standard tubes. About five seconds or less are required to match it.

A set of standard tubes are made from a solution of 19½ grams of crystallized copper sulfate in boiled distilled water made up to a volume of 500 ml.

The following volumes were used to which was added ammonium hydroxide (0.880; 32.9 per cent NH_4OH by titration).

INVERT STANDARD PER CENT	COPPER SOLUTION ML.	AMMONIA ML.
0.001	40.00	10
0.002	24.60	10
0.003	16.40	10
0.004	10.66	10
0.005	7.18	10
0.006	4.92	10
0.007	2.97	10
0.008	2.26	10
0.009	1.74	10
0.010	1.33	10
0.015	0.50	10

Each mixture was made up to 50 ml. with boiled distilled water and sealed in glass test tubes. If the invert per cent is above 0.015, invert-free sucrose plus a sufficient amount of the sample to make 7 grams can be used for the

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[24] *Intern. Sugar J.*, August, 1937, p. 300.

test and the per cent of invert present can be calculated. This method is simple and rapid for routine work and gives close checks with more elaborate procedures.

DETERMINATION OF SUCROSE BY REDUCTION METHODS

The determination of sucrose by reduction methods is employed as a rule only where the amount of sample at hand is small or where a polariscope is not available for use. It may also be used to determine sucrose in high-test molasses and inverted sirups (see p. 536). It should be understood that the method is subject to the same errors that the double polarization methods are if the acid inversion is employed (see Sec. 483). Great care should be taken when neutralizing the acid solution with caustic soda to avoid an excess and also to avoid local excess of caustic, otherwise some destruction of levulose will result. The method is as follows.

First determine the reducing sugars as invert sugar by the method of Munson and Walker (Sec. 504). Invert a second portion of the solution as follows. Pipet a 50-ml. portion into a 100-ml. flask and add 20 ml. of water, insert thermometer, and heat to exactly 65° by immersion in a hot-water bath. Remove the flask from the bath, add 10 ml. of hydrochloric acid solution (24.85° Brix at 20° C.), mix and allow to stand for thirty minutes, then cool to room temperature in cold water, remove and rinse thermometer, and transfer the solution to a liter flask. (Inversion at room temperature about 28° C. for twenty-four hours is preferable if time permits.) Almost neutralize the acid with dilute caustic soda and complete the volume to 1000 ml. (great care must be used to avoid an excess and to avoid local excess). The solution should be so prepared that not more than 240 mg. of invert sugar is present in 50 ml. of the inverted solution. After the inversion is complete, again determine the reducing sugars by the Munson and Walker method, and deduct the amount obtained before inversion from that obtained after inversion. This represents the quantity of invert sugar obtained by the inversion of the sucrose, and since sucrose yields invert sugar on inversion at the rate of 95:100, this must be multiplied by 0.95 to calculate the sucrose originally present.

$$(\text{Per cent invert sugar after inversion} - \text{Invert sugar in the material}) \times 0.95 = \text{Percentage of sucrose}$$

CHAPTER 21

DENSITY AND TOTAL SOLIDS DETERMINATIONS

513. General Remarks. The term density is very commonly used in the sugar industry synonymously with specific gravity. There are three densimetric instruments in use in the sugar laboratory: (1) the hydrometer, (2) the Westphal balance, and (3) the pycnometer. Of these the hydrometer is by far the most common. The two hydrometer scales in use in sugar work are those of Brix and Baumé. Sugar chemists also frequently term the degree Brix or the degree Baumé the "density" of the solution. This use of the word density is not strictly correct, but it is sanctioned by usage, and the word is used in this sense in this book for brevity and convenience. The graduations on hydrometers used in sugar work are termed degrees.

514. Degree Brix or Balling. This system of hydrometer graduation was devised by Balling; the data were afterwards recalculated and checked by Brix. This hydrometer is known by both names in Germany, but the name Brix is used almost exclusively in America. The degree Brix is the percentage by weight of sucrose in a pure sugar solution. In commercial sugar analysis it is customary to consider the degree Brix as the percentage of solid matter, or the total solids, dissolved in a liquid although this is true only of solutions of pure sugar. It is this feature of the Brix hydrometer, or spindle, as these instruments are commonly called, which renders it more convenient than the Baumé instrument in sugar house work.

The degree Brix, as determined by floating a spindle in sugar solution, is generally referred to as the Brix. The percentage of total solid matter in a sugar solution as determined by drying in an oven is called solids by drying, dry matter, or true solids. The two terms should always be differentiated. Modern Brix spindles are generally calibrated to read 0° in distilled water at 20° C., although some tropical countries use a temperature of 27½° C. as a standard.

The Brix of an impure sugar solution is invariably higher than the solids obtained by drying because the non-sugars generally present are of higher density than the sugars. For example, a 20 per cent sucrose solution (20° Brix) has a specific gravity of about 1.08; a 20 per cent sodium chloride solution has about 1.15 sp. gr. (35° Brix) and a 20 per cent calcium chloride solution 1.17 sp. gr. (39° Brix). Most of the organic non-sugars are also of higher density than the sugars. A further complication in the relationship between solids by hydrometer and by drying is that caused by contraction on dilution. The volume of a solution made up of a given quantity of sucrose and a given quantity of water is always less than the sum of the volumes of the water and sucrose. The relationships between Brix and solids by drying in molasses and other low-test products is further discussed in Sec. 653.

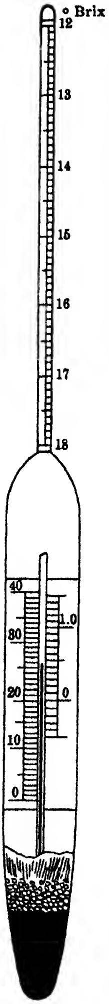

Fig. 143. Brix Hydrometer.

515. Degree Baumé. The Baumé (also spelled Beaumé) scale has no convenient relation with the percentage composition of any sugar house product. The point to which the Baumé hydrometer sinks in distilled water at the standard temperature is marked zero; the 15° was originally placed by Baumé at that point to which the instrument sank in a 15 per cent salt solution. Baumé spindles are also graduated for densities below zero, but the range of from 0° to 50° is all that is required in the sugar industry.

Gerlach and later Mategezek and Scheibler recalculated the values of the graduations of the Baumé scale. The recalculated numbers are termed the "new" or "corrected" degrees Baumé, but the terms new and corrected are misleading. The most recent Baumé scale is that calculated by the Bureau of Standards[1] based on a constant multiplier or modulus of 145 according to the formula,

$$\text{Degrees Baumé} = 145 - \frac{145}{\text{Specific gravity}}$$

all the specific gravities being at 20° C. referred to water at 20° C. (or as commonly expressed sp. gr. 20°/20° C.). The Baumé degrees according to this scale are shown in the table on p. 718. The so-called new or corrected, i.e., Gerlach, scale uses a modulus of 146.78 for specific gravities 17.5°/17.5° C.

The Baumé scale was at one time used almost exclusively in the sugar industry, but its use in the laboratory has long been discontinued in favor of the Brix scale. Planters and sugar-makers still use the Baumé scale to some extent but not under conditions where great accuracy is demanded. The use of the Bureau of Standards Baumé scale in the molasses trade is quite general. The differences between the old, the new, and the Bureau of Standards Baumé scales are shown in a table in *Bur. Standards Circ. C 440*, p. 625. The old scale is closer to the Bureau of Standards scale than the new, e.g., at 50° Brix the corresponding Baumé values are new 27.7; old 27.2; Bureau of Standards 27.28.

516. Hydrometers or Spindles. These instruments are frequently termed saccharometers when specially graduated for use in the sugar industry. A high-grade Brix hydrometer is shown in Fig. 143. This instrument is pro-

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[1] *Bur. Standards Circ. 44*, p. 159.

vided with a thermometer. Instruments for ordinary work in the factory are made of metal or of glass, and without the thermometer. The standard adopted by the International Congress of Applied Chemistry is 20° C., and this has very generally displaced the older standard of 17.5° C. For temperatures varying from these standards, corrections must be applied to the readings.

Hydrometers whose normal or standard temperature is 17½° C., when floated in distilled water at this temperature, read 0°, and the corresponding specific gravity of the water is 1.0000. In other words, the weight of a volume of the sugar solution at 17½° C. is referred to the weight of the same volume of water at 17½° C.

Hydrometers whose normal or standard temperature is 20° C., as specified by the International Congress of Applied Chemistry, when floated in water at this temperature, read 0°, and the corresponding specific gravity is 0.998234 at 20°/4°. The specific gravities of the solutions, corresponding with the degrees Brix, are given in the table, p. 718.

517. Method of Using the Hydrometer. The hydrometer is floated in the sugar solution after this has been thoroughly mixed and allowed to stand until all air bubbles have risen to the surface. The instrument should be clean and dry and should be lowered carefully into the liquid so that the stem is wet for a short distance (2 or 3 mm.) above the point where it comes to rest. Suction may conveniently be used to assist in the removal of air. A glass tube connected with a vacuum line is thrust through a large rubber stopper, which is pressed down on the top of the cylinder for a few moments. The reading on the scale is made at the point *R*, not *R′*, of Fig. 144. The point *R* is at the level of the surface of the liquid and is selected for the reading, since the meniscus varies with the viscosity of the solution. It is often necessary in dark solutions to estimate the position of the point *R*. The reading of the scale is not made until sufficient time has elapsed for the hydrometer to become the same temperature as the solution. If the temperature of the liquid varies from the normal temperature for which the hydrometer is graduated, the observed reading of the scale must be corrected. Modern instruments are graduated at 20° C., in conformity with the specification of the International Commission for Uniform Methods, and the table of corrections on pp. 728 and 729 should be used. It is necessary in accurate work to use the hydrometer at temperatures near its normal temperature. Hawaii and some other tropical countries use 27½° C. as the standard and tables for such use are given on p. 746.

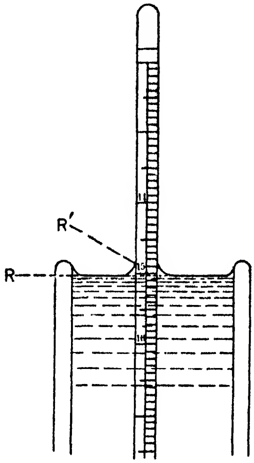

Fig. 144. Reading the Hydrometer.

518. Types of Hydrometers. The type of Brix hydrometer shown in Fig. 143 has the thermometer included, and the corrections are printed on the paper with the thermometric degrees, the height of the mercury column indicating the correction to be applied.

This type of hydrometer has many points to recommend it. The temperature recorded is the same as that at which the Brix is taken, the use of reference tables is avoided, and the standardization of the instrument includes both the hydrometer and the thermometer. On the other hand, such instruments are expensive and much more difficult to obtain of such accuracy that no correction need be applied. For these latter reasons some laboratories prefer a spindle of the same form but containing no thermometer, the temperature being taken with a floating thermometer immediately after the hydrometer is removed from the solution.

The stems of the hydrometers should be of small diameter in order that the graduation may be open and easily read. For accurate determinations the range of the instrument should not exceed 6° Brix marked in tenths, and the stem length should be at least 5 in., such graduations permitting the reading of the Brix to 0.05° by dividing the smallest graduation with the eye. All hydrometers should be tested before being put into use against solutions of pure sucrose. For general laboratory purposes a high-grade granulated sugar may be used, and the percentage of sugar may be determined with the polariscope. Hydrometers should be marked by the makers with a serial number for identification and to record the corrections found by the standardization.

Hydrometer jars or cylinders should be wide so that the spindle may float freely. A cylinder 2 in. in diameter, 15 in. high, either with or without a lip, is satisfactory.

519. The Westphal Balance.[2] The principle of this balance, Fig. 145, may be briefly stated as follows. A glass bob is so adjusted as to be capable of displacing a given number of grams, five for instance, of distilled water at a given temperature when wholly immersed in the liquid and suspended by a fine platinum wire. The bobs may be graduated for any temperature; but for sugar work 17½° C. or 20° C. (preferably the latter) is most convenient, since these are the temperatures usually employed in preparing specific gravity tables. For accurate work the temperature of the solution whose specific gravity is to be determined should be exactly that for which the bob was graduated. The balance is provided with several riders or weights. Two of these riders, 1 and 2, are each exactly the weight of the water displaced by the bob at the standard temperature, 20° C. The other riders, 3, 4, and 5, are respectively one-tenth, one-hundredth, and one-thousandth the weight of the first mentioned. When the weight 1 is hung on the hook at the end of the beam and the bob is immersed in distilled water at 20° C., the balance should be in equilibrium, the weight having the value 1.000 in this position. If the balance is not in equilibrium under these conditions, provided the bob

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[2] Adapted from *U.S.D.A. Bull. 13, Chem. Div.*, also illustration.

has been correctly graduated, suspend the bob from the hook and turn the adjusting screw until the pointers are exactly opposite one another. The weights 2, 3, 4, and 5 are placed on the beam in addition to 1 for liquids heavier than water, and have the values 0.1, 0.01, 0.001, and 0.0001, respectively, when placed on the corresponding graduations of the beam, and for other graduations 0.300, 0.030, 0.003, 0.0003, etc. Each rider is provided with a hook from which additional weights may be suspended if more than one fall upon the same graduation.

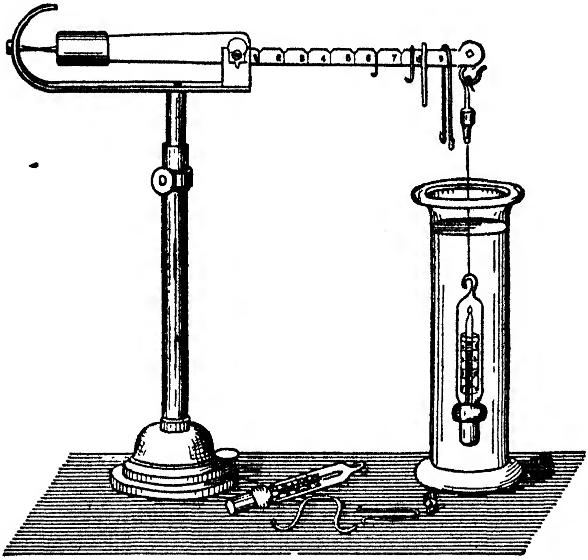

FIG. 145. Westphal Balance.

The method of using the balance is as follows. Suspend the bob of the balance as described above in the solution, at the standard temperature, and weight the beam with the riders until the balance is in equilibrium. Read off the specific gravity from the position of the weights on the beam.

Example. In determining the specific gravity of a sample of cane juice the position of the riders was as follows:

1 at point of suspension of the bob = 1.000
2 not on the beam
3 at 7 = 0.07
4 at 9 = 0.009

Specific gravity = 1.079

The degree Brix or Baumé corresponding to the specific gravity may be ascertained from the tables on p. 718.

520. Pycnometers. Pycnometers are bottles so constructed that they may be filled with a definite volume of a liquid. Given the weight of this volume, it may be compared with the weight of an equal volume of distilled water. It is not often necessary to use a pycnometer in technical work, the more rapid density determinations by the hydrometer being usually sufficiently accurate.

Pycnometers are made in a great variety of forms. One of the most convenient of these is shown in Fig. 146. The side tube provides an outlet for the excess of liquid when the stopper, a fine thermometer, is put in place, also for the overflow, as the temperature of the liquid rises. The bottle should be filled with the liquid cooled to a temperature lower than that at which the density is to be determined. As the temperature rises to the desired point, the liquid expands, and the excess is blotted with paper at the side tube. At the required temperature the cap is placed in position and receives any further liquid that may be expelled, as the temperature rises to that of the workroom. In the top of the cap is a minute opening for the escape of the air.

The pycnometer may readily be used for the older standard temperature of 17½° C. since both weighings can be made at the standard temperature. The specific gravity is calculated by dividing the weight of the solution at 17½° C. by the weight of water at the same temperature and recorded as follows:

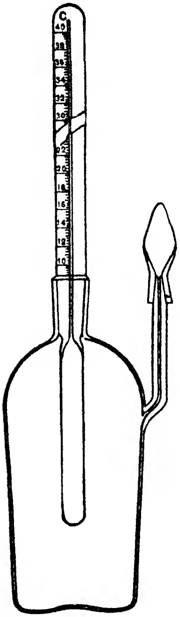

FIG. 146. Pycnometer.

$$\text{Specific gravity } \frac{17.5^\circ \text{ C.}}{17.5^\circ \text{ C.}} = 1.07956$$

The International Commission for Uniform Methods of Sugar Analysis has adopted the standard temperature of 20° referred to water at 4° C., and in determining the specific gravity at 20°/4° C. calculations may be made by the following formula: [8]

$$S = \frac{0.99703M}{W} + 0.0012$$

where S = specific gravity at 20°/4°;
M = weight of the solution at 20° C.;
W = weight of water at the same temperature.

The simpler procedure is to determine the specific gravity at 20°/20° C. and then find the percentage of sucrose (or Brix or Baumé) corresponding to the specific gravity so found in Table 27, p. 718.

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[8] Browne-Zerban, *Sugar Analysis*, 1941, p. 61.

TOTAL SOLIDS BY DRYING

521. General Observations. The determination of the total solids and moisture in sugar house materials is one of the most unsatisfactory tests in routine control work. This is due to the ready decomposition of several of the constituents and under some conditions to their tendency to occlude moisture. It is advisable to select methods that give fairly comparable results under average conditions and to use these at all times. If the drying is conducted in an air oven, the same weight of material, the same size and kind of a dish and the same temperature and heating period should be adhered to at all times for a given class of materials. A temperature that is suitable, e.g., to a high-purity large-crystal sugar, is too high for a soft sugar of low polarization. The first requires a comparatively high temperature (105° C.) to drive off the occluded water, and the other a very low temperature (60° C.) to avoid decomposing the invert sugar.

Great care must be exercised in cooling and weighing the sample after drying as all dried sugar products are very hygroscopic. It is best to employ dishes with tight-fitting covers, to use a separate desiccator with each sample, to make sure that desiccating material is active, and finally to weigh as rapidly as possible. Many control methods prescribe drying for a fixed time at a definite temperature, rather than to constant weight, since continued heating may cause a destruction of organic matter. Solids, such as sugars, are dried in the natural condition, whereas molasses, sirups, and massecuites are generally accurately diluted 1:1 with water. This offers no extra manipulation since the double dilution of these products for Brix determinations is routine procedure. Juices and other thin solutions are weighed without dilution. The liquid substances are generally spread on some absorptive material (pumice, kieselguhr, quartz sand, etc.) so as to present as large a surface as possible to the drying medium.

522. Moisture Determinations. *Sugar.* Weigh 5 grams of the well-mixed sample into a flat dish (aluminum, 60 mm. in diameter with cover is convenient). Spread the sugar evenly over the bottom and dry at 105° C. for three hours. Cool in a desiccator and weigh. The loss in weight, divided by the weight of sugar taken, multiplied by 100 is recorded as percentage moisture.

The Association of Official Agricultural Chemists tentative method calls for drying ten hours at the temperature of boiling water; cooling and weighing; then reheating for one-hour periods until loss of weight is not more than 2 mg. For large-grain sugars, heating for the last hour at 105°–110° is recommended. The shorter heating period given above is general practice in sugar laboratories.

Drying in Vacuum. The present official method of the Association of Official Agricultural Chemists for sugars[4] calls for drying in a vacuum at

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[4] *Methods of Analysis,* fifth edition, 1940, p. 484.

a temperature not exceeding 70° (preferably 60°) under a pressure not exceeding 50 mm. of Hg, for two hours; redrying and reweighing for one-hour periods until the change in weight in successive one-hour intervals is not more than 2 mg. The oven should be bled with a current of dry air during the drying to insure removal of water vapors.

This is substantially the same as the recommendation of the International Commission for Uniform Methods (London, 1936) except that the Commission specifies 10 grams instead of 5.

Liquid Products. Drying on Pumice Stone. Carr and Sanborn's Method.[5] Prepare pumice stone in two sizes. One size should pass a 1-mm. sieve and the other should pass a 6-mm. sieve, circular perforations. Digest each with dilute sulfuric acid (1 : 9) for eight hours on a steam bath then wash free from acid and heat to dull redness. Place a layer of the finer pumice stone 3 mm. thick on the bottom of a 60-mm. aluminum dish with a cover, then a layer of the coarse pumice stone 6 to 10 mm. thick upon the first layer. Add such a quantity of the solution to the tared dish and pumice stone as will yield approximately 1 gram of dry matter. In weighing the solution use a weighing bottle provided with a cork through which a pipet passes. Dry to constant weight in a water oven if the material contains little or no levulose, making trial weighings at intervals of two hours. The drying may be conducted in a vacuum oven at about 70° C. if the material contains much levulose or other readily oxidizable matter.

The weight of dry matter divided by the weight of solution used and the quotient multiplied by 100 equals per cent of total solids.

Drying on Quartz Sand.[6] Place 25–30 grams of pure quartz sand that will pass a 40-mesh but not a 60-mesh sieve and a short stirring rod in a 60-mm. aluminum dish, dry thoroughly, cool in desiccator, and weigh. Then add sufficient of the diluted sample to yield approximately 1 gram of dry matter and mix thoroughly with the sand. Heat on a steam bath for fifteen to twenty minutes, stirring at intervals of two to three minutes. Dry at 70° C. in a vacuum oven, making trial weighings at two-hour intervals toward the end of the drying period until the change in weight does not exceed 2 mg. As in the method above the drying may be at atmospheric pressure in a water oven if the material contains little levulose or other decomposable substance.

Dry sand and the dried sample absorb appreciable quantities of moisture on standing over most desiccating agents, so all weighings should be made immediately after cooling in the desiccator.

In many sugar laboratories it is customary to determine moisture in molasses and other low-grade products at 100° C. without vacuum, in spite of the proportion of levulose present, using methods similar to the official methods given above. The heating time is generally fixed at five hours, no attempt being made to heat to constant weight. It is doubtful whether

[5] *U.S.D.A. Bull. 46, Div. Chem.*, p. 45.

[6] Adapted from Official Methods A.O.A.C.

such methods give reproducible results, and their value, even in routine work, is questionable.

523. A Convenient Vacuum Oven. Most laboratories are equipped with the ordinary electric drying ovens with thermostatic control, but electric vacuum ovens are more costly. A steam-heated vacuum oven that can be made in the factory machine shop is shown in Fig. 147. Two pieces of large pipe, e.g., 8-in. and 12-in., about 20 in. long make a convenient size. These should be welded or threaded to a collar at each end to form the steam jacket. One end is completely closed and the other has an airtight removable door as shown.

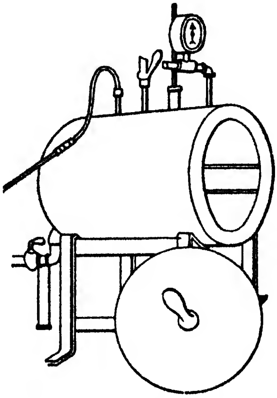

Fig. 147. Vacuum Oven.

A pressure of not more than 50 mm. of Hg (= vacuum of 28 in.) is prescribed by the Association of Official Agricultural Chemists and the International Commission for drying in vacuum. A current of dried air is bled into the vacuum oven to facilitate the removal of water vapors. Such a high vacuum cannot be attained in a factory vacuum system, and a special laboratory vacuum pump must be installed if the official method is to be followed strictly. For control purposes, connection with the factory system (generally the last body of the multiple effects) will give sufficiently low pressures for concordant results.

524. Spencer Electric Oven for Moisture Determinations. The Spencer patent electric oven was first described in 1921.[7] It is made in two sizes and is a device for passing a large volume of air over a heating element and then through a capsule containing the material to be dried. Type *A*,

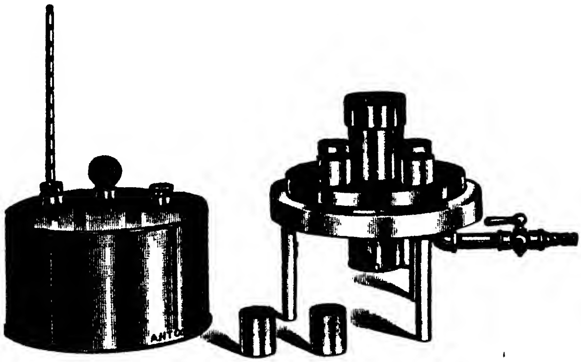

Fig. 148. Spencer Drying Oven.

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[7] Spencer, *J. Ind. Eng. Chem.*, **13**, 70 (1921).

used for sugars and sugar-containing liquids, is shown with the cover removed in Fig. 148 and also in section in Fig. 149. The larger size *B* will be described in the chapter on bagasse analysis. Both these ovens are admirably suited to routine work and are recommended by the Committee on Uniformity of the International Society of Sugar Cane Technologists and other governing bodies. The aluminum capsules are 3 cm. in diameter by 4 cm. in depth and are fitted with a bottom of metal filter cloth to permit the air to pass freely. A study of the cross-section drawing will show the path of the air over the heating element and through the capsule. The heating element is connected in series with a sliding contact tube rheostat to regulate the temperature and also with a time switch to cut off the current at the end of the drying period. The current must never be turned on before the suction is in operation, and suction must not be stopped while the current is on, otherwise the heating element will burn out. To prevent this, Spencer devised a vacuum switch (Fig. 150) which cuts off the current when the suction is stopped.

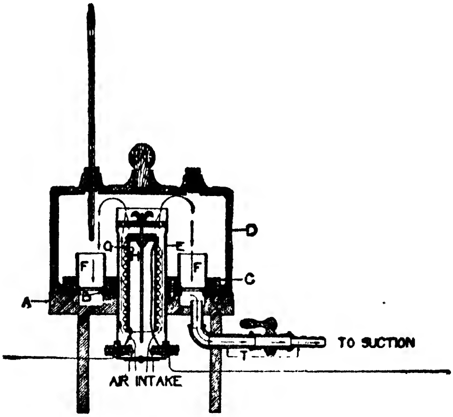

Fig. 149. Cross Section of Spencer Oven.

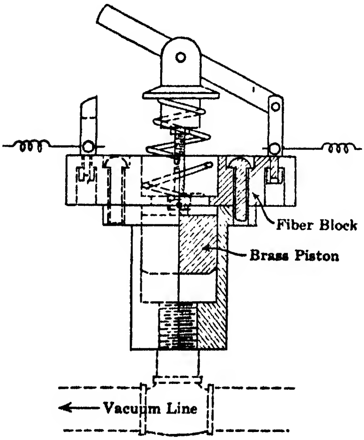

Fig. 150. Vacuum Switch for Spencer Oven.

Moisture in Sugars. Four samples may be analyzed at the same time if desired but if less than four tests are to be made the capsule openings not in use are blanked off with close-fitting corks. The capsules should be cleaned and dried and kept in a desiccator before use. To make a test, weigh a dried capsule and then weigh into it as rapidly as possible 5 grams of the well-mixed sugar sample. Place the loaded capsule in its opening in the oven, making sure that the seat is clean to avoid leakage of air around the capsule. Blank the unused openings and place the cover on the oven; open the suction (preferably a connection with a factory vacuum line), making sure that the cover fits tightly with no air leakage. The current is turned on, and all resistance is cut out so as to raise the temperature as quickly as possible to 110° C. As it approaches this heat, slide the contact to a predetermined point and regulate the temperature by small adjustments of the rheostat. With practice, there is little difficulty in keeping the air current adjusted to within one or two degrees of 110°. The oven may be fitted with a thermostat if desired. Twenty minutes is the prescribed heating period, although the greater portion of the moisture is driven off in the first five minutes. After drying, cool in a desiccator and weigh.

$$\text{Per cent moisture} = \frac{\text{Loss in weight}}{5} \times 100 = \text{Loss in weight} \times 20$$

Moisture in Molasses, Sirups, Juices, etc. The Spencer oven may be used for the determination of moisture in liquid products by absorption of the material on asbestos.[8] Several precautions are necessary to insure concordant results. The asbestos should be freshly ignited and fluffy; the material should be diluted 1 : 1 (except juices or other thin solutions) so that it will be readily absorbed by the asbestos; not more than 4 ml. of liquid should be used, only one test should be made at a time, and the oven should be brought approximately to temperature (110°) before the loaded capsule is placed in it.

The procedure is as follows. The capsule is filled with fluffy freshly ignited asbestos loosely packed, and the whole is heated in the oven at 110° for a few minutes, cooled, and weighed. The capsule holds about 6 grams of asbestos. Using a weighing bottle fitted with a dropping pipet, add about 4 ml. of the solution to be tested, a drop at a time, to the asbestos, being careful to add it slowly so that the asbestos will absorb it. Drop the liquid in the middle of the asbestos, not at the wall of the capsule, and allow the capsule to stand for a few minutes to complete the absorption. Examine the bottom of the capsule, and if there is evidence of the liquid having gone through the asbestos, discard the test and begin again. The oven is then heated to temperature, the capsule is placed in position, suction again is turned on, and the air is drawn through the sample for twenty minutes, temperature being maintained at 110° by rheostat or thermostat as for sugars. One sample only should be tested at a time as it has been found that the air will not distribute itself evenly through two or more capsules, probably due to differences in the packing of the asbestos. After removing the capsule from the oven examine the bottom

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[8] Meade, *J. Ind. Eng. Chem.*, **13**, 924 (1921).

carefully, as the liquid sometimes pulls through if the absorption by the asbestos has been faulty. To permit the use of a larger volume of asbestos a special capsule with flaring sides has been made for testing liquids, but experience has shown that, with the precautions advised for adding the solution to the asbestos, the ordinary capsule will serve.

At the end of the heating period, cool and weigh the capsule as before. The calculation for materials diluted 1 : 1 is as follows.

$$\text{Weight of capsule and material after drying} - \text{Weight of prepared capsule} = \text{Solids in diluted solution}$$

$$\frac{\text{Solids in diluted solution}}{\text{Weight diluted solution taken} \times 100} = \text{Per cent solids in } 1:1 \text{ solution}$$

$$100 - (\text{Per cent solids in diluted solution} \times 2) = \text{Per cent moisture in original material}$$

Modification for Vacuum Drying. A modified Spencer oven for drying in vacuum is described on p. 545.

TOTAL SOLIDS BY REFRACTOMETER

525. General Principles. Tolman and Smith's[9] investigations showed that most sugars in solutions of equal percentage composition have the same refractive indices, also that Gerlach's table of Brix hydrometer corrections may be used with small error to correct from room temperatures. Main was the first to use the refractometer in refinery work.[10] He found that the refractive index indicates accurately the content of solid matter in all but low products. Geerligs and van West[11] were the first to use the refractometer in cane sugar factories.

526. Refractometric Tables. Tables giving the relationship between refractive indices and percentages of sucrose were worked out by the above investigators and by Schönrock and Landt.[12, 13] To avoid the confusion due to the use of various tables, the International Commission for Uniform Methods of Sugar Analysis in 1936 made up an "International Scale of Refractive Indices of Sucrose Solutions" which is given as Table 33. This consists of Landt's values to the fifth decimal place for solutions up to 24 per cent, Schönrock's table to four decimal places from 25 to 66 per cent extrapolated as far as 70 per cent, and Main's table above 70 per cent. The International Commission also adopted a temperature correction table based on Schönrock's values of variations of refractive indices with temperature which is given on p. 753. For tropical models of refractometers standardized

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[9] *J. Am. Chem. Soc.*, **28**, 1476 (1906).

[10] *Intern. Sugar J.*, **9**, 481 (1907).

[11] *Archief*, **15**, 487 (1907).

[12] *Z. Ver. deut. Zuckerind.*, **61**, 421 (1911).

[13] *Z. Ver. deut. Zuckerind.*, **83**, 692 (1933).

at 28° for which Geerlig's tables were formerly used, the Commission adopted a set of values made up from the refractive indices at 20° corrected according to Schönrock's values for temperature variations. These values at 28° C. are included as a part of Table 33 on p. 747, and the temperature corrections for 28° C. are given on p. 754.

527. Practical Applications. The refractometer is affected only by the solids in solution; therefore if the material under examination contains insoluble matter, this is not included in the estimate of the solids. Within its limitations, i.e., in materials containing only soluble solids and a small proportion of non-sugars, the refractometer is capable of giving very accurate indications as to the solids content, usually quite as accurate as those by drying.

The refractometric results with materials of low purity are usually intermediate between those by hydrometer and by actual drying. In spite of certain advantages, such as rapidity and the small amount of solution required for the test, the refractometer has not succeeded in displacing the older methods of hydrometers and solids by drying in general routine work. An error of 0.0002 in the reading is equal to 0.1° Brix, and such accuracy is not readily attained especially for low-test materials with the ordinary Abbé type of instrument. When the Amici prisms are omitted and monochromatic light employed, the precision is greater (Sec. 530). The new Bausch and Lomb precision refractometer (Sec. 533) is accurate to three units in the fifth decimal place, but the high cost of this instrument has so far prevented its general adoption for routine work.

528. Correction for Invert Sugar. DeWhalley[14] has shown that for materials containing a high percentage of invert sugar a correction should be made of 0.022 for each per cent of invert.

DeWhalley's work was done on inverted refinery sirups and gave close checks between solids by refractometer corrected for invert and true solids by drying in vacuum at 70° C. Analyses of high-test molasses also give close check results (within the limits of the methods) between refractometer solids corrected for invert and solids by drying in the Spencer oven (see Sec. 524). An example of the use of the correction in an analysis of high-test molasses is as follows:

Refractometer reading @ 26° C. = 1.4980 = 83.0 (Table 33)
Correction for temperature 0.48
83.48
Per cent invert 49.0 × 0.022 = 1.08
Refractometer solids corrected for invert = 84.56
Solids by drying = 84.25

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[14] *Intern. Sugar J.*, September, 1935, p. 353.

529. Types of Instruments. Abbé Refractometer. The instrument in most general use in sugar laboratories is the Abbé refractometer which is manufactured in substantially the same form by several makers of optical instruments. A concise exposition of the theory of refractometry and the principles of the Abbé refractometer are given in "Polarimetry, Saccharimetry and the Sugars," *Bureau of Standards Circular C 440,* 1942, p. 255.

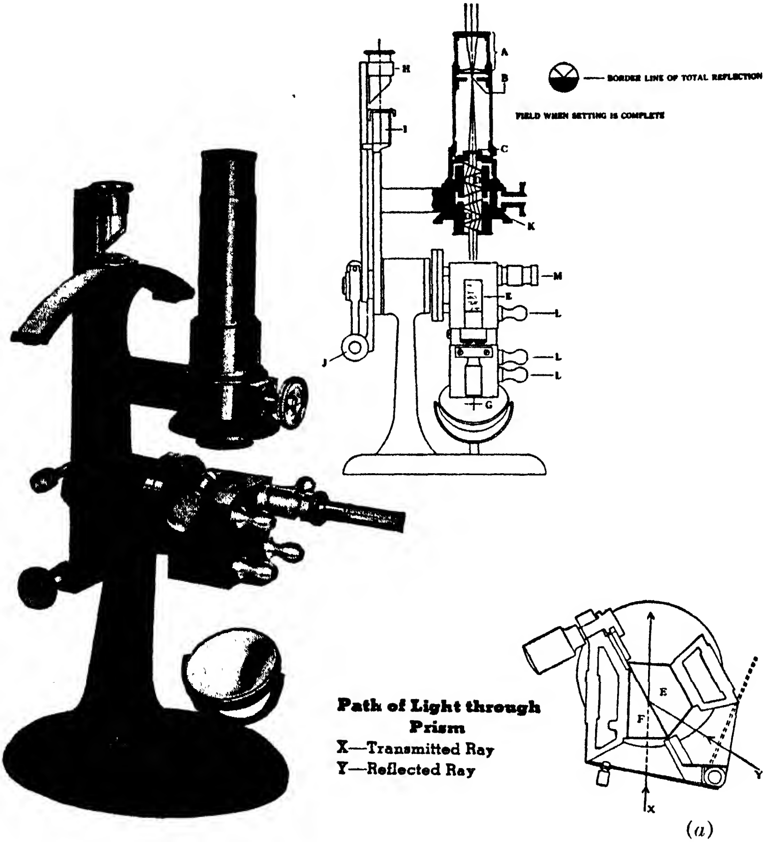

FIG. 151. Abbé Refractometer (Bausch and Lomb).

The one shown in Fig. 151 (Bausch and Lomb) consists essentially of a polished flint-glass prism, *E*, and a ground flint-glass prism, *F*, of index $n_D = 1.75$, cemented into a metal mounting, and a compensator, consisting of two Amici prisms, *DD*. The mounting of one of the flint-glass prisms is hinged so that it may be separated from the other. A drop or two of the liquid to be tested is placed upon the polished surface of the fixed prism, *E*, and the hinged prism is carefully closed against it, and the instrument is revolved to a convenient position with respect to the light. The light, preferably from an electric lamp, is reflected upon the lower prism, *F*, by the mirror, *G*. The prisms are attached to a movable arm, at the left, which carries a scale reader, *H*, for reading the index scale, *I*. To set the instrument, the movable arm is adjusted until the intersection of the reticule of the telescope cuts the line separating the dark from the light portion of the field, the final adjustment being made by means of the screw, *J*. The refractive index is then read off the scale with the scale reader, *H*. Uniform temperature conditions should be maintained by means of a flow of water from a large vessel through the tubes, *LL*. The temperature of the observation should be noted and the correction be applied, using the table of corrections on p. 754.

The percentage of dry matter corresponding to the index is given in the table on p. 747. The compensator, consisting of two small prisms, *DD*, placed in the telescopic tube, serves to eliminate the dispersion produced by the prisms. By rotating the small prisms with respect to each other by means of the milled head revolving the scale, *K*, the color is removed from the field. This compensation is necessary before making the observation. The prisms should be cleaned after use with filter paper dipped in warm water and should then be dried with soft linen. The instrument may be tested with air-free distilled water (index = 1.33298) or with standard plates. By means of a recent improvement the observations are made by reflected light when dark or opaque liquids, e.g., molasses, are to be examined (Fig. 151*a*.)

The prism box is provided with a combination shield and reflector which may be swung open so as to make light transmission possible through the fourth face of the refracting prism.

The liquid is applied to the prism face normally used, but the prism, *F*, is employed only as a retainer for the liquid specimen. Readings are taken in the same way and correction for color made in the same way as above. This feature extends the convenient application of refractometric measurements to many new fields.

530. Abbé with Monochromatic Light. The Spencer Lens Company has recently inaugurated the manufacture of the Abbé type of refractometer without Amici prisms for use with monochromatic light. This increases the sensitivity of the instrument and may result in the more extended use of refractometers for cane sugar work. The manufacturers' description follows:

> Spencer Refractometers without Amici compensating prisms are available for use with monochromatic light for those who prefer the utmost precision in an Abbé type instrument. These instruments are graduated to give index of refraction directly when used with monochromatic sodium light (589 mμ). By using special conversion tables other monochromatic light sources can be utilized in measuring refractive index. Thus, by employing conversion tables for the F (486 mμ) and C (656 mμ) lines of the hydrogen spectrum, dispersion ($n_F - n_C$) of the sample can be determined directly using a hydrogen discharge tube for the light source with suitable filters.

531. Immersion Refractometer. This is also called the dipping refractometer. The general optical construction is the same as in the Abbé, but the immersion type is designed to cover a small index range with higher accuracy than is possible with the Abbé instrument. It has never been used to any extent in cane sugar control work in this hemisphere. Instructions and special tables are supplied by the dealers.

532. The Sugar Refractometer. This instrument[15] (made by Carl Zeiss of Jena), Fig. 152, is a special form of Abbé's instrument and has been designed especially for the use in the sugar industry and the oil industry. It has the double prisms of the Abbé and differs from this in the optical nature of the glass forming the prisms, designed by Schönrock, and in having a modified comparator.

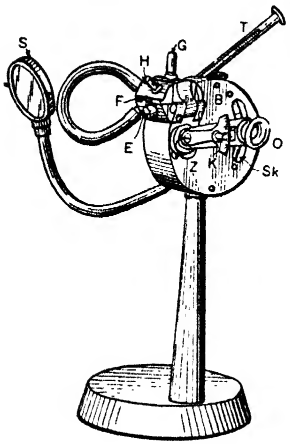

FIG. 152. Zeiss Refractometer.

The image in the eyepiece presents the following appearance. In place of cross lines, the field of view shows a circle with its center marked by a small black dot. On the right of the dot the percentage scale ranging from 0 to 50 per cent in fifths, thence to 97.5 per cent in tenths of a per cent, every integral of percentage being numbered. On the left of the dot is a scale in terms of refractive indices (ranging from 1.330 to 1.540). The fact that the reading is taken in the field of view insures a considerable saving of time.

When examining a very dark sample recourse must be had to illumination by reflected light. Full instructions are supplied by the makers for setting up and manipulating this instrument.

533. Bausch and Lomb Precision Sugar Refractometer. This instrument is especially designed for sugar work, having a range of 1.33 to 1.50. The principal difference between this refractometer and those already described is the use of a sodium vapor lamp (see Sec. 451) in place of white light. This permits the elimination of the color compensator necessary with white light. The instrument as shown in Fig. 153 is heavily mounted and so arranged as to be read easily. The optical arrangement has been described by Forest[16] and a full description and instructions are given in the reference manual supplied by the makers.

The scale is an arbitrary equi-division type fitted with a vernier which permits reading to tenths of a scale division. The accuracy corresponds to 0.00003 of the refractive index or less than 0.02 of a degree Brix.

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[15] Abstracted from Zeiss's instructions.

[16] *Proc. 6th Congr. Intern. Soc. Sugar Cane Tech.* (Baton Rouge, 1938), p. 890.

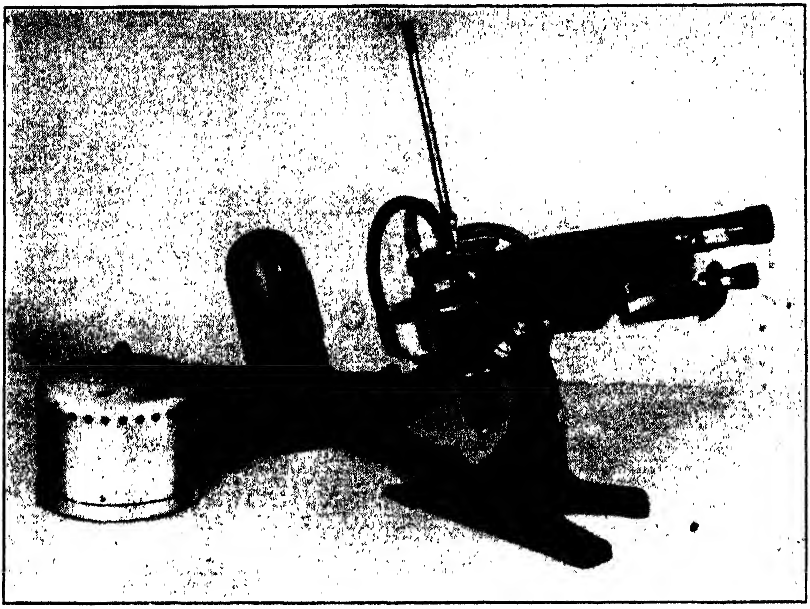

Fig. 153. Bausch and Lomb Precision Refractometer.

534. Hand Refractometer. Small hand-type refractometers such as the one shown in Fig. 154 have been developed by various optical firms. These instruments read to about one half per cent of sugar and are used in cane and beet field work to determine the maturity of the plant. Various methods of drawing juice samples from canes and beets have been devised, such as special knives and hypodermic syringes. Such equipment is generally supplied by the makers of the instruments.

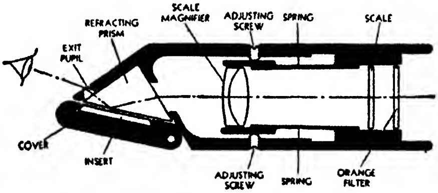

Fig. 154. Bausch and Lomb Hand Refractometer.

CHAPTER 22

DETERMINATION OF ASH

535. Definition of Ash. There are two conceptions as to what constitutes the ash in sugar products. "The residue remaining after burning off all organic matter" is the definition of the Committee on Uniformity of the International Society of Sugar Cane Technologists. This means the residue after direct incineration and would seem to be a simple definition and simple procedure. For many years it was considered the correct method—the so-called carbonate or normal ash—whereas the sulfated ash which is used for routine work was tolerated because it was easier to carry out.

A complete reversal of ideas occurred through the publication by Browne and Gamble[1] which proved that the direct incineration method is the unreliable one of the two, because of the loss of chlorine, sulfur, and other constituents to a varying and uncontrollable degree. Lundén[2] also concluded that the sulfate method gave the correct relative measure of the ash, and that the sulfated ash for different products is comparable. It is now generally recognized that neither method gives the absolute measure of the salts present, but that the sulfated ash is a relative measure of the salt content which is reproducible in check tests. For this reason all work with conductimetric ash has been with a view to checking the sulfated ash.

536. Relationship between Carbonate and Sulfated Ash Methods. The method of incineration with sulfuric acid was introduced by Scheibler in 1864, and his work with beet products showed that 10 per cent should be deducted to bring the results into conformity with the direct incineration method. This deduction has been generally continued for cane sugar products. Many investigations have shown that the difference is much greater and quite variable. Withrow and Jamison[3] found that for the Cuban raw sugar used in their experiments 33.25 per cent was the correct deduction. Spencer had a series of determinations made of the ash in final molasses from several Cuban factories and found that the sulfated ash ranged from 17 per cent to 31 per cent higher than the normal ash. On the basis of these earlier investigations the conclusion was reached that the sulfated ash was too unreliable for use, and recommendations were made to the Association of Official Agricultural Chemists and in many of the journals that its use be discontinued. As has been pointed out above the work of Browne and Gamble,

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[1] *Facts About Sugar,* December, 1923.

[2] *Z. Ver. deut. Zuckerind.,* **75**, 774 (1925).

[3] *Ind. Eng. Chem.,* **15**, No. 4 (1923).

Lundén, and others has shown that the major reason for the wide variations was the unreliability of the carbonate method.

The view that the sulfated ash is the correct relative measure of the salts has led many bodies to eliminate the correction factor. The Association of Official Agricultural Chemists, the Java Experiment Station and the Committee on Uniformity of the International Society of Sugar Cane Technologists all direct that sulfated ash shall be reported without deduction. Long practice still prompts most sugar analysts to continue the 10 per cent deduction, although the logical procedure would seem to be to eliminate the deduction. Whichever procedure is adopted should be followed consistently for all products.

It should be noted that the formulas reported later in this chapter for conductimetric ash are all based on correlation with sulfated ash less 10 per cent.

537. Choice of Method. From the preceding discussion it is evident that for all routine and control analyses the sulfated ash is the one to be used. The carbonate ash is to be used only where further analysis of the ash is to be carried out. The Association of Hawaiian Sugar Technologists[4] still continues to advocate the carbonate method for control purposes and directs that where the sulfuric acid method is employed (in the absence of a suitable muffle furnace) the results shall be calculated to carbonate ash by deducting 25 per cent for final molasses and 20 per cent for all other products.

538. Sulfated Ash Method. Weigh 2 to 5 grams of the material into a 50- to 100-ml. platinum dish (amounts of sample to be taken will be specified in chapters dealing with analysis of specific products, i.e., juice, molasses, and sugars), add 0.5 ml. of concentrated sulfuric acid, heat gently until all carbon is burned off. Cool and add a few drops more of sulfuric acid, heat until this is fully volatilized, cool, and weigh. Withrow and Jamison[5] advocate the use of sulfuric acid diluted 1:1. They found porcelain crucibles suitable for this work but objected to fused silica as giving low results. The Association of Official Agricultural Chemists specifies 10 per cent H_2SO_4 and a temperature of 550° C.

Valdez and Camps-Campins[6] in a study of the sulfated ash method advocate heating the ash for one hour at 804° C. (1480° F.) to insure complete conversion of all sulfatable metals into sulfates, except the iron and aluminum which are decomposed into the oxides. They show that large errors may enter into the determination if heating after resulfating is at too low a temperature.

Valdez and Camps-Campins[7] have shown that large samples of low-ash, e.g., refined sugar products, can be readily carbonized for the sulfate ash method as follows.

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[4] *Methods of Chemical Control,* 1931, p. 47.

[5] *Loc. cit.*

[6] *Ind. Eng. Chem., Anal. Ed.,* January, 1937, p. 35.

[7] *Loc. cit.*

The solution of the sugar product (say at 50° Brix) plus a few cubic centimeters of sulfuric acid is allowed to drop slowly from a pipet into a platinum dish heated over a small open flame so as to bring about the dropwise carbonization of the sample without any sputtering. In this way some 60 to 100 grams of the sample may be carbonized in a dish of 100-ml. capacity. The admixture of the acid with the sample facilitates the charring of the drops in the heated dish. The rate of dropping is regulated by a screw clamp which saddles a piece of rubber tubing placed over the top of the pipet.

539. Direct Incineration, Normal or Carbonate Ash. Weigh out the sample as above and heat slowly until swelling ceases. (The addition of a few drops of pure olive oil is advocated by the Association of Official Agricultural Chemists.) Then heat in a muffle at low redness (the Association specifies 525° C., Withrow 500° C.) until a white ash is obtained; moisten with a little ammonium carbonate and again heat in muffle at low red heat. (The ammonium carbonate is dispensed with in the latest Association methods.) As stated above the method is used only in investigation work and is difficult to check.

It is difficult to burn sugar or sugar house products to obtain a large quantity of the carbonate ash for complete analysis. The usual method is conducted as follows. The material is heated in a large platinum dish until it takes fire and the flames burn out. It swells greatly and is difficult to confine to the dish. (Pure olive oil or petrolatum may be added to reduce this danger.) After the material has been sufficiently charred, it is transferred to a glass mortar and is rubbed to a powder. The powder is washed upon an ashless paper filter and is thoroughly extracted with hot water. The filtrate is reserved for further treatment. The filter and insoluble matter are returned to the platinum dish and are completely ashed. The filtrate is now evaporated to dryness in the platinum dish and the dish and contents are then heated to low redness to burn off any remaining organic matter. The complete analysis of the ash so obtained is carried out according to the accepted methods of quantitative analysis.

ASH DETERMINATIONS BY ELECTRICAL CONDUCTIVITY[8]

540. Introductory. Some time after Arrhenius' fundamental investigations on the electrical conductivity of salt solutions containing non-electrolytes, the conductivity of sugar products and its relation to their ash content were studied in Europe by several investigators (about 1909–1910). Then the subject rested a number of years, and interest in it has been revived by the work of Snell[9] on maple products, by Lundén[10] and by Toedt[11] on beet

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[8] Abstracted from descriptions supplied by Drs. Zerban and Sattler.

[9] *J. Assoc. Official Agr. Chem.*, **4**, 435 (1921).

[10] *Cent. Zuckerind.*, **33**, 204 (1925); *Z. Ver. deut. Zuckerind.*, **75**, 763 (1925).

[11] *Z. Ver. deut. Zuckerind.*, **75**, 429 (1925).

products, and by Zerban and Mull,[12] and Zerban and Sattler[13] on cane products.

The advantages of electrometric ash over the chemical method lie chiefly in its speed and accuracy and simplicity.

The results as determined with high-class apparatus are comparable in accuracy with sulfated ash determinations made under most exacting research conditions, and may be made in about one-tenth the time. They are much more accurate than routine ash determinations. Conductivity measurements are particularly useful in refinery investigations of ash adsorption by boneblack.

The analysis of sugar products for ash by the electrometric method is based on the fact that distilled water is an exceedingly poor conductor of electricity, as is a highly purified solution of sucrose. Soluble mineral impurities, as well as organic salts, which are found in raw sugar, break up in water solution into electrically charged particles called ions, and as the ease with which electricity passes through a solution depends upon the relative amounts of the ions present, it is possible to estimate the percentage of mineral matter or ash in a sample of raw sugar by determining the conductivity of its solution. Resistance is the inverse of the conductivity, and it is the resistance of the test solution that is actually measured. As will be shown below it is usual to correlate the results obtained by electrometric apparatus with the sulfated ash by means of suitable equations. In many classes of routine work such correlation is not necessary and the comparative conductances will serve equally well.

The specific conductance is affected only by the salts in solution, whereas the chemical ash represents the sum of the soluble and insoluble portions, and this is one source of discrepancy between the two methods. For accurate comparison it is therefore necessary to filter the solution before making the determination of chemical ash and conductimetric ash.

541. Apparatus. Directions for assembling bridges and cells may be had by consulting the original articles of Zerban and his associates but assemblies of various types especially designed for sugar work are obtainable (Fig. 155 shows Leeds and Northrup sugar ash bridge). If such a bridge is used, no calculation of the cell constant is necessary, as this is done in effect by the bridge itself. In the determination of the cell constant, the resistance dial is set to theoretical calculated resistance of the standard KCl solution (either 0.02 N or 0.01 N), the temperature-compensating dial is turned to the temperature corresponding to the thermometer reading, the current is switched on (alternating current is essential), and the cell constant dial is adjusted until the system is in equilibrium as shown by the galvanometer. The value thus found is the cell constant, and the dial is locked at this setting so that the instrument reads in true reciprocal ohms at any subsequent setting.

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[12] *Facts About Sugar,* March 20, 1926.

[13] *Facts About Sugar,* Dec. 4, 1926; Oct. 8, 1927; July 21–28, 1928.

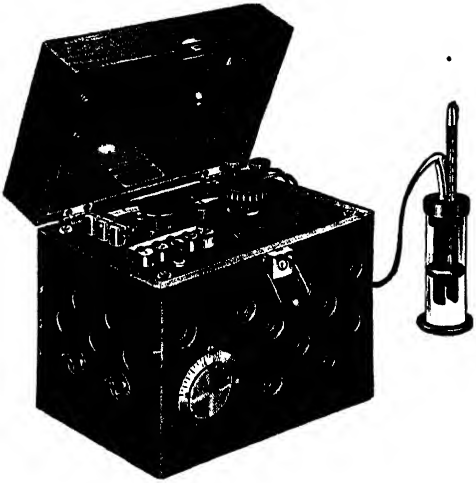

FIG. 155. Sugar Ash Bridge.

542. Single Conductivity Measurement. Twenty-five grams of a well-mixed sample of the sugar is washed into a 500-ml. flask with conductivity water, dissolved, and then poured into the cell up to the 40 mark after several rinsings with the solution. The cell constant dial being locked in the proper position, the temperature-adjusting dial is turned to the temperature indicated, and the resistance dial is then rotated until the galvanometer shows zero deflection. The instrument now reads specific conductivity of the solution.

543. Calculation of Ash of Raw Sugars by *C* Ratio. The percentage of sulfated ash is calculated by means of the equation.

$$\text{Per cent ash} = C \times K$$

where K = conductivity;
C = a constant.

This constant is widely variable according to districts, as well as to different centrals in the same district, and must be determined by each factory for its own sugar. The best way to determine ash is to construct a curve, made by the chemist from a number of representative samples of the sugar, plotting conductivity (as experimentally determined) against sulfated ash, minus one-tenth (also as experimentally determined).

Zerban and Sattler published a large number of ratios for sugars from different factories, different provinces in Cuba, and different countries.[14] The average maximum and minimum values of C for the different localities are given to illustrate the variations that occur.

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[14] *Facts About Sugar,* Dec. 4, 1926.

LOCALITY	HIGHEST	LOWEST	AVERAGE
Cuban sugars, Province of Camagüey	1853	1666	1743
Province of Habana	1823	1605	1730
Province of Matanzas	1835	1683	1755
Province of Oriente	2022	1671	1796
Province of Pinar del Río	1820	1624	1689
Province of Santa Clara	1853	1652	1765
Hawaiian sugars	1775	1476	1630
Puerto Rican sugars	1787	1501	1672
Philippine sugars	1898	1665	1816
Santo Domingo sugars	1903	1678	1802
British West Indian sugars	1773	1481	1560

544. Acid Conductivity Method. The discrepancies in the value of the constant as shown above are due, among other things, to the variation in the composition of dissolved salts. This has been studied by Zerban and Sattler,[15] and they have worked out a method whereby this factor is eliminated, and the ash can be calculated from the equation

$$\text{Per cent soluble ash} = 0.0001757 \times (9.13K + 1935 - K_1)$$

where K = the specific conductance $\times 10^6$ of the sugar solution, determined as described above;

K_1 = the specific conductivity $\times 10^6$ of the sugar in acid solution.

When the ash is determined by this method, the usual discrepancies attendant to the first method are not found, and the equation can be used for all classes of sugar with negligible variations between the calculated results and the experimental chemical ash.

In this method, the solution is prepared in the same way as for the single-conductivity method, and the first (water) conductivity is made in the same way as described above (Sec. 542). Then a 200-ml. portion of the solution (25/500) is introduced into a Kohlrausch flask, 5 ml. of 0.25 N HCl, accurately measured, is added, and the flask is shaken thoroughly. This solution is then placed in the cell, and the conductivity is again determined. These two conductivities are inserted in the equation and calculated to per cent sulfated ash. In setting the temperature-compensating dial on the Leeds and Northrup sugar ash bridge for readings "with acid," the settings must be slightly changed as follows:

Temperature of solution	20°	22.0°	24.0°	26.0°	28.0°	30.0°
Correct dial settings	20°	21.5°	23.1°	24.6°	26.2°	27.7°

The hydrochloric acid (0.25 N) used must be very carefully adjusted as to concentration. Titration against standard alkali by the use of indicators is not accurate enough for this purpose. It is best to control the strength of the acid by its specific conductance. A mixture of 200 ml. of conductivity

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[15] *Facts About Sugar,* Oct. 8, 1929.

water and 5 ml. of the acid used in this work should have a corrected specific conductance of 0.002370 at 20° C.

Care must be taken to cleanse the electrodes thoroughly after measuring the solutions containing the acid. This is best accomplished by first washing them with water and then filling the cell with 30 per cent methyl alcohol to remove the acid absorbed by the platinum black. The alcohol may be drained off for future use after standing a few minutes, and the cell is again washed and filled with water. In making routine tests all the determinations without acid may be run first and then all those with acid.

545. Soluble and Insoluble Ash of Raw Sugars in Unfiltered Solutions. Sattler, Mull, and Lorge[16] made determinations on filtered and unfiltered solutions and developed formulas that show that it is possible to calculate either the total ash or the water soluble ash from conductivity determinations on unfiltered solutions of raw cane sugar.

The method used is the acid conductivity method (Sec. 544) in which two determinations are made, one on the unfiltered sugar solution (25 grams to 500 ml.) and the second on a 200-ml. portion of that same unfiltered solution to which has been added 5 ml. of 0.25 N HCl.

Their formulas are as follows:

$$\text{Per cent total ash} = 0.001566K - 0.0001954K_1 + 0.4160 \qquad (1)$$

$$\text{Per cent soluble ash} = 0.001558K - 0.0002024K_1 + 0.4174 \qquad (2)$$

where K = the specific conductance × 10^6 of the unfiltered sugar solution
K_1 = the specific conductance × 10^6 of the acid unfiltered sugar solution

546. Conductimetric Method for Refinery Remelts and Soft Sugars. For refinery remelts and soft sugars Zerban and Sattler[17] recommend the use of the acid conductivity method (Sec. 544) with the formula

$$\text{Per cent soluble ash} = 0.0001695(9.13K + 1935 - K_1)$$

547. Conductimetric Ash in Raw and Refinery Sirups and Molasses. *Method of Three Conductivity Determinations.* Make a solution containing 2.5 grams of product plus 22.5 grams of ash-free sucrose in 500 ml. (a high-grade tablet sugar may be used for the ash-free sucrose). Three conductivity determinations are made (1) on the sugar solution itself, (2) on that of a solution made by taking 200 ml. of sugar solution plus 5 ml. of 0.25 N KOH, and (3) on that of a solution made by taking 200 ml. of sugar solution plus 5 ml. of 0.1 N H_3PO_4. The specific conductances multiplied by 10^6 are designated as K, K_2, and K_3 respectively.

$$\text{Per cent ash} = 0.0191369K - 0.002249K_2 - 0.001210K_3 + 3.07$$

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[16] *Facts About Sugar,* October, 1935.

[17] *Ind. Eng. Chem., Anal. Ed.,* **3**, 41 (1931).

A nomograph to facilitate the calculations is published with the original article, reference to which is given in Sec. 546.

548. Conductivity Water. The first distillation should be made over alkaline potassium permanganate in any convenient still after which the water is redistilled in a Pyrex flask, using a condenser of block tin. Usually, after the first still has been in use for some time, the water issuing therefrom will be found to have a specific conductance equal to the redistilled water, namely, of about 1.2 to 1.4×10^{-6}. When this state exists, the redistilling may be discontinued, as water of lower conductivity deteriorates rapidly, and besides, this conductivity is quite good enough for this type of work.

549. Choice of Conductimetric Method. Zerban[18] says, "The general rule for conductivity ash determinations in all classes of sugar products is to use the simple *C*-ratio method wherever possible, and to resort to the more elaborate formulas only when actual comparisons with chemical ash determinations prove this to be necessary." It may be added that in many classes of work the conductivity determination alone without any factor may be used for comparative work, e.g., char filter investigations on ash removal. It has also been suggested that some empirical factor might be used and the ash reported as "salts by conductance."

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[18] Browne-Zerban, *Sugar Analysis,* 1941, p. 1029.

CHAPTER 23

HYDROGEN-ION (pH) CONTROL

550. Introduction. When this chapter was written for the previous edition of this handbook, hydrogen-ion (pH) control was a relatively new procedure. The colorimetric methods had been fairly well developed but the electrometric determination was still considered too complicated for general routine work. More recent developments, particularly the introduction of the glass electrode, have changed the situation materially, and potentiometers are now made in such simple and rugged form that electrometric tests for industrial routine work are quite common. The colorimetric methods have the advantage of simplicity, the equipment required is inexpensive, and the results are sufficiently accurate for most sugar control purposes, but wherever possible the electrometric apparatus should be at hand for checking purposes and for determinations on dark-colored solutions.

551. Advantages of Hydrogen-Ion Control. The determination of hydrogen-ion concentration (pH) as a measure of the reactions of juices, sirups, and other sugar solutions has become an essential part of the control in cane sugar factories and refineries throughout the world. It has many advantages over the old system of test papers and titrations; most important of which is that hydrogen-ion concentration is the measure of the *intensity* of the acidity or alkalinity whereas the older methods measured only the *quantity* of acidity or alkalinity. Since the sucrose inverting power of an acid is a direct function of its degree of dissociation, i.e., of the amount of hydrogen-ions in solution, the importance of this advantage in the sugar industry cannot be too greatly stressed. In fact, this relationship is so exact that the rate of inversion of sucrose has been used as a measure of the H-ion concentration of biochemical solutions.[1] Another advantage of the pH control is the simplicity of the colorimetric determination. Unskilled workmen can readily be trained to make the test, and the numerical expression, or pH number, is easily understood by them. The fact that the test is as accurately carried out by artificial light as by daylight is an added advantage over test papers and titrations.

552. Theory of Hydrogen-Ion Concentration. No theoretical knowledge of the subject is necessary to the use of routine methods, but a concise statement of the principles involved is here given. In all aqueous solutions

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[1] Clark, *The Determination of Hydrogen Ions*, p. 296.

it has been proved by electrometric measurements that the product of the hydrogen-ion concentration (H^+) and the hydroxyl-ion concentration (OH^-) always equals a constant at constant temperature.

$$(H^+)(OH^-) = \frac{1}{100{,}000{,}000{,}000{,}000} = 10^{-14} \qquad (1)$$

Therefore, no matter what the concentration of hydroxyl ions in the solution, there must be sufficient hydrogen ions to satisfy the equation, so that we are able to speak of the hydrogen-ion concentration of alkaline solutions, since by so doing we are also stating indirectly the hydroxyl-ion concentration.

The direct expression of hydrogen-ion concentration gives a series of unwieldy fractions, and to avoid these Sorensen in 1909 suggested the use of the logarithm of the reciprocal of the H-ion concentration, which he found to be a function of the potential of the hydrogen electrode in determining hydrogen-ion concentration electrically. The equation

$$pH = \log \frac{1}{(H^+)} \qquad (2)$$

where (H^+) represents the concentration of H-ions in gram-atoms per liter, defines H-ion concentration quite as well as (H^+) itself. As pH is the logarithm of the reciprocal of a power of 10, it follows that it equals numerically the exponent or power to which 10 is raised. For example, in absolutely pure distilled water the H-ion concentration and the OH-ion concentration are exactly equal and since according to equation 1 the product of the two must be 10^{-14} the concentration of each mus tbe 10^{-7}. Hence by equation (2) $pH = \log \frac{1}{10^{-7}}$ and since $\log \frac{1}{10^{-7}} = 7$, pH = 7.0, which represents neutrality on the pH scale.

If the H-ion concentration exceeds the OH-ion concentration the solution is acid and the pH number is below 7.0, but if the reverse is true, i.e., if there are more hydroxyl ions than hydrogen ions in the solution, the solution is alkaline and the pH is above 7.0. Thus it is seen that the pH numbers form an acidity-alkalinity scale in which the *lower* the pH the *higher* the hydrogen-ion concentration (the pH numbers being reciprocals of H-ion concentration). From this it follows that below 7.0 the lower the pH the greater the intensity (or inverting power) of the acid, whereas above 7.0 the alkalinities *increase* with the *increase* of pH, since the hydroxyl-ion concentration increases with the increase of the pH, i.e., with the *decrease* of the H-ion concentration. Another point to be remembered is that the pH scale is a logarithmic scale, representing the powers of ten, and that therefore an acid solution of 5.0 pH, for example, would have ten times the sucrose inverting power of one of 6.0 pH while a solution of 4.0 pH would have 100 times the inverting power of the 6.0 pH solution. Similarly, a solution of 9.0 pH would have ten times the intensity of alkalinity of one of 8.0 pH, and one of 10.0 pH would be 100 times as alkaline, etc.

553. Buffer Action and Buffer Solutions. Clark[2] defines buffer action as "the resistance exhibited by a solution to change in pH through the addition or loss of acid or alkali." A buffer solution is one which represses the ionization of acids or bases or resists a change in pH when acids or bases are added. Buffer action is dependent on the nature and concentration of the constituents of the solution; on the nature of the acid or alkali added to the solution to effect the change in pH; and on the pH region where the buffer action is measured. The reader is referred to standard treatises on the subject for a full exposition of the theory. It is sufficient to say that the solutions encountered in cane sugar factory control show buffer action to a high degree, and most of the refinery materials are similarly buffered. However, solutions of pure granulated sugar show no buffer action at all whereas high-grade char-filtered liquors are only slightly buffered. In general, the lower the purity the greater the buffer action.

For the colorimetric determination of H-ion concentration, standard solutions of known pH called buffer mixtures must be employed. Buffer mixtures are buffer solutions (generally consisting of weak acids and salts of weak acids or mixtures of both) of such definite composition that they can easily be reproduced, and whose pH value has been accurately determined by electrometric measurements. Elaborate studies by many investigators have been made in developing buffer solutions of known pH, a notable example being the series of Clark and Lubs which rise in increments of 0.2 pH from 2.0 to 10.0 pH.

554. Colorimetric Determination of pH. Basis of the Test. The colorimetric determination of pH depends upon the fact that certain indicators have a transition interval in which the color varies with changes in H-ion concentration. The test is made by comparing the color of the solution to be tested, to which has been added a known amount of a suitable indicator, with a series of buffer mixtures of known pH carrying an equal amount of the same indicator. The materials required for the tests are: (1) standard solutions of known pH, (2) suitable indicators of proper strength, (3) test tubes of clear glass of 15-mm. diameter by 120-mm. length, and (4) a color comparator to facilitate matching test solution and standards.

555. Standard Solutions. The best known of the buffer solutions are those of Clark and Lubs. For the preparation of this series the following solutions are needed: 0.2 *M* NaOH, 0.2 *M* potassium acid phosphate and 0.2 *M* boric acid and potassium chloride; all of these are best purchased ready prepared. The standard buffer mixtures from pH 5.8 to pH 9.0 are made as follows.

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[2] *Loc. cit.*, p. 40.

COMPOSITION OF BUFFER STANDARDS AT 20° C.
(Clark and Lubs)[3]

pH	Milliliters 0.2 M KH_2PO_4	Milliliters 0.2 M NaOH	Milliliters 0.2 M H_3BO_3	Milliliters 0.2 M KCl
6.0	50	5.70		
6.2	50	8.60		
6.4	50	12.60		
6.6	50	17.80		
6.8	50	23.65		
7.0	50	29.63		
7.2	50	35.00		
7.4	50	39.50		
7.6	50	42.80		
7.8	..	2.61	50.0	50.0
8.0	..	3.97	50.0	50.0
8.2	..	5.90	50.0	50.0
8.4	..	8.50	50.0	50.0
8.6	..	12.00	50.0	50.0
8.8	..	16.30	50.0	50.0

Investigators in Queensland found[4] that the Kolthoff buffer mixtures had a much greater buffering effect than the Clark and Lubs series and also that the Kolthoff solutions kept without deterioration over a period of several weeks, whereas the Clark and Lubs mixtures lasted only a few days as checked electrometrically.

COMPOSITION OF BUFFER STANDARDS AT 18° C.
(Kolthoff)

pH	0.1 M KH_2PO_4 *ml.*	0.05 M Borax *ml.*
5.8	9.21	0.79
6.0	8.77	1.23
6.2	8.30	1.70
6.4	7.78	2.22
6.6	7.22	2.78
6.8	6.67	3.33
7.0	6.23	3.77
7.2	5.81	4.19
7.4	5.50	4.50
7.6	5.17	4.83
7.8	4.92	5.08
8.0	4.65	5.35
8.2	4.30	5.70
8.4	3.87	6.13
8.6	3.40	6.60
8.8	2.76	7.24
9.0	1.75	8.25

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[3] Clark, p. 107.

[4] *Intern. Sugar J.*, February, 1932, p. 64.

The buffer standards of McIlvaine have a range of 2.2 to 8.0 pH and have the advantage that only two stock solutions are required. The solutions are 0.2 *M* disodium phosphate ($Na_2HPO_4 \cdot 2H_2O$) and 0.1 *M* citric acid ($C_6H_8O_7 \cdot H_2O$).[5]

The solutions for making these are now obtainable from chemical supply houses or the standard buffer solutions themselves may be purchased ready-made at reasonable rates.

As a simple check standard at 7.0 pH for buffer solutions (which may change on standing or be improperly made up) Williams and Lyman[6] suggest a 1 per cent solution of ammonium acetate. They proved that solutions of the pure salt of widely different strengths (0.5 *N* to 0.05 *N*) made under ordinary laboratory conditions (exposure to air, unboiled distilled water, etc.) are always so extremely close to neutrality as to serve as a 7.0 pH standard for both colorimetric and potentiometric methods.

The buffer solutions can conveniently be stored in 250-ml. reagent bottles with a one-hole rubber stopper carrying a 10-ml. pipet. By providing each bottle with its own pipet it is not necessary to waste solutions to rinse the pipet.[7]

The average laboratory will find it more satisfactory to purchase the buffer mixtures ready-made, the series ranging from 6.0 to 8.4 being those most generally needed in routine factory or refinery control.

556. Indicators. The indicators for routine use are:

Bromthymol blue	0.04 per cent solution, pH range	6.0–7.6
Phenol red	0.02 per cent solution, pH range	6.8–8.4
Cresol red	0.02 per cent solution, pH range	7.2–8.8

The preparation of these dyes according to Clark follows. Grind the amount of the pulverized dye given in an agate mortar with the number of milliliters of 0.05 *N* NaOH shown.

	Gram, Dye	Milliliters, 0.05 *N* NaOH	Make to
Bromthymol blue	0.4	14.1	1000 ml.
Phenol red	0.2	12.5	1000 ml.
Cresol red	0.2	11.7	1000 ml.

Schlegel and Steuber recommend storage of dye solutions and buffer mixtures in glass-stoppered Pyrex glass bottles.[8] They object to paraffin for sealing as ordinary paraffin has an acid reaction. Their findings as to variations in the reaction of purchased dye solutions are in accord with the experience of the writer, and the preparation of the dyes in the laboratory is recommended rather than the purchase of the ready-made solutions.

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[5] Browne-Zerban, *Sugar Analysis,* 1941, p. 558.

[6] *J. Am. Chem. Soc.,* **54,** 1932, p. 1911.

[7] *Laboratory Methods, Cuban Sugar Club,* second edition, Havana, 1925.

[8] *Ind. Eng. Chem.,* May, 1927.

557. Standard Color Tubes. Add 10 ml. of each of the standard buffer mixtures from 6.0 to 7.6 pH to separate test tubes after which 0.5 ml. (accurately measured) of the bromthymol blue indicator is added to each tube. Corked and sealed with sealing wax these tubes constitute the color standards for the bromthymol blue range. Similarly, tubes containing 10 ml. of each of the standard buffer mixtures from pH 6.8 to 8.4 are prepared and 0.5 ml. of phenol red indicator added to each to make the phenol red standards. Cresol red standards range 7.2 to 8.8 may be used instead of phenol red.

For this purpose Pyrex test tubes of 15-mm. diameter by 120-mm. length having approximately the same bore should be thoroughly cleaned and rinsed with distilled water before filling. Corks should be boiled and thoroughly rinsed before inserting. The addition of a drop of toluene to each standard to prevent mold has been recommended. Standards so prepared will last three to four weeks in routine use, or longer if protected from heat and light.

Ready-made color standards in sealed ampoules may be purchased which are fairly constant for several months if kept in a cool place, but they deteriorate rather rapidly when exposed to light and routine control conditions. Cameron[9] found many errors in Queensland mills due to the use of purchased standards and purchased buffer solutions. He recommends periodic checks on all colorimetric apparatus and standards by means of the electrometric method. As will be described later non-fading glass standards are now obtainable. Standards may also be made with suitable mixtures of metallic salts.

558. Color Comparator.[10] A convenient comparator is shown in Fig. 156. It consists of a sliding tube rack carrying alternate tubes of distilled water and color standards and a metal frame or cover with suitably arranged apertures for comparing the colors. Immediately in front of the sliding rack are sockets for two removable tubes for the solution to be tested, spaced the same distance as the tubes in the rack. Ordinary daylight or any of the artificial daylight lamps serve as a light source, the simplest arrangement being to place a white surface at 45° behind the apertures and hang a shaded Mazda Daylite globe above this. Several slide comparators, following the design of the one here described, are on the market, some of which

Fig. 156. Color Comparator for pH Determinations.

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[9] *Intern. Sugar J.*, 1932, p. 64.

[10] Meade and Baus, *The Planter,* June 27, 1925.

are made of plastic instead of metal. A rotary comparator carrying a greater range of color standards than is possible with the slide type, is also obtainable.

559. Procedure of Test. Test tubes of the same bore as those used for the color standards but 150 mm. long are used for the test solutions, and those tubes for convenience should be marked for 10 ml. (marking in laboratory with Diamond Ink), 15 ml. and 20 ml. If the solution to be tested is not to be diluted, 10 ml. is measured into each of two tubes, 0.5 ml. (accurately measured) of indicator is added to one of these, and the dye and solution is mixed by rotating the tube or by a stirring rod or by closing the tube with a clean cork and shaking. Under no circumstances should the tube be closed with the finger or hand because of the adverse effect of the acid perspiration. The tube containing the dye solution is placed before the left-hand aperture, the blank before the other aperture, and the rack is moved back and forth until the standard to match the test sample is behind the blank solution. Thus any color and turbidity in the blank solution is added to the color of the standard and compensates for the color and turbidity in the test solution, while the tube of distilled water behind the test solution compensates for the water in the blank.[11] Workmen of ordinary intelligence can be trained to make a test with this comparator to 0.1 pH.

FIG. 157. Koch Pipet for pH Dyes.

Most investigators say that dilution has little or no effect on the pH of all but the purest solutions encountered in sugar work, e.g., high-grade refinery liquors and solutions of granulated sugar, whereas others show that dilution is to be avoided.

In the writer's experience dilution even with such proportions as 1 to 3 or 1 to 5 affects results noticeably in such high-test solutions as washed sugar liquor in the refinery. The best practice would seem to be to avoid dilution except where this is necessary to minimize the effects of color and turbidity. Adherence to a fixed dilution with each grade of material tested will give comparable results, and the alteration in the pH due to the dilution can be checked from time to time by electrometric methods. The use of kieselguhr and filter papers should be avoided. Filter-Cel is acid and Hyflo Filter-Cel alkaline, and all ordinary laboratory filter papers are acid.

A 1:3 dilution can easily be effected in routine control by having test tubes marked at 10, 15, and 20 ml. Distilled water is added to the 15-ml. mark, then the sugar solution to 20 ml., and the two are mixed by pouring from one tube to another. Half the mixture is then poured back in the first

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[11] Walpole, *Biochem.*, Jan. 5, 1910, p. 207.

tube, 0.5 ml. of indicator dye added and the comparison made as before. The measurement of the 0.5 ml. of indicator solution can conveniently be carried out by the Koch measuring pipet arranged as shown in Fig. 157. The soda-lime tube attachment prevents contamination by the CO_2 of the air. Solutions should be tested at or near room temperature as the pH is quite markedly affected by differences in temperature.

A further simplification of the above procedure has been devised by the writer for use by the blowup men in refinery control. Phenol red dye is diluted with neutral water until each 7.5 ml. contains 0.5 ml. of the standard dye (66.6 ml. phenol red 0.02 per cent: 1000 ml. distilled water).

Test tubes marked at 7.5 ml. are used, to one of which is added the diluted dye, to the other any clean tap water; then a 2.5-ml. (approximately) measure of material to be tested is added to each tube. Clean corks are placed in the tubes, the solutions are shaken, and the readings are taken in the comparator as before. This method does away with the use of neutral water and with the necessity of accurate measurement of the dye by unskilled workmen and reduces the possibility of contamination to a minimum. Tap water is used for rinsing tubes and corks. The diluted dye keeps for two or three days but it is best made up daily in the laboratory. Constant checks by regular laboratory methods have proved this simplification to be thoroughly reliable.

Spot Test. As described by Brewster and Raines: [12]

> . . . this consists of adding one drop of the appropriate indicator solution to three drops of the test liquid contained in the depression of a porcelain spot plate. The color developed in the test spot is matched with similar spots made by adding a drop of the same indicator to three drops of a standard buffer solution. The pH of the standard buffer which matches the test solution in hue is the same as the pH of the test solution.

The disadvantages are obvious, the greatest of which, namely, the necessity of making spot standards for each test has been overcome by the Hawaiian Sugar Planters Association by the adoption of spot color standards made by means of dyes on celluloid.[13] This has the advantage of doing away entirely with standard buffer solutions, but it does not compensate for the color and turbidity of the test solution. Cook [14] later reported on a comparison between a thousand tests by this method and by potentiometer and showed that in general agreement was within pH 0.2, more reliable results being obtained with dilution of juice 1:3 than on undiluted juice. Some wide differences were noted, particularly in the upper range of bromthymol blue, but these were obviated by using phenol red and cresol red for pH 7.2 to 7.6. Cook again reported on the spot test method with these dyes (phenol red and cresol red) [15] and found that the average person interprets the values as less alkaline

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[12] *The Planter,* Sept. 9, 1922.

[13] H. A. Cook, *Facts About Sugar,* June 20, 1925.

[14] *Intern. Sugar J.,* 29, No. 348 (1927).

[15] *Intern. Sugar J.,* March, 1933, p. 125 (abs.).

than they really are, because of inherent faults in the spot plate method. He also emphasized the need to keep indicator solutions adjusted to the specified reaction.

560. Test Papers. In Europe test papers made of cresol purple, bromthymol blue, corallin and phenol red have been indorsed by Prinsen Geerligs and Dr. Herzfeld[16] as being simpler than American colorimetric practice, but these have not found favor on this side of the water.

561. *Helige Comparator with Glass Color Standards.* This is shown in Fig. 158. Each indicator dye has a separate color disk similar to the one shown to the right of the comparator, in which are arranged eight or nine permanent glass standards made from solid glass, non-fading. The test is made as described before using a blank in one of the square tubes, and the test solution with 0.5 ml. of dye in the other. The proper color disk, inserted in the instrument, is rotated until the color of the glass and the test solution match, when the pH is read in the small aperture on the right front face of the comparator. To facilitate matching, a prism is placed in the eyepiece which brings the two colors together in the halves of a circle, similar to the field of a polariscope.

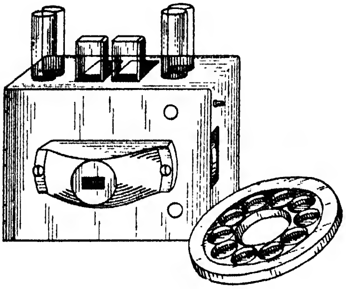

FIG. 158. Helige pH Comparator.

Care must be taken to use only correct daylight (either real or artificial) for this instrument; otherwise the color disks will not match the dye solutions.

562. Neutral Water or Dawson Water. Distilled water is always acid, ranging from 6.0 to 6.4 pH, depending upon how recently it has been boiled. The pH is so sensitive to absorbed CO_2 that a drop in pH of several tenths may be effected by pouring recently boiled distilled water from one test tube to another.

So-called Dawson water[17] or neutral water may be made by double distilling water and then boiling off one-third of the volume after which it is stored in Pyrex containers, fitted with a soda-lime tube or kept from contact with air. Schlegel and Steuber[18] found this water to be a reliable reference standard for the preparation of bromthymol blue solutions. The water will not keep its neutral character in ordinary routine determinations. An ordinary distilled water is satisfactory for dilutions for most sugar house and refinery materials, except such materials as granulated sugar or high-test white liquors, which are so slightly buffered as to be affected by the acidity of ordinary distilled water.

[16] *Intern. Sugar J.*, **27**, No: 317 (1925).

[17] L. E. Dawson, *J. Phys. Chem.*, **27**, 551 (1921).

[18] *Loc. cit.*

563. Sources of Error in Colorimetric Determinations. Certain obvious errors are to be avoided such as the use of tubes of different bores, inaccurate measurement of indicator dye, working with hot solutions, contamination of tubes or solutions by contact with hands, failure to rinse tubes before making test. The greatest possibility of error is in the use of color standards that have faded because of exposure to light, heat, or air.

Much greater precautions must be exercised in testing unbuffered or slightly buffered solutions. Here exposure of test solutions to CO_2 of the air, minor variations of reaction of dye solution itself, use of other than neutral water for dissolving or diluting, and excessive dilution may all cause such wide variations as to vitiate the value of the test completely.

ELECTROMETRIC DETERMINATIONS OF pH

564. General Considerations. The electrometric determination of pH is carried out by measuring the voltage generated by two electrodes, one immersed in the solution to be tested and the other in a solution of known concentration, the solution being joined electrically by a bridge of neutral salt solution. The hydrogen electrode is the basic standard for all electrometric pH measurements, but it is a "tool for the research chemist,"[19] and other electrodes have been developed for practical work. These are the calomel electrode, the quinhydrone electrode, the antimony electrode, and most recently the glass electrode. The theoretical considerations on which electrometric pH measurements are based may be found in reference books such as those of Clark,[20] of Kolthoff,[21] and of Dole.[22] A concise description is given in the trade publication, *Notes on Hydrogen Ion Measurements,* Leeds and Northrup Co., 1930.

565. Hydrogen Electrode. This consists primarily of a small piece of platinum coated with platinum black and held in a solution through which hydrogen gas is continuously bubbled. In another form the hydrogen is confined in the solution in a closed vessel which is continuously rocked or shaken. The hydrogen electrode formed by the absorption of the gas by the platinum black develops a potential that is directly related to the hydrogen-ion concentration. As stated above the hydrogen electrode is adapted to research work and not to routine determinations, but it is the fundamental standard on which all electrometric methods are based.

566. Calomel Electrode. The calomel electrode consists of mercury in contact with calomel in a solution of potassium chloride saturated with calomel sealed in a glass tube with a piece of platinum wire which makes contact with the mercury. The chemicals used must be extremely pure. The calomel electrode is used generally in practical work as a reference electrode

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[19] Balch and Paine, *The Planter,* Oct. 31, 1925.

[20] *The Determination of Hydrogen Ions,* second edition, 1928.

[21] *Colorimetric and Potentiometric Determination of pH,* 1931.

[22] *The Glass Electrode,* 1941.

in place of the hydrogen electrode, and it is a part of most of the laboratory and industrial pH assemblies described later.

567. Quinhydrone Electrode. This was among the earliest of the practical methods of pH determination, and it consists of a gold or platinum electrode immersed in the test solution which is saturated with quinhydrone.

Quinhydrone is a compound (or complex) in which quinone and hydroquinone are combined in equimolecular proportions. When it is dissolved it dissociates into these two substances, one of which ionizes.

In a solution containing hydrogen ions and the dissociation products of quinhydrone, the potential of the electrode is directly related to the pH value and the temperature of the solution, if the ratio of the hydroquinone concentration to that of quinone is constant and practically equal to 1. This substance is so slightly soluble in acid that the quantity necessary to saturate a sample of sufficient volume for pH measurements and to provide an excess which will insure the presence of its dissociation products is only a few milligrams. It can be estimated without weighing, as the undissolved quinhydrone does not influence the potential.

In a solution more alkaline than 8 pH quinhydrone is more soluble, and the hydroquinone dissociates and becomes oxidized. The proper ratio of quinone to hydroquinone is not maintained, and the potential of the electrode is altered to such an extent that the measured voltage is no longer a linear function of pH. For this reason the quinhydrone electrode is not suitable for use in decidedly alkaline solutions (pH 9.0 or above).

Several complete potentiometric outfits are on the market, the one shown in Fig. 159 being made by Leeds and Northrup.[23] This indicator consists of two essential parts, the standardizing system and the operating system. The standardizing system consists of a Wheatstone bridge in one arm of which the standard is connected, another arm contains a fixed resistance, and the third and fourth arms are formed by a variable resistance. The null point indi-

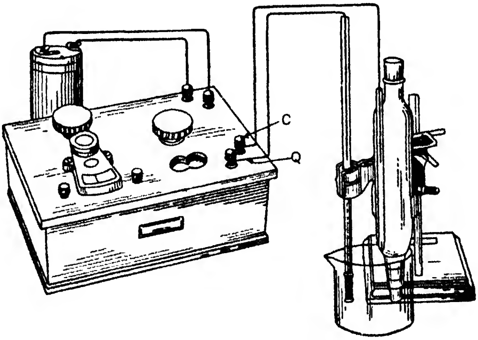

FIG. 159. Electrometric pH Apparatus with Quinhydrone Electrode.

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[23] From description supplied by makers.

cating galvanometer of a highly sensitive type is bridged across the system from the swinging arm of the resistance to the contact point between the standard cell and the fixed resistance. Power for operating the bridge is derived from a single dry cell battery of about 1 to 1½ volts.

The operating system is similar to the standardizing system; in fact, it is the same system except that a special quinhydrone cell is substituted for the standard cell. The quinhydrone cell has two parts. One part is a standard calomel half cell. The other part is a gold electrode.

To standardize the potentiometer, press the plug switch located just to the left of the galvanometer scale. This closes the Wheatstone bridge circuit through the standard cell which is mounted within the box. The needle of the galvanometer is brought back to zero by rotating the knurled knob which controls the variable resistance, after which operation the plug switch is released.

The potentiometer is ready for a determination and need not be reset, except for an occasional check. To make a pH determination of an unknown solution, the calomel cell and electrode are completely immersed in the liquid in a small beaker. Quinhydrone is then stirred into the solution until saturated.

The solution is then stirred and the plug switch, just at the right of the galvanometer scale, is pressed, closing the bridge through the quinhydrone-calomel cell circuit. The needle will then swing to the right or left, depending on the hydrogen-ion concentration of the solution. The knurled knob on the right is then rotated until the needle returns to zero, and the reading noted on the scale just in front of it. The setting should be checked several times as it takes about one-half minute for the system to come to equilibrium. Take the solution temperature with a mercury thermometer and with these data, read directly the pH value from the graph curves supplied with the instrument. The more recent models read directly in pH (1 to 9) at 25° C.

568. Antimony Electrode. The antimony electrode has replaced the earlier tungsten electrode which was first used for the continuous automatic determination of pH at the clarification station. (See p. 83.) The antimony electrode is made of a cast-antimony rod or a pellet of the metal. Its theory and practical use have been discussed by N. J. King.[24] The antimony electrode is especially adapted to this factory control work because it is rugged and is not affected by movement in the test solution. A pH meter used in Queensland for controlling the liming of the juice[25] has an antimony electrode and a calomel cell as a reference electrode.

569. The Glass Electrode. The introduction of the glass electrode in recent years has greatly simplified electrometric pH work. The development of the glass electrode dates from 1906, and the interested student is referred to Dole's book on the subject for an exposition of the theory. The electrode is a thin glass bulb of special composition (Corning No. 015 is suitable)[26] which contains an inner platinum electrode in an acid solution saturated with quinhydrone. (Other more stable combinations which will not deteriorate

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[24] *Ind. Eng. Chem., Anal. Ed.,* No. 5, 1933, p. 323.

[25] *Intern. Sugar J.,* January, 1941, p. 21.

[26] "Polarimetry, Saccharimetry and the Sugars," *Bur. Standards,* 1942.

such as silver, silver chloride, potassium chloride are used.) According to Dole[27] the glass electrode acts as a perfect hydrogen electrode, i.e., the emf. on the surface of the glass changes with the change of pH in exactly the same way as the hydrogen electrode emf. changes.

Because of the high resistance of the glass the difference in potential must be measured by a highly sensitive galvanometer, or electrometer, and amplification of the differential by means of special vacuum tubes is necessary.

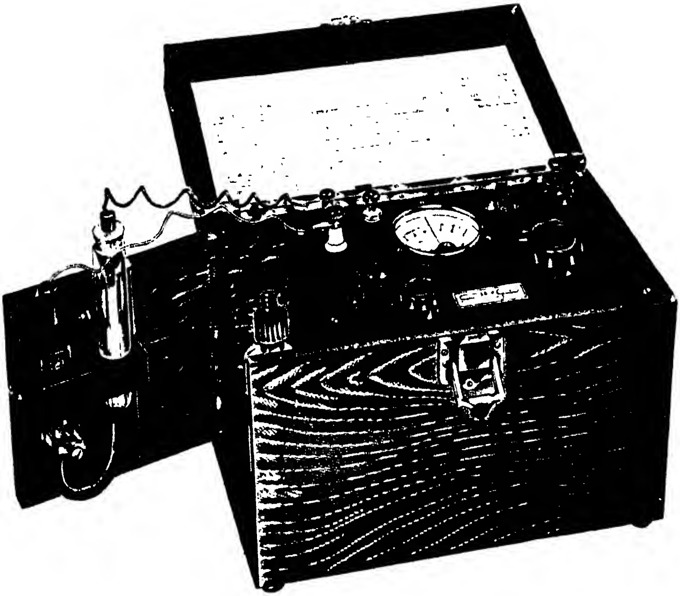

FIG. 160. Coleman pH Meter.

Several self-contained units consisting of potentiometer, galvanometer, pH electrodes, amplifying tubes, temperature compensation, etc., are on the market in compact portable form. The one shown in Fig. 160 is the Coleman pH electrometer. Similar instruments are those of Leeds and Northrup, Cameron, Helige, Beckman, and others. A portable English instrument using the glass electrode is sold under the name Ionometer.[28] These instruments may be operated by any laboratory helper of ordinary intelligence and are accurate in routine work to 0.1 pH or less. The electrodes are factory sealed and are mounted on a bracket on the door of a compartment of the case of the instrument. The instruments read directly in pH and have automatic temperature correction which can be set by the operator for any temperature

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[27] *Loc. cit.*, p. 26.

[28] *Intern. Sugar J.*, June, 1939, p. 237.

between 10° and 40° C. They are calibrated against a known buffer which is generally carried in the case as a part of the assembly.

The directions for operating these instruments are given on the inside of the lid of the case, and only a few adjustments and settings by the operator are required to obtain the reading in pH.

The range of the glass electrode instruments is from 1 to 13 pH except for highly alkaline solutions containing an excess of sodium salts. The use of shielded glass electrodes in automatic industrial pH control is described in Sec. 99.

CHAPTER 24

COLOR AND TURBIDITY IN SUGAR PRODUCTS

COLOR DETERMINATION

570. General. The need for determining the amount of coloring matter present in sugars and sugar solutions is evident since an important part of the refiner's work is the removal of such coloring matter.

The physicist defines color as the sensation due to the stimulus of the optic nerve but Zerban says,[1] "The sugar chemist is, as a rule, not primarily interested in *color* as defined by the physicist, but wants information on the quantity and nature of the *coloring matter* present in sugar products." In this chapter the term color will be used synonymously with coloring matter because it is commonly used in this sense in sugar work.

In the previous edition of this book the determination of color in sugar products was said to be in an unsettled and somewhat chaotic state. There have been many investigations and publications since that was written, but it cannot be said that the uncertainty has been removed or that simple routine procedures have been devised on which there is general agreement.

Color measurement is of two different classes: (1) the determination of the amount of color or depth of color by direct visual comparison with standards of similar color, and (2) color analysis or the determination of the constituent primary colors which the materials transmit or reflect. These two classes of measurement are differentiated by some writers as colorimetry and tintometry, spectrophotometry being a special phase of the latter.

Practically all the color measurement in the sugar industry is done in solution, the only exception being comparison of the colors of soft sugars, and of white sugars in solid form. As will be shown later, grain size, surface conditions, and other factors have great influence on the results of these reflectance determinations.

PREPARATION OF THE SOLUTION

This is the most important as well as the most difficult part of the color measurement procedure. It is also the phase on which disagreement among investigators is most marked. The aim is the complete removal of all turbidity, so that the solution is optically void when viewed against a dark background with a strong beam of light projected slantwise through the solution (freedom from Tyndall effect).

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[1] Browne-Zerban, *Sugar Analysis*, New York, 1941, p. 574.

The complete removal of all turbidity is actually not possible by any of the ordinary means of filtration,[2] and even where relatively clear filtrates are obtained, more or less of the colloidal coloring matter is removed at the same time.

Many have thought that the work of Peters and Phelps[3] with asbestos filtration completely solves this difficulty, but Zerban and Sattler in the work cited above have shown that results with this method may vary widely with different operators, depending on whether the asbestos is packed tightly or loosely.

Lundén[4] recommends clarification by centrifugal action in a gravitational field of 10,000 to 12,000 or by filtration with sand. The elaborate study of color measurement in the sugar industry by Honig and Bogtstra[5] says, "before the juices are observed spectrophotometrically they should be filtered through sand until they no longer show the Tyndall effect. Spengler and Landt (*Z. deut. Vereins,* June, 1927) point out the difficulty of doing this, and they give a method for working with special filters. They say that the influence of a small amount of turbidity is much greater than is supposed." No further reference is made by Honig and Bogtstra to preparation of solutions except to note that the colors of head sugars were measured in solutions of 50° to 60° Brix filtered once through infusorial earth.

Kieselguhr (infusorial earth, Filter-Cel, Dicalite) is one of the most convenient and probably the most widely used clarificant to remove turbidity from solutions for color work. It is recognized as having selective action on coloring matter, as shown by Zerban, Lundén, Peters and Phelps, and others, but Zerban indicates that this is true also of asbestos. Further reference to the kieselguhr use will be made later.

571. Effect of Dilution. There is one point on which all investigators are in substantial agreement, namely, that dilution is to be avoided. Because of the protective action of the high-density sugar solution the colloids are stabilized and their coagulation after filtration is prevented. J. F. Brewster of the Bureau of Standards recommends[6] that densities be kept at 60° to 65° Brix instead of 55° as recommended by Peters and Phelps and that for sugars, massecuites, or other heavy materials requiring solution the boiling water be added slowly with constant stirring so as to prevent local dilution.

If the solution is too dark, the dilution of color is done with a colorless concentrated sucrose solution, made by dissolving granulated sugar in hot water to 60° Brix and decolorizing with 2 per cent washed decolorizing carbon. The sucrose solution is digested for fifteen minutes at 80° to 85° C., after which 10 per cent of prepared kieselguhr is added, and the whole is filtered under vacuum through filter paper on a Büchner funnel. Peters and

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[2] Zerban and Sattler, *Ind. Eng. Chem., Anal. Ed.,* May, 1936, p. 174.

[3] "Color in the Sugar Industry," *Bur. Standards Tech. Paper 338.*

[4] *Z. Ver. deut. Zuckerind,* October, 1927

[5] Translation *Facts About Sugar,* May 12, 19, 26, 1928.

[6] *Bur. Standards Tech. Paper 536.*

Phelps report that this solution will not keep indefinitely and that after a few days' storage it may be found to have taken on some color. Dilution of color may also be done with dry white tablet sugar and distilled water which avoids the change in standing and the decolorization of the sugar solution with carbon.[7]

572. Filtration through Asbestos. For accurate work Peters and Phelps[8] reported filtration through asbestos as the only method that gives satisfactory transparent solutions. Their method has been generally used by those doing spectrophotometric work, and a recent modification of it by Brewster will be given in detail below, but Dr. F. W. Zerban, who has done extensive work in this field, reports[9] that of two operators using the same batch of asbestos, preparing their Gooch crucibles side by side, one operator always obtained a lighter solution than the other because he unconsciously packed the asbestos tighter than the other man. This tighter packing gave more ultrafiltration, and the results in a typical set of experiments differed by as much as 15 per cent. Zerban's conclusion is that the asbestos filtration method as described by Peters is not sufficiently standardized to yield comparable results in different laboratories.

Brewster and Phelps[10] insist that this criticism does not hold if their directions are strictly followed. They say that "there is no decrease of color ($-\log t$) after repeated filtrations through fresh asbestos pads, and results are easily duplicated with independently prepared solutions of the same sugar." They state further "in our long experience, asbestos filtration is the only known procedure whereby sugars can be correctly prepared for spectrophotometric analysis."

573. Preparation of Asbestos (Brewster and Phelps Method).[11] The asbestos is treated as follows to remove fine particles and soluble matter that might affect the color determination. Twenty-five grams of asbestos is first boiled for thirty minutes with 250 ml. of 25 per cent NaOH solution (1.28 sp. gr.) in a Pyrex glass flask, nickel or iron dish. It is filtered hot by suction in a Büchner funnel without paper, washed repeatedly with hot water, then is transferred to a flask, and heated for thirty minutes on a water bath with 250 ml. of HCl sp. gr. 1.20 and 25 ml. HNO_3 sp. gr. 1.42. It is filtered by suction again and washed with hot distilled water until all acid is removed, then dried in oven at 110° C. and stored in a glass-stoppered container to prevent dust contamination.

574. Method of Filtration. Brewster and Phelps in the work cited recommended preparing and filtering the solution at high temperatures (80° to

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[7] See *Bur. Standards Circ. C 440*, p. 331, for details of method and calculation.

[8] *Loc. cit.*

[9] *Ind. Eng. Chem., Anal. Ed.*, May, 1936, p. 168.

[10] Communication to Committee on Colorimetry, Intern. Comm. Uniform Methods Sugar Analysis, July 31, 1936.

[11] *Ind. Eng. Chem., Anal. Ed.*, 1930, p. 373.

90°) but in "Polarimetry, Saccharimetry and the Sugars,"[12] the method is modified as follows so that heating is limited to 50° C.

> The calculated amount of sample to provide a 60-Brix solution is weighed into a tared flask on a rough balance and weights equivalent to the required water are added to the pan. A small amount of hot distilled water is added and the flask is placed in a water bath heated to 50° C. and shaken to promote rapid solution. Hot water is added to the flask a little at a time until the sample is dissolved. The flask is dried on the outside and returned to the balance pan where dilution to the required weight may be completed. Purified dry asbestos is added to the solution in amount depending upon the quantity and character of the suspended matter present. Usually 0.5 gm. will suffice, but when slimy material is present and the solution is refractory in filtration, 1 to 2 gms. should be used. The flask is loosely stoppered and shaken gently at first until the warm air is expelled. The stopper is then tightened and the flask is shaken vigorously to mix the contents thoroughly and to permit the asbestos fibers and the suspended matter to become entangled. The flask is returned to the bath and the solution is allowed to warm for a short time.
>
> Two filters are prepared by pouring the asbestos in water suspension in the Gooch crucible, sucking it down with the aspirator and then packing tightly by pressing and tapping with a flattened glass rod. The pad should be about 5 mm. thick when tightly packed. The first, or preliminary filter, immediately before filtration, is warmed by washing with a small amount of hot distilled water, which is aspirated from the asbestos pad as thoroughly as possible. The warm solution is then added and a few milliliters filtered to displace the water remaining in the pad when the suction is closed off and a clean dry receiver is substituted. When the filtration is resumed the remainder of the solution, or as much as the filter will hold, is added. The pad is to be kept covered with solution during the filtration, at the end of which the suction is stopped before the pad becomes uncovered. The receiver is detached and returned to the bath for further warming while the second filter is being rinsed with hot water and drained. The second filtration is performed exactly as the first, but no asbestos is added to the first filtrate. The main portion of the filtrate is collected in a clean receiver. This filtrate is cooled, and adhering condensed water is wiped from the neck of the bottle which is then closed with a clean, dry stopper and shaken to mix the contents thoroughly. The refractometric Brix of the optical filtrate is determined.

575. Filtration with Kieselguhr. Zerban and Sattler[13] in the study of filtering agents for preparation of sugar solutions for color and turbidity determinations found that with solutions at 60° Brix as above prepared so as to avoid dilution, there is little difference between kieselguhr (Johns-Manville Analytical Grade Celite) and asbestos as prepared by Brewster and Phelps, provided the latter is used in the form of a tightly packed pad. Both remove coloring matter selectively.[14] Zerban and Sattler recommend the use of the kieselguhr as giving reproducible results in the hands of different operators. The Celite is further purified by boiling 75 grams in 1000 ml. of 1 : 10 HCl,

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[12] *Bur. Standards Circ. C 440*, p. 327.

[13] *Ind. Eng. Chem., Anal. Ed.*, May, 1936, p. 168.

[14] Brewster and Phelps say that this is not true if their procedure is followed exactly. See p. 472.

filtering hot on a Büchner funnel, and washing thoroughly with hot water. Repeat the treatment three times, dry, and ignite in a muffle. In routine work 6 grams of the Celite is well shaken with 100 grams of the sugar solution, and the mixture is filtered through double filter paper S.S. No. 595 Blue Ribbon in a 7-cm. Büchner funnel with the filtering arrangement shown in Fig. 161. The stopcock is left open at the beginning of the filtration until about 25 ml. has passed into the flask. When the solution runs clear, close the stopcock and collect the clear filtrate in the tube above without breaking vacuum.

The Java Sugar Experiment Station recommends filtration with Hyflo Super-Cel purified by heating 1 kg. with 2 liters of concentrated HCl for three hours at 80° C., washing free of acid, and heating to 110° C. Two grams of the purified Hyflo is mixed with 200 ml. of the sugar solution filtered by suction through a glass Gooch crucible with filter paper S.S. No. 575, and the filtrate is passed five times through the same filter bed.

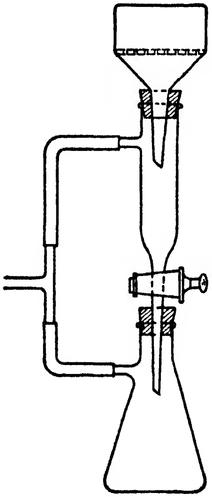
Fig. 161. Filtration Apparatus for Color Determinations.

576. Choice of Method of Preparation. In view of the divergence of opinion on asbestos filtration and the tediousness of the procedure, it would seem that the use of kieselguhr is preferable for routine work and possibly for all types of work. Pending the development of other simple methods of greater accuracy the method advocated by Zerban and Sattler is recommended. Certain precautions may be observed in all color work to insure greater comparative accuracy. (1) The same amount and kind of kieselguhr should be used in all tests. (2) Local dilution should be avoided in dissolving sugars or solid products to avoid colloid dispersion, and the Brix should be maintained at 60° or above at all times. (3) If the color is too dark to read, dilution should never be with water. A 60° Brix sucrose solution (colorless) or dry white sugar and distilled water should be used. (4) Long heating or high temperatures of solutions should be avoided. (5) Determinations should be made soon after solutions are prepared. (6) The reaction of the solutions should be regulated, or at least carefully noted.

577. Effect of pH on Color. The effect of a change of H-Ion concentration on the color of a sugar solution has been studied by many investigators. It has long been known that acid juices and sirups are lighter colored than the same juices at alkaline reactions, the coloring matter of the juice or sirup acting as a sort of indicator. The light absorption at different wavelengths also differs markedly as the pH is changed. Zerban[15] showed that in using a raw sugar solution which had been altered from 5.8 pH to 8.3 pH

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[15] *Intern. Sugar J.*, December, 1925, p. 446.

through small intervals by the addition of NaOH there was an appreciable increase in the – log t values (measure of the amount of color) with the increase of the pH, though within the range of pH of refinery work (6.6–7.9 pH) the increase is equivalent to a concentration of the same coloring matter, the quality not changing appreciably.

In all comparative color work the pH of the solutions compared should be accurately adjusted to the same value in order to avoid this effect of the change of pH.

Lundén[16] found the presence of several coloring matters in sugar products which react differently to changes in pH, and some information regarding the types of coloring matter present may be obtained by making measurements throughout a wide pH range.

578. Effect of Temperature on Color Readings. A point which is not stressed by most investigators but which may be readily seen by experiment is that many sugar solutions darken appreciably during cooling because of the absorption of oxygen. The effect may be hastened by agitation with air during the cooling period, but on reheating the increased color will disappear. This darkening is particularly noticeable in certain low-test char-filtered liquors. In making decolorization tests with boneblack or carbon, care should be taken to compare the solutions while hot or to protect them from contact with air while cooling.

APPARATUS FOR COLOR MEASUREMENT

A great many varieties and modifications of colorimetric apparatus have been developed many of which have been specially devised for sugar work or claim to be particularly suited for that purpose. The sugar technologist is confronted with a wide choice of instruments, but unfortunately none of these has been given such general acceptance as to be considered standardized. In view of the difficulties outlined above in the preparation of sugar solutions for color determination, it follows that the simpler types of apparatus which will give comparative or reproducible results are best suited for routine work. In general, colorimeters may be divided into two classes: (1) those in which the depth of color is compared with some standard (caramel, colored glass, etc.), and (2) those in which no standard is used and the amount of coloring matter is determined by measuring the light transmitted (or absorbed). In the following sections, colorimeters which have found the widest application in the sugar industry will be described.

579. Duboscq Colorimeters. The simplest method of comparing the intensity of color of two liquids is by adjusting the depth of the liquids in separate tubes until they appear to have the same color as viewed from above. The Duboscq type of colorimeter, modifications of which are made by many manufacturers of optical instruments, is designed to accomplish this adjustment and comparison. The one shown in Fig. 162 is a Klett-

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[16] *Ver. deut. Zuckerind.*, **76**, 780 (1926).

Duboscq instrument. Light falling on the reflecting plate or mirror is projected up through the clear glass bottom of the black glass tube containing the liquid to be examined. A black glass plunger, also with transparent polished glass bottom, is immersed in the liquid and transmits the light passing through the liquid, between the bottom of the tube and the bottom of the plunger, to the prism above and thence to one-half the field in the eyepiece. A duplicate tube on the other side of the instrument carries the liquid used as a standard, the light transmitted through it forming the other half of the field. Either tube may be raised or lowered by the milled heads, thereby changing the depth of liquid through which the light passes, until the two halves of the field appear the same color. By means of the small telescope attached to the rear of the eyepiece the scales showing the depth of liquid below the plunger in each tube may be read to fractions of a millimeter. In practice the standard is generally placed at some convenient fixed depth (say 20 mm.) and the test solution adjusted to match it. Then the amount of color in the test solution in terms of the amount of color in the standard is computed by dividing 20 (or whatever depth was chosen for the standard) by the depth of the test solution. A convenient adjunct to the Klett is the table in the base of the instrument by which this calculation is effected.

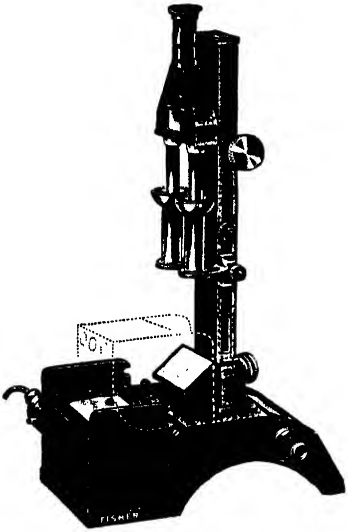

FIG. 162. Klett Colorimeter.

580. Color Standards for Duboscq Colorimeters. The Duboscq type of colorimeter is simple and convenient, and accurate for the purpose intended, that is, measurement of total color, so long as the standard and the test solution are of identical color tone. When differences in tint have to be dealt with, which occurs in dealing with many sugar liquors, there is considerable difficulty in matching the shade. The standards generally used in sugar work are caramel in glycerin, or ferric chloride solutions, and although these solutions approximate the color of washed sugar liquor or solutions of raw sugar, they differ markedly in tint from char-filtered liquors. A little ferric chloride added to the glycerin-caramel standards (suggested by T. B. Wayne) adds to the simulation of the color of the washed sugar liquor, but there are still differences in tint to be dealt with. These differences can be removed to some extent by inserting a light filter in the colorimeter between the solution and the eye. In the Klett this is easily accomplished by adding to the hollow plungers (which may be unscrewed) the following solution of metallic salts to a depth of 1 or 2 cm. (Obviously both plungers must contain the same depth of solution.) The solution which was devised by Wayne[17] consists of 250 ml. of 10 per cent nickel nitrate and 5.65 ml. of 10 per cent potassium bichromate (both solutions having been filtered through asbestos as described on p. 472) made to exactly 500 ml. with distilled water. It has an effective wavelength of 563 mμ and transmits only a narrow range of wavelengths between 540 and 570 mμ, and when used in proper thickness, the effect is to filter out the rays above and below this range and remove the differences of tint.

The standards used in the above method may be calibrated to absolute units (see Sec. 594) in a spectrophotometer, or an arbitrary standard may be used. The use of a standard that will give comparable results with other workers is an argument for the "standardized" standard. Kenneth Ritchie[18] gives a method of making a standard of ferric chloride solution which can be translated into Stammer units (see Sec. 582).

581. Brewster's Modification of the Duboscq Colorimeter. The apparatus consists of a Duboscq colorimeter and an incandescent light source with suitable spectral filters and glass photometric standards for the measurement of absorbency in sugar solutions. The absorbency of all except very pale solutions may be determined at 560 mμ by using a special yellow-green filter. Solutions of high-grade sugar products having little color are treated as a special case, and using a blue filter, measurements are made at 460 mμ. The results obtainable are satisfactory for technical purposes.

The instrument used by Brewster is the Bausch and Lomb Duboscq colorimeter with 100-mm. scales. He modified the instrument by adding a 60° prism in a suitable housing so as to give an inclined eyepiece for easy reading. The glass comparison standards consist of polished plates of carbon amber glass which is produced in various shades from dark amber to pale yellow (American Optical Company, Southbridge, Mass.). These glasses have similar transmission curves to the curves of caramel solutions and good color matches can be obtained with sugar solutions using the filters described below. Standard plates in three shades of amber glass are sufficient for the colorimetry of products ranging from the darkest that may be read up to and including off-color granulated sugar. The transmission of these plates should be close to 0.50, 0.70, and 0.80 respectively and may be calibrated in terms of transmission by the Bureau of Standards.

The glass color standard is placed on a shelf fastened to the vertical column of the colorimeter below the lowest position of the travel of the cup stages. The shelf has two openings, 25 mm. in diameter, centered underneath the cups. A slide permits transferring the standard plate from the light path of one cup to the other.

Brewster devised a filter with an effective wavelength of 560 mμ for use with ordinary tungsten lamps. It consists of one layer each of Wratten

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[17] *The Planter*, Oct. 17, 1925.

[18] *Ind. Eng. Chem.*, **19**, No. 11 (1927).

filters 21 and 61, one 6.5-mm. thickness of Corning didymium glass[19] with a colorless cover glass 1 to 2 mm. thick. The gelatin filters are cemented with Canada balsam between the two glasses, and the edges are bound with black tape. Gibson devised a filter of glass for 560 mμ which Brewster considers preferable to his.[20] It consists of Corning glass 351, 4.55 mm.; Corning didymium glass, 5.82 mm.; Jena VG-3 glass, 1.99 mm.; and Jena BG-18 glass, 1.94 mm. The filters are made of such size as to fit into the eyepiece tube of the colorimeter.

Method of Observation and Calculation. A portion of the properly prepared solution (see Sec. 576) is added to each colorimeter cup and with the appropriate filter in place at the eyepiece the zero setting is checked, and the readings are carried out by a transposition method as follows.

A standard plate is centered under one of the cups, the corresponding scale set to a definite blank reading, say 1.0 cm., and five readings taken in the usual manner. The plate is now centered under the opposite cup, the same blank scale reading set above it, and another series of five readings taken. The mean of the ten readings minus the blank setting expressed in centimeters gives the depth b of liquid for a transmittancy equal to the transmission of the standard plate.

The following example of the determination of − log t at 560 mμ for a solution of washed raw sugar is an illustration.

Refractometric Brix, 60.9, corresponding to true density, 20°, 1.29176.

$$c = \frac{60.9 \times 1.29176}{100} = 0.7867$$

Standard plate, $T = 0.579$ or $-\log T = 0.2373$.

Photometric measurements	*cm.*
Right scale, mean of 5 settings	3.332
Left scale, mean of 5 settings	3.028
Mean of 10 settings	3.180
Subtracting blank setting	1.000
	$b = 2.180$

Substituting in the Beer's law equation (see Sec. 590)

$$-\log t = \frac{0.2373}{2.18 \times 0.7867} = 0.1384$$

The calculation of results is simplified by using a slide rule with reciprocal scales.

582. Stammer Colorimeter. This is one of the oldest of the colorimeters and is still used extensively in Europe, although it is being rapidly replaced by color analysis apparatus. In effect it is similar to a Duboscq in which

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[19] *Facts About Sugar*, **28**, 228, 1933.

[20] *J. Research Natl. Bur. Standards*, **14**, 545 (1935).

the standard tube has been replaced by a brown-colored glass accurately standardized, called normal glass. A half normal glass is also provided. In the Stammer the plunger is moved up and down, instead of the tube, to vary the depth of the test solution.

The reference basis is that a solution 10 cm. high at 100° Brix equal in color to two normal glasses has a color of 1° Stammer. Stammer degrees are actually calculated by dividing 100 by the height in millimeters of test liquid which matches the color of two normal glasses and then multiplying this by $100/\text{Brix} \times$ specific gravity.

The Stammer offers even greater objections than the Duboscq on the score of tint differences between standard and solution. The normal glasses were made for beet juices and do not match cane sugar products well in tint. It has the advantage that, it gives results in units which are comparable with the work of other Stammer instruments. It has been found by several observers that the standard glasses supplied by different manufacturers differ widely, and these should always be tested by spectrophotometric analysis.

583. Ives Tint-Photometer. This instrument is rather widely used in the sugar industry. The earlier models were called Hess-Ives. It employs color screens each of which isolates part of the spectrum, but in the experience of the writer attempts to "analyze" the color of sugar solutions by the use of these screens do not result in useful figures. A 560 color screen, which actually transmits a rather broad band of yellow-green in the region of 560 mμ, is the best to use in routine sugar work.

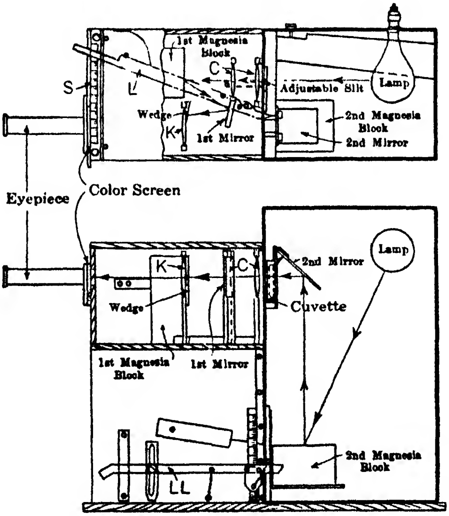

Fig. 163. Ives Tint-Photometer.

The optical arrangement as shown in Fig. 163 is such that a part of the light from the lamp is reflected from the second magnesia block to the second mirror, thence through the glass cell through one-half of the lens *K*, forming one-half of the field, while a second part of the light passes first through the adjustable slit, thence through condensing lenses to the first magnesia block where it is reflected to the first mirror, and thence through the other half of *K* to form the remaining half of the field.

The glass cell is first filled with the pure solvent (water) and put in position while the lever *L* is placed at 100, i.e., slit is wide open, on the scale *S*. With one of the color screens in place, the second magnesia block is adjusted by means of the lever *LL* until the field appears evenly colored as viewed from the eyepiece. The water cell is then replaced by a similar one containing the test solution. The field will now appear darker on one side because of the extinction of light by the color of the solution. This is compensated for by closing the slit with the lever *L* until the two halves of the field again match, when the reading is noted on the scale.

TABLE FOR TRANSLATING HESS-IVES TINT-PHOTOMETER SCALE READINGS TO MEADE-HARRIS UNITS OF COLOR

$y = K^x$, where y = scale reading; x = units of color; K = 99 (constant)

Scale Reading y	Units of Color x	Scale Reading y	Units of Color x	Scale Reading y	Units of Color x	Scale Reading y	Units of Color x	Scale Reading y	Units of Color x
100	0.0	80	22.2	60	50.8	40	91.1	20	160.0
99	1.0	79	23.4	59	52.5	39	93.6	19	165.0
98	2.0	78	24.7	58	54.2	38	96.2	18	170.5
97	3.0	77	26.0	57	55.9	37	98.9	17	176.0
96	4.0	76	27.3	56	57.7	36	101.6	16	182.0
95	5.1	75	28.6	55	59.4	35	104.4	15	189.0
94	6.1	74	29.9	54	61.3	34	107.3	14	196.0
93	7.2	73	31.2	53	63.1	33	110.3	13	203.0
92	8.3	72	32.6	52	65.0	32	113.3	12	211.0
91	9.4	71	34.0	51	67.0	31	116.5	11	219.0
90	10.5	70	35.5	50	68.9	30	119.7	10	229.0
89	11.6	69	37.0	49	70.9	29	123.1	9	239.5
88	12.7	68	38.4	48	73.0	28	126.6	8	251.0
87	13.8	67	39.9	47	75.1	27	130.2	7	264.0
86	15.0	66	41.3	46	77.2	26	134.0	6	280.0
85	16.2	65	42.8	45	79.4	25	137.8	5	298.0
84	17.4	64	44.4	44	81.6	24	141.9	4	320.0
83	18.6	63	46.0	43	83.9	23	146.2	3	349.0
82	19.8	62	47.6	42	86.2	22	150.6	2	389.0
81	21.0	61	49.2	41	88.7	21	155.0	1	458.0

In the early days of the Hess-Ives an investigation by the writer and Joseph B. Harris[21] showed that the percentage scale on this instrument was meaningless in itself but that the relationship between the scale readings and relative amounts of color was expressed algebraically as $y = K^x$, where y is any scale reading, K the reading for any unit of material, and x the number of units of material required to give the scale reading y; or $x = \log y/\log K$. To avoid the logarithmic calculation a table was devised as given on p. 480 by taking K as 99 and solving throughout for y from 100 to 0.

These units were devised solely for use with the Ives instrument and were never intended as color units in general colorimetry. They proved convenient for several years until the absolute units recommended by Peters and Phelps (to which the above units may be translated) came into vogue, and they are still of service in routine work.

584. Keuffel and Esser Color Analyzer. This is a direct-reading spectrophotometer of the highest optical precision suitable for all classes of research work and color investigations both with solids and liquids. The construction details may be seen in Fig. 164. It consists essentially of a source of light made up of two 400-watt lamps in the spherical housing, a rotating disk photometer, and a constant deviation spectrometer. Between the lamp housing and the disk of the photometer is seen the holder for liquid samples or transparent solids. Opaque samples are held at the rear of the lamp housing by the spring clips seen at the left of the figure. Full directions for the operation of the instrument are supplied by the makers.

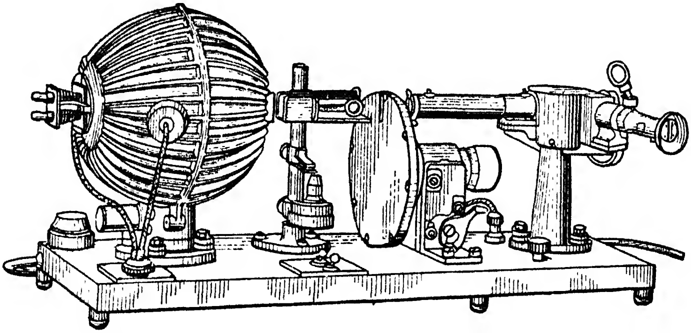

FIG. 164. Keuffel and Esser Color Analyzer.

Because of its complexity and cost the Keuffel and Esser analyzer is not suited for general routine work, but it has been widely used by investigators in the United States in research work on sugar products. Spectrophotometers of even more elaborate type which are used at the Bureau of Standards are the Bausch and Lomb, Königs-Martens, and Gaertner instruments.[22]

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[21] Meade and Harris, *Ind. Eng. Chem.*, 1920, p. 686.

[22] See "Saccharimetry, Polarimetry and the Sugars," *Bur. Standards Circ. C 440* (1942), pp. 304–310.

585. The Pulfrich Photometer. This instrument will serve for what the *Bureau of Standards Circular C 440* (p. 315) describes as "abridged spectrophotometry." The diagram (Fig. 165) shows the photometer which consists of two parallel telescopes arranged side by side and sharing a common eyepiece. Behind the eyepiece there is a device by means of which any one of seven different color filters (effective wavelengths from 430 mμ to 750 mμ) may be introduced in the path of the rays. The field of view is divided into two semicircular portions by means of a vertical line of separation so that one-half corresponds to one of the telescopes, the other half to the other telescope. The measuring device consists of two identical drumheads mounted in front of the openings of the telescopes, by means of which the brightness of the field of view may be varied by amounts which can be read in percentage transmission. The scale on the drumhead is so arranged as to afford an approximately equal degree of percentage accuracy when testing material with low or high absorption values. The instrument may be set vertically and (with certain accessories) used as a colorimeter of the Duboscq type, and in this position reflectance measurements may be made on solid substances. With an additional housing the instrument becomes a Tyndallometer and may be used as a nephelometer for the determination of turbidity.

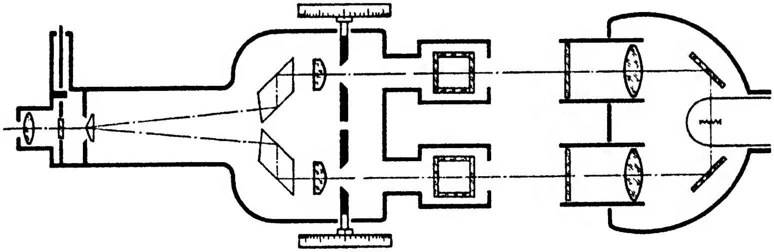

FIG. 165. Pulfrich Photometer.

586. Photoelectric Colorimeters. A large number of instruments have been devised for measuring extinction by colored solutions by means of photoelectric cells in one form or another. This does away with the human eye entirely. One of the earliest of these was described by Sandera [28] and is the apparatus schematically represented in the accompanying diagram (Fig. 166). The principle employed is measurement of the strengths of electric currents generated by light of varying intensities.

In this apparatus light from the source, *O*, passes through a suitable lens, *e*, and filter, *f*, a cuvette, *a*, containing the pure solvent and impinges on the coating, *K*, of the metallic potassium on the interior of the photo-electric cell. The photo-electric current is amplified by the electric tube (grid, *M'*, and anode, *E*); the amplified current, which is proportional to the light falling on the photo-electric cell, is measured on the millivoltmeter, *MV*. The cuvette, *b*, containing a solution of the product, is then put in place of *a* and a new

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[28] *Z. Zuckerind. Čechoslovak. Rep.*, **52**, 261 (1928).

measurement made; the difference in the two measurements gives the measure of the color of the sample. The accuracy of the readings is about 1 per cent. The instrument is made by J. & J. Frič, Prague.

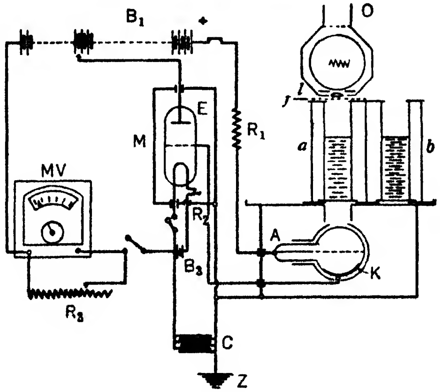

Fig. 166. Colorimeter Using Photoelectric Cell.

587. Holven-Gillett Photocolorimeter.[24] This instrument[25] was developed at the California-Hawaiian Sugar Refinery especially for sugar products. It utilizes two photogenerative cells in a balanced photoelectric circuit of the series-opposing type. Two beams of light from a single source of illumination are projected, by means of mirrors, through movable lenses, color filters (Corning 401 sextant green) and 3-cm. absorption vessels onto the respective photocells. (See Fig. 167.) Each photocell is shunted by a fixed

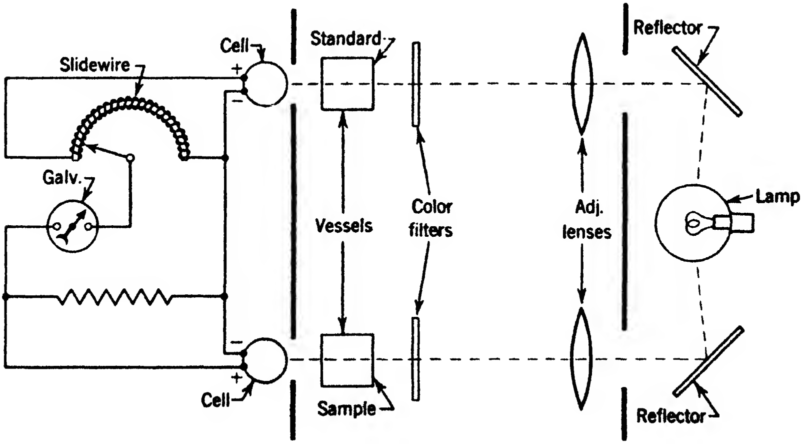

Fig. 167. Holven-Gillett Photocolorimeter.

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[24] U.S. patent 2,152,645.

[25] *Facts About Sugar,* May, 1935.

resistance, one of these being a uniformly wound slidewire type of resistance. The photocell circuits are connected in opposition through a lamp and scale galvanometer which indicates a condition of balance or unbalance in the instrument. The use of this arrangement eliminates errors due to light variations, voltage fluctuations, resistance changes, and other such factors. Direct reading in percentage light absorption permits calibration in $-\log \mathbf{t}$ units. Vessels of various sizes are used to extend the color range of the instrument.

In operation, both vessels are filled with a zero light absorption standard such as distilled water, the variable contact on the slidewire is set at zero, i.e., with its full resistance in circuit, and one or the other of the lenses is adjusted until an initial balance is obtained. The sample vessel is then filled with the sample, and the slidewire is rotated until the galvanometer again balances. The color is then read directly on the slidewire which is calibrated in per cent light absorption and either degrees Stammer color or $-\log \mathbf{t}$.

By use of additional color filters either trichromatic or complete spectral color analysis of a sample may be made. For trichromatic color analysis, Corning 243 signal red and Corning 554 lantern blue color filters are used in addition to the sextant green filters.

Turbidity may be measured by first balancing the instrument with the filtered sample in both vessels and then placing the unfiltered material in the sample vessel and rebalancing.

588. Color of Refined Sugars. Color by Reflectance. The color of refined sugars is a specialized subject and is treated in Chapter 30. In general, the solutions are so nearly free of color that determinations must be made in much deeper layers than can be employed with most of the colorimeters here described.

The color of refined sugars and soft sugars by reflectance has been a subject of considerable study, and such reflectance measurements will also be discussed in Chapter 30. Grain size and the preparation of the sample play a large part in the accuracy of such tests. Since the actual color present is generally of greater importance than the appearance of the sugars (which the reflectance measures) it would seem that the color of the solutions is of more value than the apparent color of the solid sugars.

COLOR NOMENCLATURE FOR SUGAR SOLUTIONS [26]

589. Introductory. It is only within the past two decades that absolute measurements based on optical laws have been in use in the sugar industry. Peters and Phelps were the first to carry out studies along these lines, and most investigators since then have made use of the absolute measurements. These measurements have been recommended by the International Commission for Uniform Methods (London, 1936) for factory and commercial control.

590. Lambert-Beer's Law. This is the fundamental law (or combination of two laws) of color measurement which states that the amount of light

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[26] See Peters and Phelps, *Bur. Standards Tech. Paper 338* (1927).

transmitted by a colored solution is inversely proportional to the power of the units of thickness and the power of the units of concentration of the solution. For example, if 1-cm. thickness of a solution transmits 80 per cent of the incident white light, 2 cm. of the same solution will transmit 80 per cent of 80 per cent, or 64 per cent, of the light. Similarly, if 1 cm. of a solution of 10 per cent concentration transmits 80 per cent of the incident light, 1 cm. of the same material in 20 per cent concentration will transmit 64 per cent of the light. If both concentration and thickness were doubled in the above example, the transmission would be $0.8^4 = 0.4096$ or about 41 per cent of the incident light. The mathematical demonstration of the law which follows uses symbols adopted by the Bureau of Standards following the recommendation of the Optical Society of America and these symbols and nomenclature are now generally used in sugar colorimetry.

T = transmittancy, or the fraction of the incident light which is transmitted through a vessel with parallel polished glass ends containing the colored solution, after correcting for reflection at the surfaces and for absorption of light, if any, by the pure solvent. The solvent for sugar solutions is pure water which has an absorption which is negligible.
t = specific transmissivity = transmittancy reduced to unit conditions of thickness and concentration.
b = thickness in centimeters of the absorbing solution.
c = concentration in grams original colored dry substance (sucrose + nonsugars + coloring matter) per 1 ml. of solution.

Lambert's law states that the amount of light transmitted when the thickness of the absorbing medium is varied (concentration remaining the same) is inversely proportional to the power of the units of thickness or

$$t = \sqrt[b]{T}$$

Similarly Beer's law is that the light transmitted varies inversely as the power of the concentration (thickness remaining the same) or

$$t = \sqrt[c]{T}$$

Combining these, the Lambert-Beer's law becomes

$$t = \sqrt[cb]{T} \quad \text{or} \quad -\log t = \frac{1}{cb}(-\log T)$$

591. Specific Absorptive Index. Minus log t. It follows from the above [26a] that $-\log t$ is a measure of the coloring power, i.e., intensity of absorption, of the unknown amount of coloring materials associated with 1 gram of saccharine dry substance.

$$-\log t = \text{specific absorptive index.}$$

[26a] For the complex mathematical demonstration see Peters and Phelps *loc. cit.*

Peters and Phelps found that for the ordinary coloring matter of sugar products the solutions appear brightest to the eye at the wavelength λ of 560 mμ or, expressed in spectrophotometric terms,

$\lambda = 560$ mμ is the wavelength of the optical center of gravity of the luminosity curve for unit quantity of coloring matter of sugar products.

It is for this reason that the wavelength of 560 mμ was chosen as a standard by the Bureau of Standards. The Europeans use 590 mμ (Lundén) and 610 mμ (Spengler and Landt), whereas the Java Experiment Station uses 500 mμ. The International Commission for Uniform Methods (London, 1936) recommends measurement of $-\log \mathbf{t}$ at 560 mμ.

592. *Q* Ratio.

Q = Absorption ratio = the specific absorptive index at any one wavelength compared with the specific absorption index at the standard wavelength of 560 mμ $= \dfrac{-\log \mathbf{t}_\lambda}{-\log \mathbf{t}_{\lambda 560}}$.

Q is a measure of the quality of absorption of a solution; it is the relationship between unit absorption at 560 mμ and absorption at other wavelengths throughout the spectrum. A series of Q ratios taken at definite intervals of wavelengths and plotted as a curve will show the color trend of the solution being studied, whereas comparisons of the Q ratio curves of two solutions, e.g., before and after char filtration, will show the differences in the kind of color in the two solutions studied.

593. Determination of $-\log \mathbf{t}$ Value in Practice.[27] After a suitably transparent solution has been prepared with a correct dry substance concentration and a suitable color concentration, a parallel-sided cell of suitable thickness is filled and placed in one beam of a photometer. A similar cell filled with distilled water or with a colorless sucrose solution is placed in the comparison beam in order to compensate for losses by reflection at the cell surfaces and eliminate corrections for this effect. A spectrophotometer, a simplified spectrophotometer, or other color-measuring device which is capable of measuring transmittancy for monochromatic light of wavelength 560 mμ, or for a narrow band of the spectrum whose effective wavelength is 560 mμ, may be utilized. If a mercury vapor lamp is used as a light source, measurements of $\mathbf{T}$, or $-\log \mathbf{T}$ are made for the green line, 546 mμ, and the yellow lines, 578 mμ. The reading for 560 is then obtained by deducting 48 per cent of the difference between $-\log \mathbf{t}$ at λ 546 from $-\log \mathbf{t}$ at λ 578; the result is $-\log \mathbf{t}$ at λ 560.

The transmittancy measurement $\mathbf{T}$ determined for the concentration, c, and thickness, b, is reduced to unit basis, as regards concentration and thickness, by means of the equation

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[27] Peters and Phelps, *loc. cit.*, p. 283.

$$t = \sqrt[cb]{T}$$

or

$$-\log t = \frac{-\log T}{cb}$$

which expresses Lambert-Beer's law. It is to be noted that the photometer scale may be graduated in terms of **T** (transmittance), or directly in $-\log$ **T**, the latter direct reading eliminating one step in the calculation. The thickness, *b*, for any cell is a constant, and *c* is readily calculated, utilizing for this purpose the refractometric and density measurements.

Whether or not the colored solution has been diluted, the concentration, *c*, is always expressed as grams of original colored dry substance per 1 ml. of the final solution upon which the transmittancy measurement is made.

594. Absolute Color Units. Peters and Phelps also give a calculation for color units *n*.

$$n = \frac{-\log t_{560}}{0.00485}$$

These color units have not come into use as the $-\log$ **t** values themselves at 560 mμ are directly proportional to the units of coloring matter.

There are two obstacles to the use of $-\log$ **t** values in routine and commercial procedure: first, $-\log$ **t** is usually a decimal fraction which makes its use cumbersome for comparative purposes; second, the concept of a minus logarithmic value is difficult for the layman to grasp. The color units suggested by Peters and Phelps would obviate both the objections but the $-\log$ **t** value has become too much a part of the literature to change now to the Peters-Phelps units. To overcome the decimals the writer has suggested that $-\log$ **t** be multiplied by 100, and this has been adopted independently by Fort and McKaig.[28] The $-\log$ **t** value × 100 should have a convenient name to overcome the second objection and C. A. Fort[29] has suggested Lambert-Beer units or L-B units as an appropriate designation. If these suggestions are adopted

$$\text{L-B units} = -\log t \times 100$$

TURBIDITY

595. Definition. To the average sugar man turbidity means cloudiness which will not settle out of a solution on standing as opposed to suspended matter or insoluble matter which will settle out if the solution is allowed to stand a reasonable time. It is recognized that this definition is not scientifically accurate and that all suspended matter of whatever particle size may be considered as turbidity. Determinations are generally made on solutions

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[28] *U.S.D.A. Tech. Bull. 688* (1939).

[29] Personal communication.

that have been allowed to settle for a reasonable period, e.g., thirty minutes, or the coarser suspended material may be screened out by suitable means such as 200-mesh silk bolting cloth.

596. Turbidity Determination. The quantitative determination of turbidity in sugar products is a much more complicated (and less standardized) procedure than the determination of color. Exact turbidity measurements are fortunately not so important for routine control work as color determinations although the presence of turbidity is of great importance in the clarification of cane juices and in refinery press-filtered and char-filtered liquors. Turbid juices are known to produce raw sugars of poor filterability and refining quality. Cloudy liquors in the refinery will have a much greater adverse effect on the quality of refined sugars than will clear liquors of a deeper color tone.

When a beam of light is passed through a turbid solution, the very small particles cause scattering, and the larger particles reflect and refract the light. Part of the light is transmitted and part absorbed so that turbidity measurements may be based on either the amount of light transmitted or on the amount scattered and reflected (the Tyndall effect or Tyndall beam).

It is not within the scope of this book to detail the research methods which have been devised for exact turbidity measurements. The student is referred to the work of Zerban and his colleagues [30] and to standard textbooks on the subject [31] for a discussion of the theory and practice. "Polarimetry, Saccharimetry and the Sugars," [32] gives a concise treatment of the methods and apparatus used by different investigators. Certain simplified procedures, recognized as limited in value and application, are given below.

Suitable turbidity standards for comparison are not readily prepared for sugar work because of the complex nature of the colloid suspension. The color in sugar products offers an additional difficulty. In general, routine turbidity tests are qualitative or give comparative figures only.

597. Horne-Rice Turbidiscope.[33] This is readily made in the plant shop. It is of value for examining refinery liquors qualitatively for turbidity and is described as follows:

> The source of light is a good, nitrogen-filled, concentrated filament, incandescent lamp bulb, hung in a tin cylinder fitting quite closely around it and extending a little above the top of the lamp and about 1 cm. below the filament. About 1 cm. below this cylinder a second tin cylinder is attached by three soldered wires, and both are painted black on the outside and inside to prevent dispersion and reflection. Around each cylinder is a metal annulus perforated with holes to accommodate test tubes. At the bottom is soldered a broader annulus of tin to serve as a support for the test tubes. It can also serve as a base for the apparatus, but it is better to support it by the lamp

[30] *Ind. Eng. Chem., Anal. Ed.,* **3,** 326 (1931); **6,** 178 (1934); **7,** 157 (1935).

[31] Yoe and Klienman, *Photometric Chemical Analysis,* Vol. II, *Nephelometry,* 1929.

[32] *Bur. Standards Circ. C 440,* pp. 341–350.

[33] *Ind. Eng. Chem.,* **16,** No. 6 (1924).

socket, so that it can be turned easily and will be air-cooled. When the solution to be examined is put into a tube and set in the rack, there becomes apparent Tyndall's phenomenon of the reflection of light by the minute suspended particles, all viewed against a black background.

598. Kopke Turbidimeter. This simple device has found rather wide acceptance, particularly in Hawaii, where its use is prescribed for routine determinations on clarified juice.[34] Its accuracy has been questioned by King[35] who found that the results were influenced by many factors, including density, color, particle size, and the personal equation. It consists of a white porcelain plate, about 30 mm. in diameter, with crossed lines forming small squares on its upper surface. A hollow glass tube is attached vertically to one edge of the plate. This tube is about 15 cm. long and is graduated in tenths from 0 to 12 cm., the zero being on a level with the top of the plate.

Hold the turbidimeter in a vertical position with the plate touching the side of the container nearest the source of light. Filtrate jars are suitable containers. The sides must be clean to avoid cutting down light. The end point is when the lines on the plate are at the point of disappearance. At this point close the tube with the finger, remove from solution, and read the scale. At least three readings should be made on a sample.

Artificial illumination is preferable. Arrange a 75-watt light with an ordinary conical 10-in. metal shade so that the lower edge of the shade is 10 in. above the table. Use the so-called "daylight" lamps made of blue glass. The position of the samples should be as nearly under the edge of the shade as will permit making the observation from directly above. The polariscope hood is a convenient place for making these determinations.

599. The Colonial Sugar Refining Company Turbidimeter.[36] This was devised by the Colonial Sugar Refining Company of Australia and is based on the extinction of light by the turbid liquid. It is simple to operate and requires no reference standard.

A 1-in. hole is provided concentrically in the top of the housing (Fig. 168) and is covered by two pieces of glass, the lower of which has a ground surface. Between these two glasses is placed a grid consisting of parallel lines of India ink ruled on white paper. The lines are 1/16 in. thick spaced 1/16 in.

A glass cylinder, e.g., Nessler tube, with graduations from the bottom of 1/10 in., is enclosed in a tightly fitting cardboard sheath (painted black internally) and is mounted directly over the grid. A diaphragm, the aperture of which is smaller than the internal diameter of the tube, is fitted over the grid to prevent light passing up the walls of the tube. To control the amount of light reaching the grid from the lamp, two adjustable diaphragms are situated in the lamp housing between the lamp and the grid.

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[34] *Chemical Control for Cane Sugar Factories* (Hawaii), 1931, p. 52.

[35] *Louisiana Planter,* **82,** 211 (1929).

[36] Laboratory Manual for Queensland Sugar Mills, second edition, 1939, p. 32.

For use with clarified juice, the most satisfactory degree of illumination is obtained when the grid is just obscured by a 2-in. depth of a 1 per cent iodine solution placed in the glass tube and viewed from the top. The iodine solution is first poured into the tube to a depth of 2 in., and the relative positions of the diaphragms are adjusted till the grid is just no longer visible. Care must be taken that the distance of the observer's eye from the top of the tube does not vary, and extraneous light should be eliminated as completely as possible.

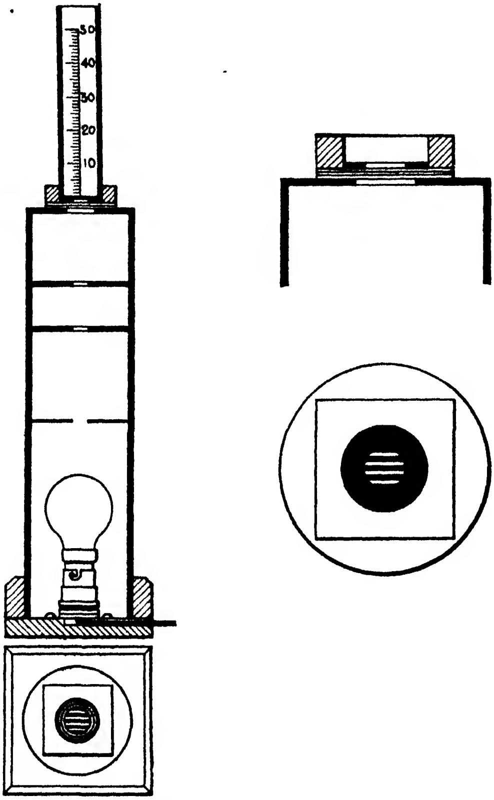

Fig. 168. Colonial Sugar Refining Company Turbidimeter.

Two measurements are necessary. The sample of clarified juice is allowed to stand for thirty minutes to permit suspended matter to settle, and the supernatant liquid is then decanted without disturbing the sediment. This decanted juice is divided into two portions, one being examined directly and the other after filtration, using Filter-Cel and a Büchner filter. The filtered portion should be brilliantly clear; if turbid, it should be refiltered or discarded.

The depths of each solution required to obscure the grid in the standardized instrument are now determined, and the turbidity is calculated as follows:

$$\text{Turbidity} = \frac{10}{a} - \frac{10}{b} = \frac{10(b-a)}{ab}$$

where a = depth of unfiltered juice in tenths of an inch which just obscures the grid;

b = depth of filtered juice in tenths of an inch which just obscures the grid.

As a criterion of good work an average value of 0.3 is specified, inferior work giving higher values.

600. Continuous Photoelectric Measurement of Turbidity. Gillett and Holven[87] describe a method of measuring the turbidity of sugar liquors continuously by a photometric clarity meter actuated by the increasing and decreasing intensity of the Tyndall cone with increased and decreased turbidity. The apparatus was devised primarily for use with filtrates from pressure filters in refinery work (see p. 300), but could undoubtedly be modified for use with other sugar products. A laboratory photoelectric turbidimeter based on similar principles is also described.

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[87] *Ind. Eng. Chem.*, **28**, No. 4 (1936).

CHAPTER 25

SAMPLING AND AVERAGING

601. General Remarks on Sampling and Averaging. One of the most difficult, and often unsatisfactory, problems for the cane sugar chemist is that of securing representative samples of the juices and various products at different stages of the manufacture. If a sample does not strictly represent the average composition of the material, the analytical work will usually be of little or no value. "The analysis is no better than the sample." This point cannot be too strongly emphasized, and the chemist in charge should study and check his sampling methods with the same care that he checks his analytical procedures.

In order that the samples may be representative they must be drawn continuously in proportion to the quantity of the material. Or they must be secured at intervals, drawing a definite quantity in each sample, from a measured or weighed quantity of the material, the size of the sample always bearing the same relation to the amount of material sampled. This second method is the one usually practiced and is termed sampling by aliquot parts.

The importance of a proper method of sampling is illustrated by the following example.

Given four lots of sirup *A*, *B*, *C*, and *D* from which an average sample is to be drawn. Let $A = 1000$, $B = 800$, $C = 500$, and $D = 200$, and let each lot differ from the others in analysis. Manifestly a mixture of equal parts of sirup from these lots would not be a true average sample, but a mixture of 10 parts of *A*, 8 of *B*, 5 of *C*, and 2 parts of *D* would represent the average composition of the sirup.

Of equal importance to accurate sampling is the proper preservation of samples. Sugar house products, particularly juices and other thin solutions, are subject to rapid deterioration in the tropics. The subject will be discussed at length later, but it is emphasized here as a general principle. Cleanliness of sampling devices, containers, and all utensils which are used for sampling is also essential, and these should be sterilized before using.

In averaging the analyses of the various materials in a cane factory, it is advisable to use the weights rather than the volume. Thus the weights of the juice, the sucrose (pol), and the apparent solids (Brix) should be recorded daily and at the end of a "run" or period the sum of the daily weights of sucrose (pol) divided by the weight of the juice and the quotient multiplied by 100, will give the mean per cent of sucrose (pol) in the juice and so on. Similarly the analyses of the sugars, and, so far as possible, those of the other products should be averaged.

602. Sampling the Cane. It is practically impossible to secure a moderate-sized sample of sugar cane that will be even fairly representative of that of a field. The best the chemist may hope to accomplish, under favorable conditions, is to obtain a sample that will in a very general way indicate the condition of the cane. The difficulty in sampling is due to the great variations in the analysis of canes from the same stool and also from various parts of even a small field. Frequently in a large factory, receiving cane from many fields, the daily average analyses will differ but little from day to day whereas single analyses may vary widely from the average.

In sampling cut cane a few stalks should be selected from every second or third row, crossing the field one or more times, according to its size, in sampling. The large sample, after mixing the canes, should be reduced by subsampling to one of convenient size for the laboratory. This method is frequently impracticable, since the carts often follow close behind the cutters.

It is even more difficult to sample standing cane, since a tropical field is almost impenetrable. In this case a few entire stools of cane should be secured from various parts of the fields, but not from near ditches or headlands, and these canes should be reduced to a convenient number, after mixing them thoroughly.

Where practicable the best method of sampling the whole cane is to await its arrival at the factory, then grind several cart- or car-loads apart from other cane, and analyze the juice.

The Hawaiian method for sampling cane is described as follows: [1]

> Cane is sampled for determination of fiber only, no practicable method having been developed for sampling and sub-sampling cane with a sufficient degree of accuracy to allow the determination of sucrose per cent cane by direct analysis. Several samples of cane for fiber determination should be taken daily, the number necessary depending on how much the cane varies in fiber content. The sticks comprising the sample should be taken at random, care being taken to avoid selection. Sample by fields if known, taking several sticks either from the cars or the cane carrier. Nine sticks make a convenient sample. In lieu of this, at regular intervals take 3 or 4 sticks at a time from the carrier, place in a cool, shaded place and allow the sample to accumulate until the end of the sampling period. Sub-sample down to a convenient number of sticks.

The *Laboratory Manual for Queensland Sugar Mills* (second edition, 1939) gives the following method for sampling cane as quoted from the Cane Prices Regulations:

> Sticks shall be taken from sample trucks in amounts approximating the same weight from each such truck. It is imperative that sufficient sticks be selected to give a truly representative sample of the total cane from which it is drawn.
>
> Sticks are arranged in order of descending length on the ground, with tops all one way. Each stick is then cut into three (equal lengths) without being displaced from position. The group is then subsampled by taking the first, fourth, seventh, etc., from the top sections; the second, fifth, eighth, etc., from

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[1] *Chemical Control for Cane Sugar Factories* (Hawaii), 1931, p. 27.

the middle sections; and the third, sixth, ninth, etc., from the butt sections. These bundles are kept separate. Alternate top and bottom half of each section is fibrated.

The fibrated material is thoroughly and quickly mixed and a portion weighed out for analysis. Every precaution must be taken to keep the edges of the fibrator in a sharp condition and to see that the necessary speed is maintained to ensure that the cane treated is finely and satisfactorily shredded.

Kerr and Cassidy[2] discussed the methods of sampling cane and the probable error for different methods, different cane varieties, and the number of stalks of cane sampled. Their results are of interest as emphasizing the difficulties involved in accurate cane sampling. Davies[3] studied the best method of subsampling the bagasse obtained from cane sampled by hand milling.

603. Sampling Bagasse. To a certain extent the bagasse presents the same difficulties as the whole cane. The bagasse, however, is more or less well mixed in its passage through the mills and irregularities may be overcome by sampling across the conveyor.

Samples of bagasse should include all that on the entire width of a section of the bagasse carrier, and it should be transported to the laboratory in clean galvanized iron cans, tightly covered. A piece of sheet metal the width of the carrier and bent into a V-shape longitudinally makes a convenient sampler. It should be remembered that loss of moisture in the sample introduces a very sensible error in the estimation of the moisture content. The sampling, subsampling, comminuting, and handling of bagasse samples should be done as rapidly as possible with a minimum of exposure to the air. On reaching the laboratory the sample should be quickly and thoroughly mixed and subsampled. The small sample may be analyzed immediately or stored six hours in a closed box in the presence of chloroform and ammonia. A sponge should be saturated with the preservatives in the proportion of one part chloroform to six parts of strong ammonia and be attached to the inside of the box cover.[4]

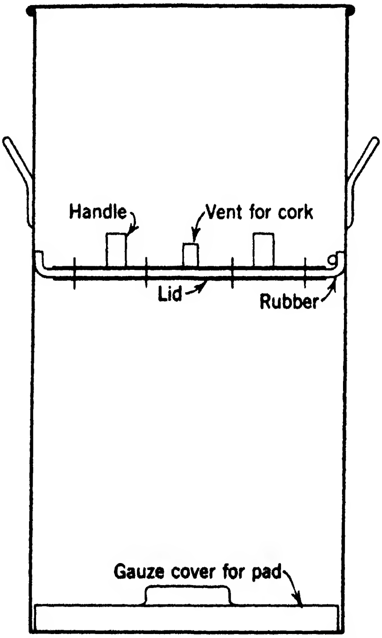

Fig. 169. Australian Bagasse Sample Can.

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[2] *Intern. Sugar J.*, May, 1938, p. 180.

[3] *Intern. Sugar J.*, January, 1932, p. 42.

[4] Spencer, *Ind. Eng. Chem.*, **12**, 1197 (1920).

A mixture of formalin and ammonia has also been recommended. A convenient can for compositing bagasse samples is in use in Queensland and is shown in Fig. 169. It is described by Behne[5] as follows:

> The actual size is immaterial but the special feature is the "piston type" lid. This consists of two discs of heavy-gauge galvanized iron about two inches smaller in diameter than the internal diameter of the container, with an overlapping piece of rubber insertion riveted between them. Two handles are placed conveniently on the top, and a hole is provided at the centre to take a small rubber stopper. This hole acts as a release valve or vent, to allow the escape of air as the lid is pressed into the container. The lid is pressed down till it just touches the top of the bagasse, when the stopper is inserted and the whole is then air tight. In this way there is no free space above the bagasse, and condensation on the walls of the tin is reduced to a minimum. The preserving pad, which consists of a piece of cotton wool impregnated with the required amount of ammonia and chloroform, is placed under a gauze screen at the bottom of the container. . . .
>
> As the effect of the preservative depends on its penetration to all parts of the sample, the bagasse must not be packed too tightly; otherwise the upper portion of the sample remains untouched by the fumes even though the pad is still saturated.

Behne in the article cited reviewed the sampling and preservation of bagasse and found that the methods of sampling and preservation in general use give accurate results if care is exercised in mixing the samples.

Automatic bagasse samplers have been devised and are in use to some extent in Java, but they appear to have one or more objections not present in the hand method described above. Some do not sample clear across the carrier, whereas others do not insure a sample the full depth of the blanket, both of which are requisites to accurate bagasse sampling.

SAMPLING THE JUICE

For the International Society of Sugar Cane Technologists method of mill control (see Chapter 35) the juice samples required are mixed juice and last-expressed juice, whereas for the older methods of control (involving the use of a milling factor, Sec. 613) the first-expressed juice (generally the crusher juice) is also sampled.

604. Sampling Devices for Crusher Juices. The juice should be sampled automatically and in proportion to that extracted. The milling is usually very uniform under good conditions of equipment and operation, hence samplers may be operated by some part of the mill mechanism, preferably a roll shaft. Certain types of samplers may be driven by a reciprocating or other part of the juice pump.

The Calumet is an efficient type of sampler (Fig. 170) devised at the Calumet Plantation, Louisiana, and shown in the figure as designed by Spencer to draw juice from the crusher canal. A modified type is necessary for sampling juice under pressure in pipelines.

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[5] *Intern. Sugar J.*, October, 1937, p. 392.

The construction of the Calumet sampler is shown in the sectional scale drawing, with the device bolted to the crusher canal. A hole is bored through the plunger, near the end, to receive the sample. A lip or projection closes the lower side of the hole while filling. The plunger clears the trash from this projection at each stroke it makes. When the plunger is withdrawn it discharges the sample through the lower end of the hole into the gutter. A gland is provided to prevent leakage of juice.

The Calumet sampler for drawing juice from a pipeline is of somewhat different construction. The plunger should be about 1 in. in diameter and have a stroke of about 3 in. A juice cavity about ½ in. in diameter is drilled longitudinally into the end of the plunger and is fitted with a screw for adjusting the size of the cavity and consequently of the sample. A 3/16-in. hole is drilled at right angles to the cavity and through it. Corresponding holes are drilled through the pump barrel for the inlet of air and the discharge of the sample from the plunger. Two rings of packing, controlled by a follower and packing ring, are placed around the plunger, one at the outer end of the barrel and the other between the juice inlet and outlet. There should be an oval opening in the packing ring where it passes the juice outlet, to admit of adjustment. The barrel of the sampler is screwed into the pipe from which the juice is to be drawn and is clamped into place with a locknut. This sampler may be operated from a reciprocating part of the juice pump, reducing the speed, if need be, by a mechanism such as used in pumping oil to a bearing. The vertical or outlet hole in the plunger is made small to reduce the cutting of the packing.

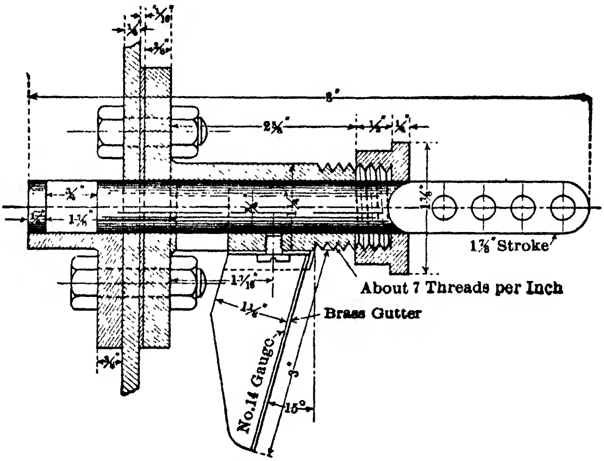

FIG. 170. Calumet Juice Sampler.

605. Mercedita Sampler. A sampling device known as the Mercedita sampler specially designed for taking samples of crusher juices is shown in

Fig. 171 and consists essentially of a link motion operated from a crusher roll which actuates a swinging arm *A* carrying a small sample cup *C*. At each rotation of the roll the arm and cup are swung out into the falling juice (position $A^2 - C^2$) and then withdrawn, the cup being turned upside down

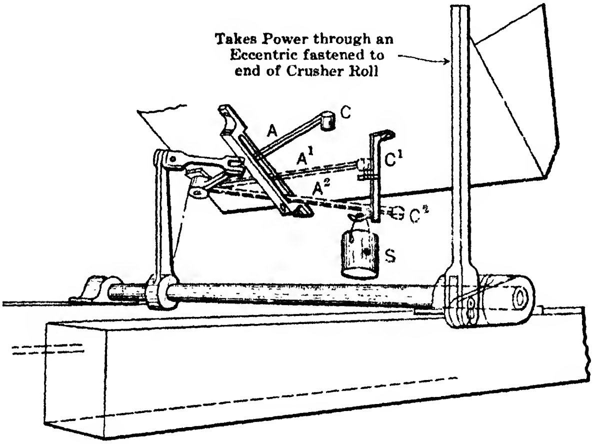

FIG. 171. Mercedita Juice Sampler.

($A^1 - C^1$) by a tilting device as it comes over the sample containers. This arrangement has been found superior to any other sampler in simplicity and in freedom from clogging and fouling, but it has the objection that it does not sample across the whole flow of juice.

606. Other Devices. A device that is sometimes advocated is a heavy wire leading from the stream of juice to the sample bottle. With a short wire the error from evaporation is slight with cold juices, but with hot juices the evaporation is sufficient to affect the figures appreciably. The method should not be used where other means of sampling are available.

An undershot water-wheel, just dipping into the juice and driven by the current, may be used to sample from a canal. The axle of the wheel should be hollow and a few of the spokes should be tubular and communicate through the axle with the sample jar. The hollow spokes should terminate in ladles which serve to take up a little of the juice and deliver it through the spokes and axle to the jar. The juice in the canal should be thoroughly mixed by baffles before reaching the sampler.

The difficulty of keeping small diameter pipes clean is an argument against arrangements such as these.

A can with conical cover, in which is located a small hole covered with gauze, is sometimes used. This is placed where the juice is falling, and the stream flowing over the gauze keeps it clear of fiber, while a small amount of juice enters the can.

Samples may be drawn from the discharge pipe of a direct-acting pump by means of a spring-controlled relief valve. The valve should be adjusted to open at the moment of highest pressure at each pump stroke.

607. Advantages and Disadvantages of Different Devices. The difficulty with most samplers is their liability to clog, tendency to foul, uncertainty as to their sampling in proportion to the quantity of liquid passing them, and the probability of not drawing an average sample under certain conditions, e.g., when drawing from a canal into which the juices from several mills flow. The Calumet sampler draws a correct sample when it is connected with the discharge line from a pump and is operated by the latter. When this sampler is connected with the canal leading from the crusher, its sample is from only a part of the juice, but it is usually a good approximation of a representative sample.

Particles of juice-soaked bagasse have been found to hang up on the projecting parts of the Calumet with consequent souring, and for this reason the Mercedita sampler is preferred, although it does not sample across the full stream of juice. The value of a mechanical sampler is in direct ratio with the ability to keep it clean. Hand sampling, where the honesty and dependability of the sampler can be checked regularly, is second only to the best mechanical devices, and drip samplers are not generally to be recommended.

608. Sampling the Mixed Juice. The accuracy of the factory control is based on the analysis of the mixed juices so the samples of this material should be taken with extreme care. Where weighing tanks are used (as in most modern plants) the best method is to have the scale man take a measure of juice from each weighing tank as it is being discharged. These small samples are poured into a wide-mouthed container which is taken to the laboratory at the end of each hour. The container should be replaced by a clean one for the succeeding hour's samples. The sample containers should be well stoppered between subsamples. Because of the importance of this mixed juice sample it is considered advisable in the tropics to keep the sample at the juice tanks cool by surrounding the container with ice. This obviates any possibility of deterioration during this hour when the sample is kept without any chemical preservative.

Automatic sampling of the mixed juices is complicated, particularly in a factory operating more than one tandem of mills, and does not give an accurately proportioned sample. Drawing a small amount from each weighing tank as described and compositing the hourly samples in the laboratory according to the number of weighing tanks filled is the best way of assuring a weighted sample. An automatic sampler for mixed juice has been devised by Baldwin[6] which draws a small sample for each scale tank by the same motion that raises the foot valve to empty the scale tank.

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[6] McCleery, *Intern. Sugar J.*, February, 1935, p. 75.

The device is described as efficient and free from the danger of juice deterioration, which is so hard to avoid with samplers using small pipes. This objection might be raised to the Conklin mixed-juice sampler[7] which has a ⅝-in. pipe 39 in. long as a part of the sampling device.

An automatic sampler for clarified juice from weighing tanks where the control starts on the hot juice has been devised by Waddell.[8] The device is actuated by a float in the scale tank as the tank empties, and the sample is delivered through a rubber tube to a closed bottle to avoid evaporation. The bottle is in a cooling trough. The drawing accompanying the original article should be consulted for details.

609. Sampling Last-Expressed Juice (Residual Juice). This juice should be sampled by hand across the entire width of the bagasse roll of the last mill at the same time that the bagasse is sampled.

610. Care of Samplers and Containers. In order that the sampler itself may not be a source of infection and cause decomposition of the samples, it must be kept thoroughly clean and be frequently sterilized. Cleansing by means of a steam jet is usually the most convenient and efficient method. All sampling devices should be thoroughly sterilized several times daily. The sample jars should be washed with hot water after each use and be completely dried. The chemist should fully realize that in analyzing samples that are improperly drawn or cared for he is wasting his time and is obtaining misleading results.

Where measured samples are drawn at intervals by the workmen, they may be conveniently stored in wide-mouthed, glass-stoppered jars. The stopper should have a small perforation in it to prevent sticking when the temperature of the sample falls. A convenient size for the jars is 3 liters, and the mouth should be fully 13 cm. (5 in.) in diameter. The object of the large mouth is to obviate the use of a funnel and to prevent the workmen from spilling juice on the edges of the jar. Small metal cups with long handles are convenient for measuring the samples. The size of the cups depends upon the number of tanks that are filled daily; usually a 3- to 5-ml. cup for the sucrose sample and a 10- to 15-ml. one for the density sample are suitable sizes. These cups should be thoroughly rinsed with juice each time before drawing the samples, and after the addition of samples the contents of the jars should be well mixed.

Seamless enamel-ware containers, preferably white inside, provided with tight-fitting covers are recommended in preference to glass by the Committee on Uniformity of the International Society of Sugar Cane Technologists. Containers for different types of juices should differ in shape or outside color so as to be readily distinguishable.

611. Chemical Preservatives. A preservative must be added to the samples in compositing. Samples for use in the Brix and ash determinations may be preserved by the addition of mercuric chloride in the proportion of 1

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[7] *Chemical Control for Cane Sugar Factories* (Hawaii), 1931, p. 28.

[8] *Intern. Sugar J.*, July, 1936, p. 276.

part to 5000 parts of juice. One-half a milliliter of a saturated alcoholic solution of the salt per liter of juice will give this proportion. These samples may also be preserved with formalin (40 per cent formaldehyde) using 0.3 to 0.5 ml. per liter, but the mercuric chloride is preferable. Formaldehyde should never be used in compositing samples for glucose determinations as it is copper reducing. Spencer discontinued the use of formaldehyde as a preservative for laboratory samples about 1919.[9] Its use in storing juices in the factory during enforced shutdowns is valuable, although it is recognized that some inversion due to the acidity of the juice takes place in its presence (p. 175).

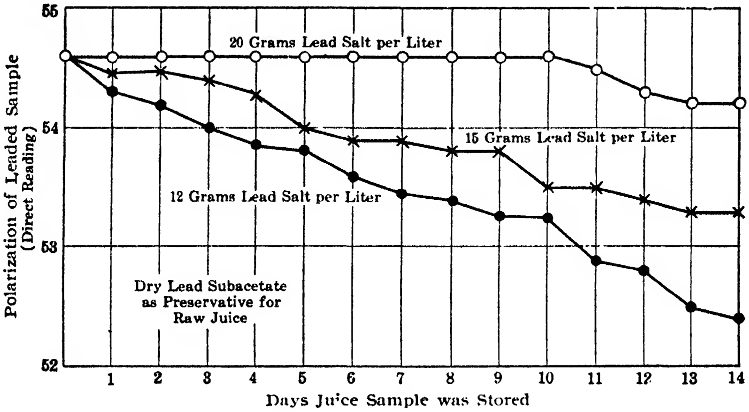

FIG. 172. Preservation of Juice with Horne's Dry Lead.

The most satisfactory preservative of juices for sucrose and glucose tests is Horne's dry lead subacetate. This is very efficient when used in the proportions of 20 grams per liter of juice. Spencer made many tests in Cuba with this salt and found that this proportion would keep juices without change in polarization for several days. The curves in Fig. 172 show the rates of deterioration with various amounts of dry lead in one set of these tests.[10] The lead should be weighed and added to the sample in proportion to the amount of juice added to the composite. For example, if 200 ml. are to be composited each hour, 2 grams of the dry lead salt should be added with this portion of juice, the whole being thoroughly shaken. The practice of adding all the preservative required for the completed sample to the container before compositing is started should never be permitted.

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[9] *Ind. Eng. Chem.*, **12**, 1197 (1920).

[10] É. Lumeau (*Sugar*, November, 1928) indicates that 6 grams of dry lead per liter is sufficient for juice storage. Spencer's investigations showed juices to be highly variable as to the amount of the preservative required; 20 grams per liter being the safe limit for all juices. In his earlier work, he prescribed 12 grams, later raising this to 15, and finally to 20 grams per liter.

It is preferable to composite the samples in the laboratory itself, thus giving the chemist a good control over sample boys, besides making sure that subsamples are drawn at proper intervals.

612. Practice in Different Countries. The Committee on Uniformity of the International Society of Sugar Cane Technologists notes the following regarding the diversity of opinion in different countries as to chemical preservatives for juice. Java employs 100 mg. of mercuric chloride (not medicinal tablets containing sodium chloride) for each liter of juice, but it is noted that for complete sterilization larger quantities are necessary. Cuba and Puerto Rico recommend mercuric chloride only for the sample to be used for the Brix determination, at the rate of 0.5 ml. of a saturated solution (about 35 to 40 mg. of $HgCl_2$) per liter of juice; this, it is stated, does not affect the Brix of the juice. Hawaii uses mercuric chloride only in samples for the determination of reducing sugars, at the rate of 250 mg. per liter of juice. Since mercuric chloride coagulates protein, Natal and Mauritius prescribe a solution containing 95 grams mercuric iodide, 50 grams potassium iodide, 40 grams formalin, and 200 grams water; 0.2 to 0.5 ml. of this solution is added to each liter of juice. Hawaii, India, Japan, and the Philippines use formalin in samples for both Brix and pol determination, 1 ml. per liter of mill juices, and 0.5 ml. for clarified juice, filtered juice, and sirup under 60° Brix. But formaldehyde has been proved to be rather unreliable. For pol determination Cuba, Louisiana, Natal, and Puerto Rico recommend Horne's dry lead, and this has been found to be quite satisfactory. Mauritius prefers dry neutral lead acetate because it does not affect the rotation, whereas subacetate does. Olivier has reported that juices preserved with dry neutral lead acetate, if filtered at once after clarification, undergo no deterioration in twelve hours or more, but that, if the juice is left in contact with the precipitate, there is decided decomposition in less than six hours.

613. Sampling for Dry Milling Factor. In the older methods of mill control (see Chapter 35) and to obtain certain information about the operation of the mills it is customary to operate the mills periodically without imbibition water. Special sampling is necessary to obtain the so-called milling factor which is used to reduce the degree Brix of first-expressed juice (generally crusher juice) to terms of undiluted juice. The juice from the crusher (or crusher and first mill) and that from the entire system are separately sampled during the period when the mills are operating with no maceration water whatsoever. The sampling should be by automatic devices and should be continuous for an hour or longer. The first sample is of relatively high Brix, polarization, and purity, owing to the comparatively moderate pressure applied in crushing the cane. The Brix of the second sample is lower than that of the first on account of the very heavy crushing by the mills which extracts the rind juice and many of the impurities of the cane. The factor is the relation between the Brix of the two samples. The following example illustrates the calculation and use of the factor.

Example. Brix of the two samples, respectively, 20° and 19.5°.

$$\text{Factor} = \frac{19.5}{20} = 0.97$$

Brix of the first-expressed juice in regular milling, 19.6°, then,

$$19.6 \times 0.97 = 19.0° \text{ Brix of the normal or undiluted juice}$$

During the time that these dry milling tests are being made enough cane of uniform quality for at least a half hour's milling should be on hand. All conditions of pressure, speeds, etc., should be maintained as in normal operating conditions. (See also Secs. 783 and 784 for further discussions of dry milling factors.)

614. Preserving Juices by Refrigeration. In South Africa there has been considerable progress made with a method which preserves sugar juices by freezing. Foster and Karlson[11] found that juices could be kept in a refrigerator frozen solid at minus 10° C. almost indefinitely without deterioration. They showed that the juice should be analyzed immediately after thawing out as it tends to deteriorate rapidly when standing. G. C. Diamond studied this matter further and worked out a special refrigerator for this purpose. A machine which he describes[12] carries six or more containers made of copper 14 in. deep, 5 in. in diameter, tapering to 4 in. at the bottom, which is hemispherical. They have brass covers fitted with rubber stoppers 1 in. in diameter through which samples can be introduced, and the containers are supported on a projecting flange which rests on a shelf in the refrigerator. Samples were kept for various periods from twelve to four hundred hours at 3° C. Figures are given which show no change in the reducing sugar ratios, Brix, or sucrose in ten days' storage time.

Mention has already been made (Sec. 608) of cooling juice samples by surrounding them with ice in order to prevent any deterioration during the hour in which the juice sample is kept at the weighing tanks without preservation. This practice has been mandatory for many years in the factories of the Cuban-American Sugar Company. It is a precaution which is well worth carrying out as even slight deterioration in the mixed juice is reflected in the entire control of the house.

615. Sampling the Filter Press Cake. The composition of the filter press cake varies in different parts of the press and of the cake itself. This makes strictly accurate sampling impracticable since this work must be left in the hands of the pressmen. The best approximations and comparisons are obtained by cutting pieces of the cake systematically from various parts of the press.

The instrument shown in Fig. 173 is very suitable for this sampling. It is made of heavy brass and of such a size that it may readily be grasped by

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[11] *Intern. Sugar J.*, 1932, p. 104.

[12] *Intern. Sugar J.*, November, 1936, p. 427.

the hand over the cover. This latter is fastened to the body or receptacle by bayonet catches and a set screw. The cutter is a brass tube 1/16 in. thick and about ½ in. in diameter at the cutting edge. The body of the tube is coned towards the receptacle, so that the plug of cake will readily pass into the latter. The cutting edge should be of the thickness of the tube to prevent damage to the filter cloth, or a guard should be placed on the cutter to prevent complete penetration of the cake.

Several of these samplers should be provided so that one may be filled from each press. The pressmen should cut a number of plugs in the usual procedure, e.g., from various parts of the second cake, then from the fifth cake, and so on. The plugs are accumulated in the receptacle, one remaining in the cutter and closing it. A small tube, open at both ends, is attached to the inside of the cover and holds a sponge saturated with a mixture of one part chloroform and six parts strong ammonia. The sample should be sent to the laboratory each time a press is filled. This provides a check on the sampling and count of presses cleaned. The sampler should be thoroughly cleaned after removal of the plugs and returned to the presses.

The sample may also be obtained by means of a brass tube, fitted with a piston for removing the plugs. A cork borer may be employed for the purpose, but must be used with care on account of risk of cutting the filter cloth. The plugs should be stored in a covered vessel in an atmosphere saturated with ammonia and chloroform.

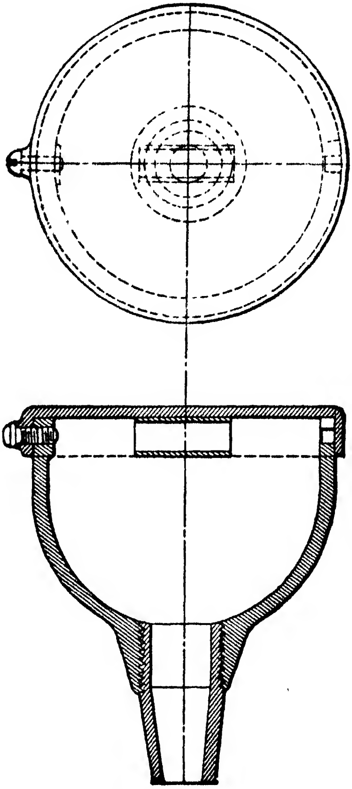

FIG. 173. Sampler for Filter Press Cake.

It is difficult to control or check the sampling of the press cake, especially as there is a natural tendency for the pressmen to sample only the hardest parts of the cakes. With the use of systematic methods of washing the cake, the error of sampling, however, becomes less important as the loss of sugar is small.

A much more elaborate method of sampling filter cake is prescribed in Java where all the cake is mixed with water to a homogeneous paste and is sampled as it is pumped from the factory. Moisture is determined in this paste and in the original cake, and the polarization of the diluted cake is calculated to that of the original from the relationship of the two dry substances. The procedure seems too complicated for the results achieved.

The samples of press cake, collected as has been described, are not usually separately analyzed, but are composited, preferably during six-hour periods. Each time that a filled receptacle is received by the laboratory, the plugs of press cake should be removed and chopped into fine pieces and thoroughly mixed. A measured portion of the minced cake from each press should be placed in a jar in an atmosphere of the chloroform-ammonia mixture as above. The united subsamples should be thoroughly mixed and analyzed once every six hours.

If preferred, the subsamples may be composited during a twenty-four-hour period as follows. Weigh 25 grams of the six-hour sample and wash it into a glass mortar with water and 6 ml. of lead solution. Rub the cake to a paste to incorporate the lead-salt with it. Cover the mortar pending the receipt of the next sample and proceed as before, adding the second portion to the first, with additional lead. The third and fourth subsamples are treated in the same way, and finally the mixture is washed into a 400-ml. flask, diluted to the mark and polarized.

616. Sampling Sirups. When the sirup may not be thoroughly mixed in the tank before sampling, a "thief" sampler may be used. This consists of a tube, long enough to reach the bottom of the tank and provided with a suitable ball or other type of valve. The tube is passed through the sirup, to the bottom of the tank, and on its removal takes with it a sample of each layer of sirup.

In factories which pump all the sirup through a single pipeline to the storage tanks, this material may be sampled by a Calumet pipeline type of sampler (Sec. 604) and with increased accuracy as compared with hand methods. This method is especially desirable when the sirup analysis is used as a basis for the calculation of available sugar. Probably as satisfactory a method as any is to have the multiple effect operator take a small portion of the sirup every fifteen minutes as it leaves the effect, compositing it without preservative.

617. Sampling Massecuite and Molasses. Massecuites are not usually of uniform density or composition because of imperfect circulation in the vacuum pan. The sample of massecuite should therefore be drawn, little by little, as the strike is being discharged from the pan if the greatest accuracy is required. In ordinary practice a grab sample taken as the pan is discharging will serve, or preferably, portions may be taken at the beginning, the middle, and the end of the strike as it runs from the pan.

If the samples of the several strikes of massecuite included in the day's work are to be composited to form a single sample, a measured portion of each must be used. If the pans are of uniform size, one measure will answer for all, otherwise there should be a set of proportionate measures, one for each pan. A small stemless funnel of glass forms a convenient measure.

Molasses is sampled in the same way as sirup, and, according to the exigencies of the work, the samples are analyzed separately or are composited. A composite sample of the final molasses should be prepared from time to time for the determination of the sucrose by the Clerget method and the solids by drying for the calculation of the true purity. These samples should represent a definite quantity of the material.

SAMPLING RAW SUGAR

618. Importance. The sampling of raw sugar has received more attention than the sampling of other sugar products because of the economic importance and also because of tariff regulations. Sampling raws falls into two rather distinct classes: first, sampling sugar as it is produced, and second, sampling from packages. The sampling from packages is a more complicated operation or rather one requiring greater care because the sugar may not be evenly distributed throughout the package owing to drying out, wetting, or staining, and the composition may also vary sharply from bag to bag. Sugar as produced is generally sampled by strikes and is of fairly homogeneous composition.

619. Sampling Sugar as Produced. This is most readily and most accurately done by the workman at the sugar scale who should remove a sample from each package of sugar as he weighs it. The samples should be thrown into a tin box provided with a funnel-shaped opening to receive them, as shown in Fig. 174. The can is conveniently placed on a shelf at the scaleman's side in order to prevent stooping. The composite samples so obtained should be analyzed at frequent intervals, preferably an analysis every six hours, and the number of packages represented by the sample should be recorded for use in calculating the averages. The can shown at the right of the figure is the type generally used in refineries where raw sugar is received in packages. The funnel top of the can is replaced by a closed top when the can is full, or the lot of bags is complete.

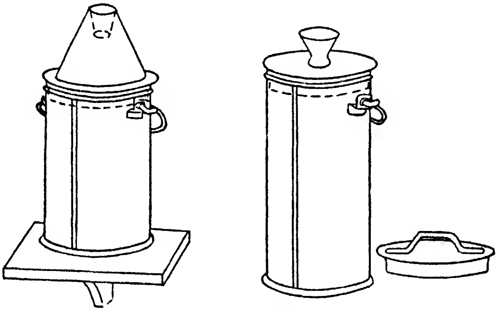

FIG. 174. Sample Cans for Sugar.

An automatic sampler for sugar dropping from automatic scales is used in Hawaii. It is a turbine type attached to the side of the hopper above the bag. When the charge of sugar drops, it strikes the "turbines," and a very small portion lodges in the spoon on the end of each arm. Owing to the impact, the "turbine" is whirled around, and the small sample of sugar is thrown into the receiving box hung on the side of the hopper, thus automatically taking a sample from each bag of sugar. The receiving box is arranged so that it is airtight and is removed at the end of each strike or at the completion of a certain number of sacks.

620. Sugar Trier. In sampling from packages a "trier" is employed (Fig. 175). The trier is a trough-like instrument, which, being plunged into a quantity of sugar, will, on withdrawal, remove a sample representative of the sugar through which it has passed. The diagram shows the standard "short" trier required by the U.S. Treasury Department. The instructions for sampling sugars as given by the Treasury Department are as follows:

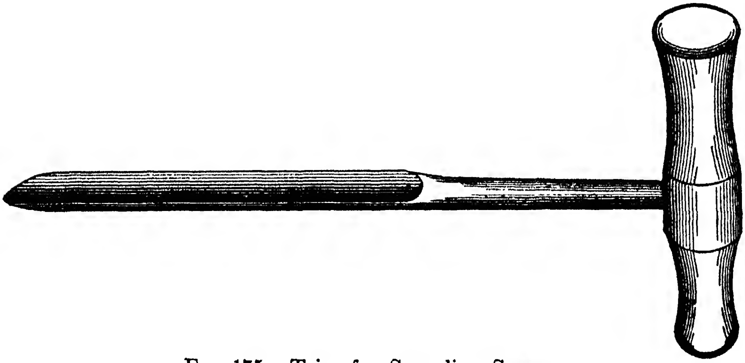

Fig. 175. Trier for Sampling Sugars.

> In the sampling of baskets, bags, ceroons and mats the short trier will be used, care being exercised to take the sample fairly from the central contents of the packages, and in such manner that the samples from each class of packages shall be uniform in quantity. When the hard condition of the sugar renders the use of the short trier impracticable, the knife may be used.

The Treasury regulations formerly required the sampling of all packages in a lot, and this conforms to commercial usage, but because of the labor shortage incident to the war the Customs regulations now permit sampling one bag in every draught of four to seven bags.

621. Mixing Sugar Samples. As has been described above the cans in which sugar samples are kept should have tight-fitted covers, and mixing should be done as soon after the samples are completed as possible. A convenient sugar mixing table is made in the form of a U-trough with one end open, and all surfaces which come in contact with the sugar are covered with sheet zinc. (Absorbent materials such as paper should never be used.) Lumps should be crushed and thoroughly incorporated with the rest of the sample. The method of mixing followed in the New York Sugar Trade Laboratory is as follows:

> The sugar is poured out from the can upon a clean sheet of plate glass, all pieces of bagging, baskets, mats, etc., are removed, and the sample is thoroughly mixed with a clean steel spatula. Lumps are reduced by means of a steel rolling pin and incorporated with the rest of the sample. The plate glass and steel rolling pin are cleaned and wiped perfectly dry each time before using. The reduction of lumps is of greatest importance in securing uniformity of sample; the difference in polarization between the lumps and the fine portion of some sugars has been found to vary several per cent. The mixing and sub-sampling should be done as rapidly as possible to avoid evaporation.
>
> When samples are brought into the laboratory during freezing weather, the cans or bottles are first allowed to come to approximately the room temperature before opening and mixing. This is done to guard against condensation of moisture upon the cold sugar, which would lower the polarization.

622. Automatic Bag Counter. A convenient device for counting bags of raw sugar when hand-trucking (checking out of cars to warehouses, etc.) is shown in Fig. 176. The counter consists of two raised runways with sides (not shown) over which the truck wheels pass while the truckman walks between. The left-hand wheel of the truck trips a treadle which is connected to the counter by means of springs as shown. The action should be regulated so as to avoid jar and vibration on the counter itself.

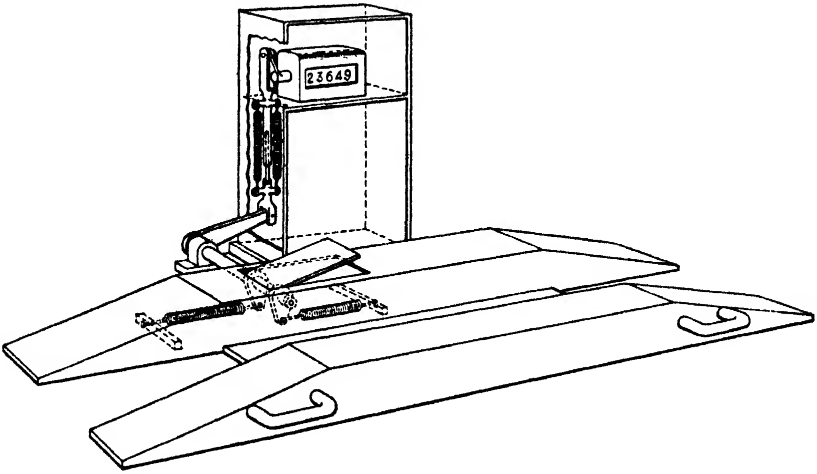

Fig. 176. Counter for Trucked Bags.

CHAPTER 26

ANALYSIS OF THE SUGAR CANE

623. Need for Direct Analysis. The direct analysis of the cane is rarely needed or carried out. As will be shown in the chapter dealing with mill control (p. 597) no general analysis of the cane is made for any of the systems employed and, at most, the only constituent directly determined is the fiber. The usual method of judging the sugar content of cane, e.g., for maturity or richness, is to analyze the juice obtained by laboratory milling.

624. Preparation of Sample. The first step in the analysis of the cane is the preparation of the sample. As has been shown a representative sample of the cane is almost impossible to obtain. With knife or shears, the sample of whole cane cannot be prepared rapidly enough to avoid error by evaporation, nor do such methods result in the reduction to a suitable state of division for a thorough extraction of the sucrose. In the analysis of whole canes it should be noted that the composition varies greatly in different parts of the stalks, thus complicating the preparation of the sample.

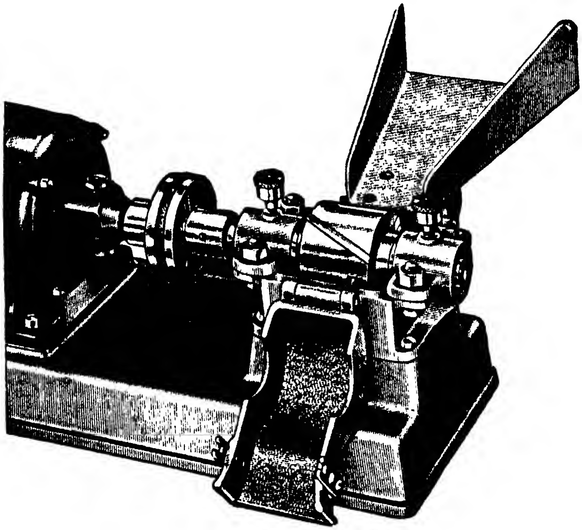

Fig. 177. Warmoth-Hyatt Cane Disintegrator.

Whole cane can be rapidly and properly shredded by means of the Warmoth-Hyatt disintegrator, Fig. 177. Knives are fitted into the revolving shaft, which is connected directly to the motor. In use the cover, shown open in the illustration, is closed and bolted, and the cane (cut into short pieces) is fed down the hopper and is shredded, dropping through an opening in the base, not shown. The machine is usually mounted on a table with a drawer underneath in which the sample is collected. The knives can be removed for sharpening or replacing.

625. Polarization. Direct Method. For the direct estimation of the polarization in the whole cane proceed as follows: Place 100 grams of cane prepared as above in a suitable dish or beaker and add approximately 200 ml. of boiling water and boil for ten minutes. Carefully drain off the liquid and add another portion of 200 ml. of water, and again digest for ten minutes. Repeat these digestions with water in all seven times, and after the last, press the residue in a hydraulic or other powerful press, uniting the portions of solution drained from the chips. Cool the liquid and weigh it and also determine its degree Brix. To approximately 100 ml. of this solution in a sugar flask add dry lead subacetate for clarification. After thorough mixing and filtration polarize the filtrate, using a 400-mm. observation tube. Divide the polariscope reading by 2, since a double-length tube is used, and calculate the per cent pol in the solution by Schmitz's table, p. 755. From the per cent pol in the solution and the weight of the latter, calculate the weight of pol. This number is the weight of pol in 100 grams of cane or the percentage of pol in the cane.

626. Estimation of Sucrose (Pol). Indirect Method. The following is the customary method of indirect analysis of the cane in working with or without saturation. The weight of the cane is taken as reported by the cane department; the weight of mixed or diluted juices from all the mills is ascertained by direct weighing or by measurement and calculation by the laboratory; the weight of bagasse is estimated by deducting the weight of the mixed juice from the sum of the weights of the cane and the imbibition water; the sucrose (or pol) is determined in the juice and the bagasse by direct analysis. Manifestly the weight of sucrose (or pol) in the cane is the sum of the weights of sucrose (or pol) in the juice and bagasse and this number divided by the weight of the cane and multiplied by 100 gives the percentage of sucrose (or pol) in the cane.

Several conditions may lead to inaccuracy in the above method. The juice may be diluted by leakage of the water used in cooling the mill journals; the bagasse parts with more or less moisture by evaporation in passing through the mills; there may be inaccuracies of weights, measures, and analyses. The first of these need not be expected where modern mills of good construction are operated. With the old types of mills, however, it is often necssary to run cooling water upon the bearings, and a part of this is liable to leak into the juice. Such leakage may usually be detected by noting the relation between the percentage of saturation water and the dilution number. The error from the evaporation of the moisture cannot be eliminated, but it is probably fairly constant in a given milling plant. Inaccuracies of weights, sampling, and analysis are usually avoidable. Inaccuracy in the measurement or weight of the saturation water, which is used in calculating the weight of the bagasse, should be avoidable, but it is a frequent source of error. (See also Sec. 772 for discussion of bagasse weights compared with estimated weight.)

627. Direct Determination of Fiber. The direct determination of fiber in cane has been sanctioned by the Committee on Uniformity of the International Society of Sugar Cane Technologists as one of the two methods for arriving at the weight of the bagasse. (The other method is by difference as shown in the preceding paragraph.) The practice is the official method in Hawaii for factory control and is also employed in Australia in the determination of the price of cane. The method of sampling cane in these two countries has already been described (Sec. 602). An essential requirement of accuracy in the fiber determination is that the cane be shredded fine enough to rupture the cells, otherwise there is danger of incomplete extraction. The Warmoth-Hyatt disintegrator (Sec. 624) or similar apparatus is recommended, although coppers, meat slicers, or an ordinary wood plane are permitted in Hawaii, provided thorough disintegration of the sample is accomplished before it is analyzed.

The Hawaiian method, recommended by the International Society of Sugar Cane Technologists, is as follows: [1]

> Transfer the weighed finely divided sample to a strong linen bag and tie with heavy thread. Wash in running water until the washings are clear, squeeze out surplus water, place the bag in a heavy canvas bag and press in a screw press. From 600 to 1000 pounds per square inch is a suitable pressure. The pressed mass is then broken up, the bag examined to see that no holes have been made during pressing, treat with cold running water for at least two minutes and repress. Five or six alternate soakings and pressings will extract a properly prepared sample. The thoroughness of the extraction may be roughly checked by crushing some of the larger particles between the teeth. If juice cannot be detected by taste in the larger pieces the extraction may be considered complete. Dry sufficiently that the fibre may be easily removed from the bag, transfer to a tray, removing the particles adhering to the bag by rubbing, and dry at 125° C. Three hours' drying should suffice. Weigh quickly to avoid absorption of moisture. Instead of pressing, the sample can be extracted by washing in cold running water for 24 hours.

The Queensland method for fiber determination in cane recommends extraction in cold running water for one hour, with hand squeezing of the calico bag at intervals. This is followed by immersion in boiling water (circulating) for one hour with further squeezing at intervals. The Committee on Uniformity says that boiling water may convert pentosans and pectins, which are constituents of the fiber, into soluble products to some degree, thus reducing the result of the analysis.

628. Indirect Determination of Fiber. *Provisional Method (Hawaiian).* The fiber may be determined indirectly from the moisture in the chopped or disintegrated cane. Every hour or two a sample of the cane on the car-

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[1] *Methods of Chemical Control*, second edition, 1931, p. 33.

rier is reduced to a fine state of division. About 10 grams of the fine sample is weighed in a double watch glass and dried for four hours at 110° to 120° C. or better under vacuum at 100° C. This gives the per cent moisture in the cane. Calculate the Brix of the absolute juice from the Brix of a sample of first expressed juice taken at the same time as the cane sample, using a milling formula obtained by dry milling (see p. 501). Calculate fiber in cane by the following formula:

$$\text{Fiber \% cane} = 100 - \frac{100 \times \text{\% Moisture in cane}}{100 - \text{Brix of absolute juice}}$$

629. Calculating Fiber in Cane Indirectly from Bagasse Figures. The following indirect methods of fiber determination are those usually employed in mill control, the second method being preferable: (1) The bagasse is sampled and the fiber is directly determined in it. The bagasse produced per cent cane times the per cent of fiber in the bagasse times 100 equals per cent fiber in cane. (2) The per cent sucrose (pol) in the bagasse and the purity of the residual juice and the moisture are determined. The percentage of juice solids in the bagasse is calculated from the polarization and the purity of the residual juice. The sum of the juice solids and the moisture deducted from 100 gives the percentage of fiber in the bagasse. The purity of the juice flowing from the bagasse roll of the last mill in the train is considered to be that of the residual juice (see p. 574). The following example illustrates the second indirect method.

Example. Per cent of bagasse, 25; sucrose (pol) in the bagasse, 4 per cent; moisture, 48 per cent; residual juice purity, 78 per cent.

Then,

$$\frac{4}{0.78} = 5.13 \text{ per cent juice solids}$$

$$100 - (5.13 + 48) = 46.87 \text{ per cent fiber in bagasse}$$

$$25 \times 0.4687 = 11.72 \text{ per cent fiber in the cane}$$

Spencer compared a large number of analyses by method 1 with the calculated fiber by method 2. The agreement by the two methods was so good that he discontinued the direct determinations of fiber in the bagasse in the factories under his supervision. (See Sec. 716.)

CHAPTER 27

ANALYSIS OF THE JUICE

630. Determination of the Density. The density of the juice is almost always determined by means of a hydrometer (Sec. 516), though a pycnometer (Sec. 519) or the Westphal balance (Sec. 520) may be used. The Brix spindle is the more convenient hydrometer for this purpose, since its readings are used in calculating the coefficient of purity.

The readings on the Baumé scale, or the specific gravity as determined by the Westphal balance or pycnometer, may be converted into degrees on the Brix scale by means of the table on p. 718 or p. 736, according to the standard temperature selected.

The juice is first thoroughly strained through a fine-mesh screen (a cone of wire gauze is convenient) to remove particles of bagasse fiber and other suspended matter. Samples of juice for Brixing or drying should be preserved with either mercuric chloride or formaldehyde, preferably the former. Lead-preserved juices should never be used for these determinations.

In using the hydrometer, fill a wide cylinder to the brim with the sample of juice and set it aside for the escape of air bubbles. The time required for this varies from a few minutes to half an hour, but usually ten minutes suffice. A vacuum connection as described in Sec. 517 facilitates the removal of the air bubbles and should be used in all routine work.

The spindle should be lowered into the cylinder, after the escape of the bubbles, causing the juice to overflow and carry away with it the froth and mechanical impurities floating upon the surface. It is well to blow on the surface of the juice as it overflows to help remove the froth. The spindle should now be lowered farther into the juice, until it floats, care being taken to see that the stem is wet for a few tenths above the point of which the spindle will come to rest.

After allowing sufficient time for the temperature of the spindle to reach that of the juice, read the scale as directed in Sec. 517 and illustrated in Fig. 144. The temperature of the juice should be noted for use in correcting the observed density.

The use of hydrometers calibrated at 17½° has been largely discontinued and most modern instruments are standardized at 20° C., although Hawaii and Java and some other tropical countries use hydrometers standardized at 27½° C. For instruments standardized at 20° the temperature corrections may be found in the table on p. 728. For example, if the reading of the spindle is 21.20 at 25° C., the correction found under the 20 column and opposite the 25° temperature in the side column = 0.32 and the corrected Brix is 21.20 + 0.32 = 21.52. For hydrometers standardized at 17½° C. the correction may be made by using the same table and correcting to 20° in the same way. Then the correction is added to 17.5° with the sign changed. In the example above the correction would be 21.20 + 0.32 + 0.15 = 21.67. If the temperature is below 20° C. the correction to 17.5° C. is made by taking the algebraic sum of the minus correction to 20° and the plus correction from 17½° to 20°.

631. Total Solids by Drying. The determination of total solids by drying in juice is not recommended for ordinary control work. If it is found necessary to do this for investigation purposes, the juice sample should be weighed by difference from a weighing bottle, preferably one fitted with a ground-in pipet and rubber bulb. Use one of the methods of drying on pumice or quartz sand (Sec. 522) or the Spencer oven, type *A*, absorbing the juice on asbestos (Sec. 524). In any case employ sufficient juice to give about 1 gram of dry matter. Observe precautions for protecting the sample from absorbing moisture after drying (Sec. 521). The drying on quartz or sand should be done in a vacuum oven at a temperature not higher than 70° C. The weight of dry matter divided by the weight of juice used, multiplied by 100 equals per cent of total solids.

632. Total Solids from the Refractive Index. The refractometer may be used for estimating the solids as described on p. 444. The dipping refractometer may also be used for juices.

633. Relationship between Brix, Solids by Refractometer, and Solids by Drying. It is to be understood that the total solids as obtained by the refractometer do not correspond either to those obtained by direct drying or to the apparent solids shown by the Brix hydrometer. The differences are due to the presence of salts and other non-sugars which have a different density and different refractive index from the sugars present in the juice (see p. 431). Fort and McKaig[1] worked out a relationship between the Brix and solids by drying of over two hundred samples of crusher juice in Louisiana (1931 crop) which was correlated on the ash content. They found that if the product of the ash per cent solids and the Brix is multiplied by the factor 0.0120 and this result is subtracted from the Brix the true solids will be closely indicated.

Example. A juice has Brix of 17.0° and (carbonated) ash of 2.5 per cent on solids. Then,

$$17.0 \times 2.5 \times 0.0120 = 0.51$$

which indicates that the true solids will be

$$17.0 - 0.51 = 16.49\%$$

Such a relationship probably applies to Louisiana juices only but similar factors may be applicable generally.

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[1] *Ind. Eng. Chem., Anal. Ed.*, September, 1936, p. 333.

634. Determination of Polarization.[2] *Special Pipet (Spencer's or Crampton's).* This method is generally used only for juices that have not been preserved with lead subacetate, i.e., special samples and the like.

The pipet,[3] Fig. 178, is so graduated that if filled to the mark corresponding with the observed (uncorrected) degree Brix, it will deliver two normal weights of the liquid. Makers now graduate these pipets to deliver either 52.096 grams for use with the older system of Mohr's 100-cc. flasks at 17½° C. or 52 grams with the 20° C. standard and 100-ml. flasks. The usual graduation is for a range of densities from 5° to 25° Brix in tenths. These instruments, called sucrose pipets, are preferably made with a delivery tube about 4 in. long. With the short tube the pipet may be supported by the flask while draining, leaving the chemist free to continue a series of such measurements with other pipets.

FIG. 178. Sucrose Pipet.

In using this pipet in the analysis of a juice, proceed as follows. Determine the density of the juice with a Brix hydrometer, noting the degree Brix without temperature correction. Fill the pipet with juice to the mark corresponding with its observed degree Brix, and discharge it into a 100-ml. flask. Add 3 to 5 ml. of 54° Brix lead subacetate solution, complete the volume to 100 ml. with water, mix thoroughly, and filter the contents of the flask. Polarize the filtrate, using a 200-mm. tube, and divide the polariscope reading by 2 to obtain the polarization. The juice should not be expelled from the pipet by blowing, and sufficient time should be allowed for thorough drainage.

The sucrose pipet may be used in connection with juices preserved with Horne's dry lead or with juices clarified by the lead salt by making the measurement after filtration at the temperature of the Brix observation. It is to be understood that the Brix is to be taken in a portion of the juice preserved with formaldehyde or mercuric chloride. This use of the pipet obviates that of Schmitz's tables, but involves completion of the volume to 100 ml.

The calibration of pipets should be verified against a

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[2] The use of the term sucrose for polarization has been general in the raw sugar industry, particularly in the western hemisphere. The practice has been condemned by the Committee on Uniformity of the International Society of Sugar Cane Technologists.

[3] This pipet was devised by C. A. Crampton and G. L. Spencer, independently and at about the same time. It is termed "Crampton's" or "Spencer's sucrose pipet" by the dealers.

balance. A volume of sugar solution corresponding to an uncorrected degree Brix should be measured in the pipet. If the instrument is correctly graduated it should deliver two normal weights of the solution (52.096 grams or 52 grams, depending on which flask it is intended to be used with).

It is not advisable to use these pipets with liquids of a higher density than 25° Brix or of greater viscosity than cane juice. These pipets are usually used in the analysis of miscellaneous samples of juice and in the rapid testing of diluted massecuites and molasses for guidance in the vacuum pan work.

635. Determination of the Polarization. General Methods. The use of Horne's dry lead subacetate for preserving juices for polarizing has become so general as to be almost universal in Cuban and Puerto Rican practice. (See Sec. 611.) It has proved of great convenience in factory control, since it does not dilute the samples of juice that it preserves.

The polarization of lead-preserved juices is extremely simple. Filter after thorough mixing, polarize the filtrate in a 200-mm. tube and calculate the polarization by Schmitz's table for undiluted solutions (p. 755). The polarization is ascertained from the table as follows. Suppose that the observed[4] degree Brix of the duplicate sample (preserved with mercuric chloride or formaldehyde) is 18.1 before correction for temperature; the polariscope reading on the filtered lead solution is 60.5. Under the column 18 (the nearest degree Brix to that observed) opposite 60 (the whole number of the polariscope reading) is 14.56; add to this the number 0.12 which is found in the small table opposite 0.5, the tenths of the polariscope reading. The completed number, 14.68, is the polarization of the juice.

Any juice may conveniently be analyzed by Horne's[5] method, using the dry lead salt for clarification. A portion of the juice after Brixing is placed in a cylinder or flask, 3 to 4 grams of the dry lead subacetate is added for each 100 ml. of juice taken, and after thorough mixing and filtration the filtrate is polarized in a 200-mm. tube as usual. Calculate the polarization by Schmitz's table as described above.

The amount of lead subacetate employed must be gaged with caution to avoid an excess because of its reaction with levulose (Sec. 477). Fully matured tropical cane contains but small traces of levulose and occasionally none at all, whereas unripe or damaged canes may contain much. In most control work the error due to the effect of the lead on the levulose is disregarded, but it may be eliminated by the following procedure, but with the introduction of a precipitate-volume error: Shake the sample thoroughly, and while the precipitate is still in suspension measure 100 ml. of the mixture in a sugar flask and dilute it to the 110-ml. mark with dilute acetic acid of sufficient strength to acidulate slightly the contents of the flask. Filter, polarize, and

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[4] Wherever the term "observed" or "uncorrected" Brix is used it is understood that the Brix uncorrected for *temperature* is meant. If the hydrometer employed has been found by standardization to require a correction this hydrometer correction must be applied to obtain the "observed" or "uncorrected" Brix.

[5] *J. Am. Chem. Soc.*, **26**, 186; *Intern. Sugar J.*, **6**, 51.

calculate the polarization by Schmitz's table (p. 755), adding one-tenth to allow for the dilution.

A preferable method is that according to Cross,[6] using dry oxalic acid instead of acetic acid and dry lead and thus eliminating both the levulose and the precipitate-volume errors.

After the addition of the dry lead salt, with thorough mixing, add dry powdered oxalic acid in sufficient quantity to precipitate almost all the lead in solution and filter. Polarize the filtrate and calculate the polarization by Schmitz's table, p. 755.

In both these methods note that the acid is added to the leaded solution before filtering. Filtering first, then acidifying, does not correct the levulose error.

The use of dry powdered neutral lead acetate is specified for the analysis of juice in Mauritius, and Olivier [7] recommends its use as a preservative specifying that the precipitate produced by the lead should be filtered off. The defecated and filtered juice is reported to "keep fairly well without further addition of antiseptic." [8] An objection to the neutral lead salt is that it does not always clarify the solution sufficiently for accurate reading in a 200-ml. tube. It is also doubtful whether it has the preserving power of the basic salt.

636. True Sucrose by Clerget's Method. The determination of sucrose (true sucrose) by double polarization is not required for general factory control but the determination was carried out by Spencer in the factories under his control for a Clerget balance or sucrose balance in the technical accounting. Such procedure is also recommended by the Committee on Uniformity of the International Society of Sugar Cane Technologists. The Committee's recommendations are fully met in the following method for sucrose in juice. The general subject of the Clerget methods is discussed in Chapter 19.

Clarify the juice sample by the addition of Horne's dry lead subacetate, 3 to 4 grams per 100 ml., or in factory control work use a composite sample of the juice preserved with the dry lead. After the lead precipitate and the juice have been thoroughly mixed allow the precipitate to settle and then pipet two 50-ml. portions of the supernatant liquid into each of two 100-ml. flasks. Do not delead the solutions. Proceed with the Clerget according to Jackson-Gillis method IV (Sec. 488), adding 10 ml. of salt solution (231.5 grams per liter) to one flask for the direct reading and inverting the 50 ml. in the second flask as per directions.

Pipettes should be calibrated with a sugar solution of the approx. density of juice, not with distilled water, as the differences in viscosity are sufficient to cause important errors.[9]

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[6] William E. Cross, *Louisiana Bull. 125 Agr. Expt. Sta.*, p. 39.

[7] *Rev. Agr. Maurice* (1933), No. 68, p. 48.

[8] *Intern. Sugar J.*, September, 1933, p. 360 (abs.).

[9] Committee on Uniformity of the International Society of Sugar Cane Technologists.

The calculation for juice does not give the Clerget sucrose immediately, but a figure which is to be used with Schmitz's tables as in the following example.

Example. Observed Brix of duplicate sample, 18.1.

P	60.2
$-P'$ at 25.7° C.	18.3
$P - P'$	78.5

From the table given in Sec. 637 the factor for 18° Brix juice is 132.36 and the correction for 25.7° C. (from Table 49) is

$$-3.02 = 129.34$$

$$\frac{78.5}{129.34} = 60.69$$

Consulting Schmitz's table as before:

Opposite 60 in column for 18° Brix	14.56
Interpolating for 0.69	0.17
Clerget sucrose	14.73

The leaded composite sample for Clerget work may be kept as long as five days if proper precautions as to sterilizing containers and protecting the sample from contamination are observed.

637. Clerget Divisors for Juice. It has been shown in Sec. 491 that the Clerget divisor should be based on dry substance concentration and not sucrose concentration. The formula for the divisor at 20° C. is $132.63 + 0.0794 (g - 13)$ in which g is the grams of solids in 100 ml. of the solution taken for inversion. These divisors have been calculated in the following table for juice from 8° to 26° Brix. The temperature correction (to be subtracted from the divisor given in the table) is $0.53(T - 20)$ in which T is the observed temperature to tenths of a degree of the invert solution at the time of reading. The temperature corrections are worked out in Table 49, p. 796.

Brix Juice	Clerget Divisor	Brix Juice	Clerget Divisor
8	131.92	18	132.31
10	131.99	20	132.39
12	132.07	22	132.47
14	132.15	24	132.55
16	132.23	26	132.63

638. Reducing Sugars. *Preparation of the Sample.* If the juice sample contains no preservative it may be filtered directly through dry kieselguhr, no lead or other clarification being used. (Sec. 497.) Samples preserved with mercuric chloride may also be treated this same way, but juices containing formaldehyde should never be used for glucose determinations as formaldehyde reduces copper. Before adding the kieselguhr add dry sodium oxolate (about 0.25 gram for each 100 ml. of juice) to remove lime salts. (See Sec. 498.)

For juice samples preserved twenty-four hours with Horne's dry lead (20 grams per liter of juice) Harris[10] has shown that results corresponding with the reducing sugars found in the fresh juice untreated (filtration through kieselguhr only) may be obtained by the following procedure. To the well-mixed leaded juice, unfiltered, add 0.75 gram of powdered oxalic acid for each 100 ml. of sample, shake thoroughly, allow to stand a few minutes, and then filter.

In his investigation, Harris showed that neutral salts (potassium or sodium oxalate, sodium phosphate, and others) are useless as reagents to break up the lead levulosate formed by the presence of the basic lead salt. He also proved that results are valueless if the lead precipitate is filtered off and the deleading agent added to the filtrate. This method, using oxalic acid to delead lead-preserved juices for glucose work has proved of real value for control tests and the establishment of the glucose balance of the factory.

In spite of these findings by Harris, directions are still given in some methods for deleading the juice preserved with basic dry lead after filtration.

McAllep and Cook[11] recommend neutral lead acetate clarification for juices for control tests (presumably with samples preserved with mercuric chloride), then filtering and adding disodium phosphate-potassium oxalate solution (see Sec. 498) to an aliquot portion of the filtrate to remove all lime and lead. The amount of lead solution specified is 1 gram per 10 grams of juice with 3 ml. of the phosphate-oxalate mixture.

639. Method of Determination. Gravimetric. The Spencer glucose pipet is similar in principle to the sucrose pipet described in Sec. 634 in that it delivers a certain weight of juice corresponding to the observed Brix, i.e., uncorrected for temperature. For lead-preserved juices the Brix is taken on a parallel sample preserved with mercuric chloride or formaldehyde as previously described. The pipets listed in the instrument-maker's catalogues are generally made to deliver 50 grams, in which case, by adding the 50 grams so delivered to a 500-ml. flask and making to the mark with water, each 50 ml. will contain 5 grams of juice, whereas the use of a 250-ml. flask will give 10 grams per 50 ml.; by delivering two volumes of juice from the pipet, 20 grams per 50 ml.; a second pipet calibrated to deliver 60 grams may be used with a 200-ml. flask to give 15 grams, and two volumes to 200 ml. will give 30 grams per 50 ml. Many laboratories carry glucose pipets calibrated for 20, 30, 40, and 80 grams, using a 200-ml. flask to obtain the dilution desired. By means of a Spencer glucose pipet make up a solution of the deleaded filtrate of such strength that 50 ml. will contain 5, 10, 15, 20, or 30 grams of the juice, depending on the amount of reducing sugars present. Taking 50

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[10] *Ind. Eng. Chem.*, **13**, No. 10, 925 (1921).

[11] *Facts About Sugar*, **23**, 806 (1928).

ml. of this filtrate, proceed with the Herzfeld method of gravimetric glucose, determining the copper as cupric oxide or as copper by the Votoček-Wedderburn method, or as both, using one method as a check on the other. From the weight of copper the reducing sugars may be found by reference to the table on p. 790, according to the percentage of sucrose in the juice.

The Munson and Walker method is recommended in the Hawaiian official methods for the determination of reducing sugars in juice.

640. Eynon-Lane Method. The Eynon-Lane volumetric method (Sec. 506) is recommended in many countries and has the approval of the Committee on Uniformity of the International Society of Sugar Cane Technologists. The solution of juice should be so prepared that 25 to 40 ml. (125 to 200 mg. of reducing sugars) will be used in the titration The glucose pipet described above is convenient for "weighing" the amount of juice taken. The *Queensland Laboratory Manual* recommends the electrometric modification (Sec. 509) for juice determinations, and this will undoubtedly prove a simple method for routine when practice has been attained.

It is evident that the volumetric method may be used either with lead-preserved juices, deleaded according to the directions given by Harris with powdered oxalic acid, or with the deleading solution of McAllep and Cook following clarification with neutral lead acetate, or with solutions clarified with kieselguhr only and decalcified with dry sodium oxalate.

The calculations for the Eynon-Lane method are shown in the following example.

Example. With juice at a Brix of 16.0° and polarization of 14.0, a solution has been prepared (with glucose pipet as described above) to contain 25 grams of original juice in 100 ml. of the prepared solution for titration. This solution therefore contains $14.0 \times 25 = 3.5$ grams of sucrose per 100 ml. Titration with this solution requires 28.5 ml. to precipitate the copper in 10 ml. of Fehling's solution. From Table 47, p. 794, by interpolation between the 3-gram and 4-gram column for sucrose concentration it is found that 100 ml. of the solution contains 171 mg. of invert sugar. Therefore

$$\frac{100 \times 171}{1000 \times 25 \text{ (mg. of juice)}} = 0.684\% \text{ reducing sugars in the juice (as invert sugar)}$$

641. Determination of the Ash. *Carbonated Ash or Normal Ash.* The carbonated ash is usually determined only in research work and not in commercial analysis. Dry 10 grams of juice in a weighed platinum dish, and proceed to incinerate the residue as given in Sec. 539.

Sulfated Ash. Dry 10 grams of the juice in a shallow tared fused silica or platinum dish. Proceed as in Sec. 538. Most sugar laboratories continue to deduct one-tenth from the weight of the sulfated ash to allow for the increase in the weight of ash due to the formation of sulfates instead of carbonates, although the official methods of the Association of Official Agricultural Chemists specify that the weight as found shall be taken as sulfated ash. Where the practice is to deduct one-tenth the calculation becomes: Weight of sulfated ash × 9 = per cent ash. If the Association of Official Agricultural Chemists' method is followed:

Weight of sulfated ash × 10 = Per cent sulfated ash.

It should be emphasized that it makes no difference in control work whether the deduction is made or not, provided that one method or the other is followed consistently throughout the entire control.

It is usually more convenient to measure 10 ml. of the juice for the ash determination rather than to weigh 10 grams. In such cases the calculation is modified by dividing by the specific gravity of the juice.

642. Electrometric Ash. The ash in juice may be determined electrometrically by the methods given on p. 450.

643. Acidity of the Juice. *By Titration with Phenolphthalein.* This test was formerly much used in the control of clarification but has been largely superseded by pH for this purpose. The acidity of raw juice is determined in Louisiana as a measure of the acceptability of the cane at the mill, e.g., following a freeze, burning, or the like. Normal cane juice is always acid. The acidity is generally expressed in terms of the number of cubic centimeters (or millimeters) of deci-normal alkali (usually NaOH) required to neutralize 10 ml. of the juice, phenolphthalein being the indicator most commonly employed. It is necessary to state what indicator is used since the end point of different indicators is not the same. Neutrality to phenolphthalein, for example, is definitely alkaline to litmus, to cite the two commonest indicators in sugar work.

To carry out the titration, pipet 10 ml. of the juice into a porcelain dish or casserole, dilute with about 25 ml. of water, which is neutral to phenolphthalein, add two to three drops of neutralized phenolphthalein solution (Sec. 897) and then run into the mixture, from a buret, tenth normal sodium hyroxide solution (Sec. 913) until there is evidence of a pink color. Record the number of milliliters of the alkali used as the acidity of the juice. With dark-colored juices it is sometimes difficult to distinguish the change in color promptly. As an aid to the eye place alongside the dish in which the titration is to be made a duplicate one containing the same juice and water mixture. If this is nearly neutralized with the caustic solution it will then be possible, by comparison with this blank, to note the first approach of the pink color as the alkali is added to the test sample.

The Committee on Uniformity of the International Society of Sugar Cane Technologists recommends that the acidity be expressed as milligrams of CaO per liter of juice. For this reason $N/2.8$ or $N/28$ is suggested as the standard alkali so that the number of milliliters of alkali used is directly convertible to parts of CaO.

pH *of the Juice.* This is the most exact and valuable method of recording the acidity or alkalinity of juices. For a discussion of the theory and usefulness of the pH numbers, consult Chapter 23. It must be clearly understood that acidity of the juice by titration and pH will bear no constant relation since the former is total acidity, while the H-ion concentration (pH) is the *effective* acidity.

Fort and McKaig[12] show that attempts to correlate acidity and pH values with phosphate content or ash were unsuccessful, and they conclude that "the pH value and acidity in cane juice are the result of so many factors that apparently no simple relationships exist."

The pH determination may be made by the spot method (p. 463) or preferably by dilution with neutral water and comparison with standards in tubes as described in Sec. 559. The dilution for routine work should be fixed, say one part juice to five water. The pH is not generally made on the untreated raw juice but on the mixed juice, after the addition of lime, to determine the correct reaction for clarification (p. 80) and for this purpose bromthymol blue (6.0–7.6 pH) and cresol red (7.2–8.6) will cover all the desired ranges. The method outlined on p. 463, using diluted dye for the dilution of the juice, simplifies the procedure, particularly if workmen are called upon to make the test.

Simple electrometric apparatus of various makes (Sec. 569) using the glass electrode are now on the market and are not too expensive for the ordinary sugar house laboratory. The method is much more accurate and quite as rapid as the colorimetric methods and is to be preferred, at least for checking purposes if not for continuous routine work.

644. Phosphoric Acid (Phosphate) Determination. The importance of the phosphate content of the juice has been discussed in the chapter on clarification (p. 90), and the following colorimetric determination is prescribed in Queensland (Australia), as a daily test on the composite sample of mixed or first expressed juice preserved with mercuric chloride.

Reagents. (*a*) Ammonium Molybdate-Sulfuric Acid Solution. Twenty-five grams of ammonium molybdate is dissolved in 200 ml. of warm water and filtered. Two hundred and eighty milliliters of arsenic- and phosphorus-free concentrated sulfuric acid is diluted to 800 ml. When both solutions are cool, the molybdate solution is added slowly, with shaking, to the sulfuric acid solution. Finally when the mixture has attained room temperature, it is diluted to 1 liter.

(*b*) Stannous Chloride Solution. This is prepared by dissolving 25 grams of stannous chloride in 1 liter of dilute hydrochloric acid solution (1 volume concentrated HCl plus 9 volumes water). The solution should be stored in a glass bottle with delivery cock near the bottom to provide the solution in drops. It should be protected from the air by floating a layer of white mineral oil about 5 mm. thick over the surface.

(*c*) Standard Phosphate Solution. A stock solution is prepared by dissolving 0.1916 grams of recrystallized potassium dihydrogen phosphate (KH_2PO_4) in water, and diluting to 1 liter. This solution contains 100 parts P_2O_5 per million. A second solution, for use as a test standard, is prepared by diluting

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[12] *U.S.D.A. Tech. Bull. 688,* p. 10.

50 ml. of the stock solution to 500 ml. This contains 10 ppm. of P_2O_5. As this solution is likely to decompose during storage because of microorganisms, the standard dilution should be prepared at frequent intervals.

The standard for comparison is prepared by diluting 5 ml. of phosphate solution to 95 ml. with distilled water, adding 4 ml. of ammonium molybdate-acid solution, and mixing in an Erlenmeyer flask. When six drops of stannous chloride solution are added the characteristic blue color is developed. After standing ten to twelve minutes the color begins to fade; it may be restored by the addition of one drop of stannous chloride solution, and it will then be permanent for another ten to twelve minutes.

It is found that the inorganic phosphate content of cane juice ranges from 0.002 to 0.05 per cent. The volume required for the test will vary accordingly, but experience within a given mill area soon shows what concentration may be expected. Taking an average of 0.02 gram P_2O_5 per 100 ml., a convenient aliquot of first expressed or mixed juice for the test will be 0.25 ml. This is best obtained by diluting 5 ml. of juice to 100 ml. and taking 5 ml. of the dilute solution for the determination. This volume is further diluted to 95 ml., and reagents are added as described for the standard.

The intensity of the blue color is determined against the standard by the use of a colorimeter. Where this is not available a pair of Nessler tubes is quite satisfactory.

Calculation. 5 ml. juice diluted to 100 ml., and 5 ml. taken for the test.

Reading. 10 cm. standard equivalent to 8.6 cm. test solution. One hundred milliliters standard contains 0.05 mg. P_2O_5. Therefore 100 ml. test solution contains

$$\frac{10}{8.6} \times 0.05 = 0.0581 \text{ mg. } P_2O_5$$

This represents 0.25 ml. of juice; therefore 100 ml. of juice contains

$$\frac{100}{0.25} \times 0.0581 = 23.2 \text{ mg.} \quad \text{or} \quad 0.023\% \ P_2O_5$$

Where the method is employed for clarified juice the volume of juice required will, of course, be increased proportionately.

A modification of this method, using a Helige comparator (p. 464) with a color disk like the one for determining P_2O_5 in soils is followed in Mauritius and has been described by Olivier and others,[13] and a simplified modification [14] is suggested by Beater.

645. Phosphoric Acid Determination. *Uranium Acetate Method.* This method may be used for more occasional determinations or as a check on the preceding method.

a. Dissolve 35 grams of chemically pure uranium acetate in distilled water. To this add 50 ml. glacial acetic acid and make up to 1 liter.

[13] *Intern. Sugar J.,* June, 1934.

[14] *Intern. Sugar J.,* January, 1938, p. 30.

b. Dissolve 100 grams of chemically pure sodium acetate in distilled water and add 50 ml. glacial acetic acid. Make up to 1 liter.

Add 1 ml. NH_4OH to 100 ml. of juice, acidify with acetic acid and add 10 ml. of solution *b*. Titrate with solution *a*, using powdered potassium ferrocyanide on a drop reaction plate as an indicator. The solution usually settles sufficiently to allow a small portion of clear liquor to be removed for the end point determination. When the end point has been reached the liquor gives a brown precipitate of uranium ferrocyanide when brought into contact with potassium ferrocyanide. Solution *a* should be standardized against an accurately weighed sample of chemically pure tricalcium phosphate, which has been dissolved in nitric acid, precipitated by adding a slight excess of ammonia and redissolved in a moderate excess of acetic acid.

The greater portion of the uranium solution should be added in the cold but the titration should be finished at 90° to 100° C. The amount of P_2O_5 present is calculated from the milliliters of uranium solution used.

646. Lime Salts in Clarified Juice, Etc. A rapid method by titration with standard soap solution is given by Kreke and Dekker,[15] but for more exact determinations precipitation as oxalate and titration with permanganate are recommended. The juice or solution is acidified with acetic acid, purified kieselguhr is added, and the mixture is boiled and filtered. The filtrate is again brought to boiling, the calcium is precipitated with an excess of hot ammonium oxalate solution, and the analysis is completed as usual.

647. Analysis of Clarified Juice. The analysis of the clarified juice is made by the same methods as that of the normal juice. If the carbonation process is used (see Sec. 134), the juice must receive an additional treatment with carbonic acid, after the first carbonation and before the analysis, to precipitate all the lime it contains.

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[15] *Intern. Sugar J.*, February, 1931, p. 73.

CHAPTER 28

ANALYSIS OF THE SIRUP, MASSECUITES, AND MOLASSES

SIRUP

648. Analysis of the Sirup. The tests usually required in the examination of the sirup from the evaporators are the density and the polarization. In special investigations or in tracing inversion, reducing sugar determinations are necessary, and the sucrose should then be determined by Clerget's method, p. 401. pH determinations are now usually made at intervals of from one to three hours as a routine procedure. The colorimetric method, diluting 1 : 3 or 1 : 5 as directed for the juice (p. 462), may be used for this test, or the electrometric method may be used without dilution.

The normal weight of sirup should ordinarily be used for the polarization, and this should be weighed and not measured with a sucrose or other pipet. The solutions for the polarizations should be acidulated with acetic acid after clarification with lead subacetate but before filtration.

For the usual factory requirements, the sirup may be analyzed by the methods described for the juice, except that the portions for the tests must be weighed and not measured.

MASSECUITES AND MOLASSES

649. Determination of the Density. The determination of the density of massecuites and heavy molasses presents certain difficulties which cannot readily be avoided and which compel the acceptance of numbers that are little more than close approximations, according to the methods used.

As has been explained, the degree Brix of a solution of sucrose is the percentage by weight of the pure sugar dissolved in it. But in the sugar industry it is usually considered to be the percentage by weight of solid matter, whether sugar or not, in solution. This implies that the solids other than sugar, the non-sucrose, are of the same specific gravity as cane sugar. This is practically true for the carbohydrate bodies, but it is not true for the inorganic salts which are associated with the sugars in the massecuites and molasses. These salts having such high specific gravity, as compared with the carbohydrates, influence the density determinations in a very marked degree. The organic non-sugars are also of higher specific gravity than sugar.

Since the ratio of non-sucrose to the sucrose increases with each stage of the manufacture, as commercial sugar is removed, the difference between the apparent percentage of total solids, as indicated by the hydrometer, and the true percentage, as ascertained by actually drying the material, becomes larger.

The results by refractometer are much closer to true dry substance than the figures obtained by hydrometer, and in the products now under discussion refractometer Brix is usually intermediate between Brix and dry substance.[1] It is evident from these remarks that calculations involving Brix in massecuites and molasses must be accepted with caution and then only for comparative purposes when similar conditions of analysis are maintained.

Furthermore, the density (Brix) of a solution calculated from spindling at one dilution is different from that calculated from the hydrometer number ascertained at another dilution. Thus, for example, if one part of a final molasses is dissolved in two parts of water and this solution is spindled, the Brix of the molasses calculated from this spindling will be higher than it would be had one part of the molasses been dissolved in only one part of water. This difference is partly due to the contraction of the solution of sugar on dilution with water and partly to a similar contraction of the solution of the salts in the molasses. This difference would be observed even though one were dealing with a pure sucrose solution instead of molasses. Obviously massecuites and molasses are too dense to be directly spindled, hence one must accept numbers obtained by dilution and spindling that are at best only comparative. The true solids of a final molasses may be from 5 to 10 per cent below the numbers indicated by dilution and spindling.

650. Dilution at Which Brix Should Be Made. This is a controversial subject, particularly in the eastern hemisphere, and may become of greater interest in the western hemisphere if the International Society of Sugar Cane Technologists methods for available sugar are adopted here (Sec. 809) with the gravity purity which is sucrose (by Clerget method) divided by Brix. Noël Deerr, who first introduced the idea of gravity purity, said that "to be strictly comparable, gravity solids (Brix) determinations should be made in solutions of the same non-sugar concentration."[2] For low-test products such as molasses, this would involve such high dilution that the resulting multiplication would introduce large errors. As a compromise, the Hawaiian methods for Brix prescribe dilution to a density about the same as that of the mixed juice (1:5 or 1:6). The Java method for molasses is 1:10 dilution, which requires the use of a pycnometer.

The International Society of Sugar Cane Technologists, "System of Factory Control," says (p. 47):

> In practice, the possible error in the gravity purity of final molasses caused by dilution to juice density instead of to equal concentration of non-sugars amounts to only 0.2 which is less than the permissible possible errors on Brix reading and polarization.

If the concept of gravity purity is to be introduced into the control methods (as recommended by the International Society) the dilution to approximate juice density should be done before determining the Brix of molasses. If apparent purities are used as is general in Louisiana, Cuba, and Puerto Rico, the Brix by double dilution as described below will be continued. The double

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[1] See Browne-Zerban, *Sugar Analysis,* New York, 1941, p. 107.

[2] *Chemical Control,* Hawaiian Association, 1931, p. 34.

dilution method is official for most commercial transactions and is therefore generally necessary whether the higher dilution is used or not.

651. Brix by Double Dilution. Dissolve a weighed quantity of the material in an equal weight of distilled water. Transfer a portion of the solution to a cylinder and determine its degree Brix. Correct the degree Brix for the temperature error as described on p. 433, and multiply the corrected number by 2 to ascertain the degree Brix of the material. This is the customary commercial and factory method. The true percentage of solids is usually several degrees lower than the apparent number obtained by spindling this 1 : 1 solution of the massecuite or molasses.

For density determinations of massecuites by this method hot water must be used and great care should be taken to dissolve all crystals. After complete solution the mixture is cooled and the weight is adjusted with cold distilled water to exactly twice the weight of massecuite taken. A small motor-operated stirring device has been found convenient for mixing double-dilution solutions. Before using the hydrometer all air must be removed either by suction or by allowing the solution to stand for twenty minutes.

652. Solids by Drying. This may be determined by drying on pumice stone or on quartz sand preferably under vacuum and at a temperature of 70° C. Many sugar laboratories use these methods at boiling-water temperature and atmospheric pressure. The heating period is usually fixed arbitrarily (say five hours) as constant weight at this pressure and temperature is not attainable with low-purity materials because of the decomposition of levulose.

The Spencer electric oven may be used conveniently for these materials (Sec. 524). There are indications that the very rapid evaporation in this oven may keep the material at a relatively low temperature until drying is nearly complete, when levulose and other organic materials are not so readily decomposable. Moisture in strained honey (which is very high in levulose) was determined in this oven[3] with no appearance of decomposition after twenty minutes' heating.

653. Relationship between Brix and Solids by Drying. The salts present in low-purity products increase the specific gravity and therefore affect the Brix by hydrometer as explained above. Many investigators have suggested methods for converting Brix readings into total solids, most of the calculations involving the percentage of ash. As an example of this type of formula, Sylmans[4] found that the total solids in Java molasses could be estimated quite closely by multiplying the sulfated ash (without deduction) by a factor and subtracting this product from the Brix (determined on 1 : 10 dilution and multiplied by 10). The factor found was 0.72 for defecation factories, 0.74 for carbonation factories, and 0.86 for sulfitation factories. He concluded that the results by the calculation are closer to true values than dry substance by refractometer.

It is evident that such a formula must specify the method of Brix determination and what is meant by ash (whether sulfated or carbonated).

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[3] Meade, *Ind. Eng. Chem.*, **13**, No. 10 (1921).

[4] *Intern. Sugar J.*, March, 1933, p. 116.

Davies[5] worked out a series of formulas for specific factories, showing relationship between Brix, refractometer solids and solids by drying, by "direct correlation" and "non-sugar correlation" which gave satisfactory results.

Calculations of this type are valuable mainly for checking purposes. If a particular factory or refinery works out a relationship on the basis of ash or non-sugars, it will serve to indicate errors in control work or unusual conditions in the composition of molasses. It is obvious that if there were a direct and calculable difference between Brix and total solids in such products as molasses, it would not be necessary to do dry substance at all, and the Brix would serve for all purposes. Unfortunately such a fixed and unvariable relationship does not exist.

654. Brix or Solids by Refractometer. The method of using the refractometer has been described on p. 444. This method gives results that approximate more closely those by drying than do the determinations by hydrometer.

If the material contains crystals of sugar these must be dissolved, since the refractometer indicates only the solid matter that is in solution. When solution is employed the calculation is made as is indicated farther on in this paragraph. Dilution methods involve the contraction error of similar methods by spindling. The error may be reduced by working with very concentrated solutions. If a volume of water is added to a molasses solution, for example, the volume of the mixture is not the sum of the volumes, but is a smaller number, and the concentration is higher. It is, therefore, necessary as in hydrometer methods that the same conditions be observed in order that the results may be comparable.

In the event of testing a highly colored material, the method of Tischtschenko may be used. Mix the material with an equal weight of a solution of pure sucrose of known composition and of as high concentration as is practicable and determine the refractive index. Ascertain the percentage of solids in the mixture by means of the table on p. 747. The percentage of solids in the material is ascertained by deducting the per cent of solids in the sucrose solution from twice the solids in the mixture. The principle of the method of calculation for other mixtures is the same as that of the dilution formulas.

If necessary to dilute the material with water, the calculation is made as follows:

Let x = the required percentage of solids (Brix);
W = weight of the material used;
w = weight of the diluted solution of b Brix.

Then

$$Wx = bw \text{ and } x = bw/W.$$

655. Specific Gravity. A method originally devised by Sidersky applicable to molasses but not to grained strikes has been modified for use by

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[5] *Intern. Sugar J.*, October, 1932, p. 402.

the U.S. Treasury Department and adopted by the International Commission for Uniform Methods of Sugar Analysis, London, 1936.[6]

A special 100-ml. volumetric flask with a neck of approximately 8 mm. inside diameter shall be used. Weigh the flask empty, and then fill it with molasses, using a long-stem funnel reaching below the graduation mark, until the level of the molasses is up to the lower end of the neck of the flask. The flow of molasses may be stopped by inserting a glass rod of suitable size into the funnel so as to close the stem opening. Remove the funnel carefully to prevent molasses coming in contact with the neck, and weigh the flask and molasses. Add water almost up to the graduation mark, running the water down the side of the neck to prevent mixing with the molasses. Allow to stand several hours or overnight to permit the escape of bubbles. Place the flask in a constant-temperature water bath, preferably at 20° C., for a sufficient time for it to reach the temperature of the bath, then make to volume at that temperature, with water. Weigh. Reduce the weight of the molasses to vacuo and calculate the density.

Example. Weight of flask, 37.907 grams. Weight of flask and molasses, 167.148 grams. Weight of flask, molasses, and water 174.711 grams.

167.148 − 37.907 = 129.241 = Weight of molasses in grams (in air with brass weights)

174.711 − 167.148 = 7.563 = Weight of water in grams (in air with brass weights)

Calculating volume of water from weights in air at 20° C., divide the weight of water in air by the weight of 1 ml. of water in air at 20° C. (Table 9, p. 696),

$$\frac{7.563}{0.99718} = 7.584 \text{ ml.}$$

Volume of flask at 20° C., 100.060 ml.
7.584 ml.

Volume of molasses 92.476 ml.

To reduce weight of molasses to vacuo:

$$\text{Volume of brass weights } (8.4 = \text{Density of brass weights}) = \frac{129.241}{8.4} = 15.4 \text{ ml.}$$

Volume of molasses (approximate) = 92.5 ml.
Volume of weights = 15.4 ml.

Net volume of air displaced = 77.1 ml.

77.1 × 0.0012 = 0.093 gram (buoyancy correction to be added to weight of molasses)

Weight of 1 ml. of air at 760 mm. at 20° C. = 0.0012 gram.

129.241
0.093

Weight of molasses in a vacuum = 129.334

$$d_{4^\circ}^{20^\circ} = \frac{129.334}{92.476} = 1.3986$$

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[6] *Bur. Standards Circ. C 440* (1942), p. 251.

656. Newkirk's Method.[7]

The difficulties met in the accurate determination of the density of molasses are due to (1) high viscosity, (2) included gases and (3) dissolved gases.

As a result of the high viscosity the use of hydrometers is prevented, and the thorough mixing or intermingling of the various components of the batch or sample of molasses is made difficult. Obviously, in materials of this character a representative sample can not be obtained without vigorous agitation, which causes entrainment or inclusion of large amounts of air. Finally, the viscosity retards the escape of the entrained or included air. The effects of included air upon the density of the liquor are too evident to need discussing. The removal of the dissolved gases before taking the density may in some cases be essential and in others not.

Investigations at this Bureau established the fact that the removal of gases was best effected by the use of a vacuum. In order to permit this to be done as readily and effectively as possible, an improved pycnometer was designed.

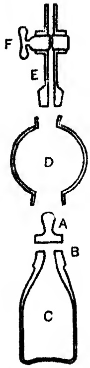

Fig. 179. Pycnometer for Molasses.

It consists of a bottle, *C* (Fig. 179), fitted with an enlargement at the top, *B*, ground optically flat, and closed with another optical flat, *A*. An expansion chamber, *D*, is ground to the bottle and fitted with a vacuum connection, *E*. To avoid loss of water due to evaporation under reduced pressure, the connecting tube is fitted with a stopcock, *F*, so that when the proper vacuum has been reached the apparatus can be closed off from the vacuum source. With this provision the volume to be filled with water vapor is very small, and the amount of water evaporated will be negligible. The bottle is so shaped as to have a smooth gradual slope to the top, so that the bubbles will rise with the least effort to the expansion chamber. The joints of the expansion chamber, vacuum connection, and stopcock are ground to an accurate fit.

In using the pycnometer, the expansion chamber, after lubrication of all joints with molasses, is placed on the bottle. The molasses to be analyzed is flowed into the bottle and into the expansion chamber until the latter is one-third full. The vacuum line is then connected and the pressure reduced until the gas expands to visible bubbles. The apparatus is immediately closed off by turning the stopcock, *F*, and the whole is placed in the thermostat for accurate work or in the balance case for control work. When all the bubbles have collected in the expansion chamber and the temperature has reached equilibrium, the volume is fixed with the plate after removing the expansion chamber. It is then wiped and weighed.

The densities are determined by correcting weights to vacuo and comparing to the weight of an equal volume of water at 4° C. in vacuo. The calculations may be carried out in the same way as in the example in the previous section.

657. Weight per Gallon of Molasses. This is a figure of great commercial value since molasses is sold both by weight and by the gallon, and the relationship between the two is frequently desired. The torsion balance shown in Fig. 180 was designed by H. J. Bastone for determining the weight per gallon. It is equipped with a beam for taring the weighing bottle and a

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[7] *Bur. Standards. Tech. Paper 161.*

second beam graduated in pounds per gallon from 10.80 to 12.05 in 1/100-lb. intervals. In an investigation of this instrument Snyder and Hammond[8] found it more accurate and convenient to fix the volume by sliding a glass disk over the flat polished top of the weighing bottle rather than by wiping off the stopper. After filling the bottle, the stopper was inserted and the glass disk was carefully slid over its top surface, thereby removing the surplus molasses. By this procedure only a very thin film of molasses remained between the disk and the top of the stopper. The calibration of the volume of the bottle as well as the direct determinations were made in the same manner. Their conclusion was as follows:

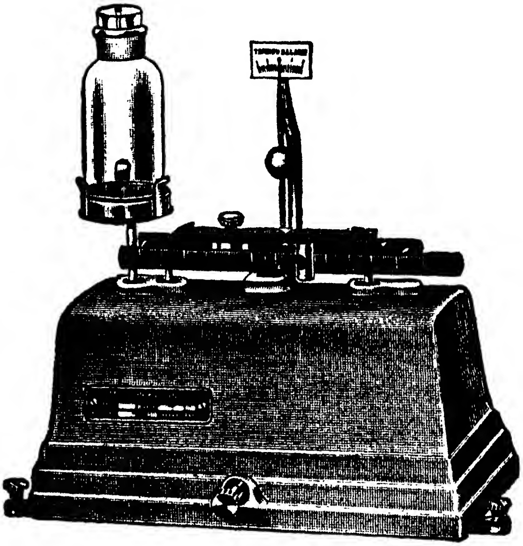

FIG. 180. Balance for Weight per Gallon of Molasses.

> The torsion balance seems entirely satisfactory for most determinations of weights per gallon of molasses provided the usual precautions are taken to allow the foam to subside and occluded gases to escape. It is obvious that the same precautions must be taken in any method of direct determination of density or weight per gallon of molasses. Any direct reading balance will be found useful in commercial work and routine testing as compared with other methods since results may be obtained without resort to the tedious calculations necessary in a density determination.

658. Weight per Gallon—Actual and Calculated. Because of entrained air, heavy molasses will never show an actual weight per gallon approaching that calculated from the density obtained in the laboratory. A large series of carefully controlled tests by William B. Saladin at Con-

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[8] *Bur. Standards Tech. Paper 345.*

stancia, Cuba, showed that the weight per gallon of recently pumped Cuban molasses is approximately 86 or 87 per cent of the air-free weight as determined from the density. These figures were obtained on molasses of about 88 apparent Brix, unchanged as it comes from the centrifugal machines. Settled Cuban molasses of the same type will weigh about 11.50 to 11.70 lb. per gal. Molasses that has been slightly reduced with water and heated before pumping to storage tanks will show weight-per-gallon figures much closer to those obtained from the true density. Refinery barrel sirups stored for considerable time in large tanks and filled into tank cars by gravity and allowed to settle twenty-four hours before measuring the gallonage average about 11.85 lb. actual weight per gallon as against an air-free weight of 12.15 determined in the laboratory. This same type of material *pumped* into tank cars shows an average of 11.72 lb., the difference being the entrained air due to pumping.

Frothing or foaming due to the formation of CO_2 in the molasses (Sec. 286) by the so-called froth "fermentation" will also disturb the weight-per-gallon figures. It is obvious that a foaming molasses will weigh less per gallon than one which contains no occluded gas or air.

659. Weight of a Unit Volume of Massecuite. A modification of Sidersky's method described in Sec. 655 may be used to ascertain the weight of a certain volume of massecuite. The selection of the unit volume will, of course, depend upon whether the cubic foot or gallon or metric measures are used.

A device for making this measurement is shown partly in section in Fig. 181. This consists of a cylindrical vessel of any convenient size and preferably of metal. The rim of the cylinders should be ground true, a strip of metal, *CC'*, should be provided, which extends from side to side of the cylinder and supports a glass tube drawn to a capillary, as shown in *TT'*. Pins *PP'* in the rim of the cylinder and fitting in corresponding holes in the strip of metal insure replacing the latter always in the same position. Fill the cylinder to approximately the point *w* with massecuite, place the capillary tube in position, then run in water from a buret very cautiously until the tube is reached. The instant the water reaches the tube it rises some distance into it by capillarity. This may be more plainly seen if colored water is used. A previous determination of the volume of the cylinder to the bottom of the tube should be made with water. With this volume and that of the water required to complete the volume with the massecuite, that of the massecuite is readily ascertained and may be compared with the weight of the material.

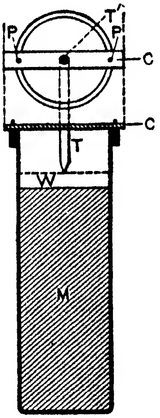

Fig. 181. Apparatus for Weighing a Unit Volume.

In making this test the massecuite should be of the temperature at which the measurements of this product are to be made in the factory, and correction for the expansion of the cylinder should be applied.

It is evident that this method cannot be used where vibrations of machinery are felt.

660. Routine Purity Determinations. For the ordinary purposes of the factory, for the control of the various processes of manufacture, especially the vacuum pan and crystallizer work, apparent purities, i.e., polarization divided by Brix, are all that are needed. True sucrose and solids by drying are not required for any of this work. The most important point in connecnection with this work is to adopt certain conditions of analysis and adhere to them with all similar materials, in order that the results may be comparable.

The ordinary water purity which is so generally used in refinery practice is applicable for this purpose. Dissolve the massecuite or molasses in water and dilute the solution to any convenient Brix between 15° and 20°. Mix thoroughly, allow to stand until all air is removed (or use vacuum connection as described on p. 433 to aid air removal), and determine the Brix with a standardized hydrometer, making the temperature correction and hydrometer correction as directed in Sec. 517. After Brixing, add to a portion of the solution (measured approximately) sufficient Horne's dry lead subacetate to clarify; then add a spoonful of dry kieselguhr and filter through a rapid filter paper. To 50 ml. of filtrate in a 50–55-ml. flask add dilute acetic acid until the contents of the flask are acid to litmus, then make to the 55-ml. mark. Polarize.in a 200-mm. tube and add one-tenth to the reading to correct for the extra dilution. If the solution is so diluted that the corrected Brix falls between 12° and 20°, Horne's expanded table on pp. 766–784 may be used to obtain the coefficient of purity. If the corrected Brix does not fall within the range of the table, the purity may be calculated by using the table of factors calculated by Rice from Casamajor's formula modified for hydrometers standardized at 20° C.

In a modification of the above, which obviates acidification and one-tenth dilution in a separate flask, the lead salt is added as directed above and well shaken to make sure that the clarification is correct. Then dry powdered oxalic acid is added a little at a time until the leaded solution is faintly acid to litmus, after which kieselguhr is added, and the whole is thoroughly mixed by violent shaking and filtered. The filtrate is polarized in a 200-mm. tube, and the purity is determined as before.

The purity is all that is usually required in these tests. As the amount of lead used has considerable effect on the purity of low-grade material, it is well to use the same amount for tests on similar grades. For higher-purity materials than molasses and factory massecuites no acidification is necessary. (See directions for the use of Horne's table, p. 766.) It should be noted that because of the large dilution of the material, the purity is lower than when determined by double-dilution methods.

661. Determination of Polarization. For the purpose of comparing the commercial sugar produced in the various periods, or runs, of the crop as well as determining the total loss of sucrose (or pol), it is essential that the final or commercial molasses samples be analyzed to obtain correct results as nearly as the processes of analysis will permit. Both polarization and true sucrose (Clerget) are required for these records. The best and most convenient method is to make these determinations on the 1 : 1 sample made up for the Brix by hydrometer (Sec. 651). Average daily figures for the Brix polarization and purity of the various grades of massecuites and molasses (besides final molasses) are also generally recorded, and these are determined by the same methods.

For the polarization, weigh one normal weight of the 1 : 1 solution into a 200-ml. flask (or one-half normal weight of undiluted material if preferred), add a minimum of 54° Brix lead subacetate solution to effect good clarification, make to the mark, shake thoroughly, and filter. Place 50 ml. of the filtrate in a 50–55-ml. flask, and acidify with dilute acetic acid, making to the mark with water. A solution of acetic acid of such strength that 5 ml. will just acidify the 50 ml. of filtrate may be prepared for routine tests. The amount of lead for a given grade of material need not be varied greatly, if at all, from day to day, and for routine work it is well to prescribe limits for the quantity to be added because of the effect of an excess of lead on the results. Polarize the acidulated solution in a 200-mm. tube and multiply the reading by 4.4 for the polarization.

For dark-colored final molasses the normal weight of the 1 : 1 solution in a 300-ml. flask should be used, in which case the polariscope reading is multiplied by 6.6. This multiplication increases any error in reading, but it is more accurate than attempting to read a darker solution such as would be obtained with less dilute material.

662. Sucrose by Clerget. This figure is rarely determined on any low-grade material except final molasses, so the directions here given are for that grade only.

The Jackson-Gillis method IV has been officially adopted by the International Commission for Uniform Methods (London, 1936) and by most other governing bodies (p. 403) and therefore should be the method employed. Weigh two normal weights of the 1 : 1 solution (or one normal weight of the original molasses if preferred) into a 300-ml. flask, make to the mark, and mix. Pour the solution out in a cylinder, add sufficient Horne's dry lead to clarify, avoiding an excess. A weighed amount can be prescribed for routine work, 6 to 8 grams being sufficient for cane molasses. Mix by violent shaking and filter. Deleading is not essential. Pipet two 50-ml. portions of the filtrate into two 100-ml. flasks and proceed as directed in Sec. 488. If the Walker method of inversion is used, 2 ml. of the acid used for inverting should be added to the cold solution before inversion procedure is started to take up excess lead. Inversion for twenty-four hours at room temperature (about 28° C.) is preferable if time permits.

A small amount of zinc dust may be added to the inverted solution as described on p. 401. This is permissible according to the International Society of Sugar Cane Technologists but decolorizing carbons should never be used.

Polariscopic readings (both direct and invert) should be the average of several concordant observations. Multiply these average readings by 6 to obtain the values for P and P'.

663. Calculation of the Sucrose. The Clerget formula for the Jackson-Gillis method IV as given by the Bureau of Standards,[9] using the concentration factor of 0.0794 as recently determined by Jackson and McDonald is

$$S = \frac{100(P - P')}{132.63 + 0.0794(m - 13) - 0.53(T - 20)}$$

Where $P - P'$ are the direct and invert readings multiplied by 6 as figured above, m is the grams of solids in 100 ml. of the solution and $0.53(T - 20°)$ is the temperature correction. Calculation will show that the expression $132.63 + 0.0794(m - 13)$ for molasses of usual density (76 to 84 per cent solids) varies from 131.86 to 131.89, so that the calculation may be dispensed with, and the figure 131.88 may be used for all ordinary molasses without appreciable error. The temperature correction $0.53(T - 20)$ can be calculated or may be obtained from the table on p. 796.

Example.

Direct reading $+5.6 \times 6 = 33.6\ (P)$
Reading after inversion $-2.3 \times 6 = -13.8\ (P')$
$47.4\ (P - P')$

Temperature = 24.3° C.

Factor 131.88
Temperature correction (Table 49) 2.28
129.60

$$\text{Sucrose} = \frac{47.4}{129.60} \times 100 = 36.57$$

Using the Jackson-Gillis table (p. 796) which is based on $P - P'$ calculated to normality, i.e., divided by 3, gives 131.85 for the example above as the divisor to which the same temperature correction may be applied (2.28) = 129.57. This divided into $P - P' = 36.58$.

As will be seen, the two methods of calculation give results very close to each other. This is partly because of the use of Jackson and McDonald's higher concentration factor in the formula based on solids concentration. For practical purposes it therefore makes little difference which method is used since the accuracy of a Clerget determination in molasses is at best ±0.20 per cent.

664. Determination of Reducing Sugars. The amount of molasses or massecuite taken depends upon the percentage of reducing sugars present.

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[9] *Bur. Standards Circ. C 440*, p. 155.

Convenient weights for final molasses are 5 grams in 500 ml. if the invert sugar exceeds 26 per cent; 7.5 grams to 500 ml. for invert sugar between 26 and 18 per cent, and 10 grams to 500 ml. for invert sugar between 18 and 10 per cent. These give respectively 0.25, 0.375, and 0.5 gram in the 50 ml. to be used for the test. Prepare the solution by adding kieselguhr and 0.25 gram dry sodium oxalate after making to the mark (see p. 411). The addition of the oxalate is recommended to remove the lime salts which lower the copper-reducing power. Filter and take 50 ml. of solution, determining the reducing sugars gravimetrically by the Herzfeld method (Sec. 501). The copper precipitate may be oxidized to cupric oxide, or reduced to metallic copper by the Votoček-Wedderburn method (Sec. 502), or determined in both ways as a double check. The percentage of reducing sugars as invert sugar is found by reference to Rice's expanded Meissl-Hiller table, p. 785.

The solution may also be prepared by taking double the proportions indicated above, clarifying with neutral lead acetate (3 ml. for each 10 grams of molasses), filtering, and deleading and decalcifying an aliquot portion of the solution with 10 ml. of the mixed solution of disodium phosphate and potassium oxalate recommended by Cook and McAllep (p. 411). This method will remove any reducing non-sugars precipitable by lead which may be present.

The Eynon-Lane volumetric method (Sec. 506) may be used with either method of preparation of the solution, provided the lime is removed by means of oxalate. The electrometric modification advocated in Australia (Sec. 509) is recommended for molasses work. For most Cuban or Louisiana molasses the weight to be taken is 5 grams to 500 ml. (equivalent to 1.0 gram molasses per 100 ml.). The calculation is as follows. Assume that a molasses solution prepared as above gives a titration of 32.2 ml. to precipitate the copper from 10 ml. of Fehling's solution. From Table 47, p. 794, under 0.5 gram sucrose (the nearest approximation to the sucrose in 100 ml. of the solution), it will be found that a titer of 32.2 ml. represents 158.8 mg. of invert sugar per 100 ml. Therefore,

$$\frac{100 \times 158.8}{1000(\text{Milligrams of molasses})} = 15.88\% \text{ reducing sugars as invert sugar}$$

665. Determination of Ash. Using 3 or 4 grams of molasses (accurately weighed), proceed with the sulfated ash method as described in Sec. 538. The deduction of one-tenth for the sulfating may be omitted if the ash in all products is calculated in the same way. (See p. 448.)

666. pH Determination. Colorimetric pH determinations are out of the question on dark low-grade products such as molasses or solutions of massecuites. Electrometric methods may be employed, but in general the results are not of interest except in special investigations.

667. Analysis of High-Test Molasses. The analysis of inverted cane sirup called high-test molasses (Sec. 333) offers no special problems. The methods outlined for final molasses in the previous paragraphs may be used with minor modifications. As has been shown in Sec. 334 the Brix (double dilution), dry substance, and refractometer (corrected for invert sugar) will check each other closely because of the high total sugars and low ash content.[10] Clerget sucrose may be done by the Jackson-Gillis method IV (Sec. 488), or sucrose may be determined chemically by the Munson and Walker method before and after inversion (p. 430). The determination of ash is the same as for final molasses. Reducing sugars are best done by the Munson and Walker method (Sec. 504) using the table on p. 793 for the calculation. The column of the table headed "0.3 gm. total sugars" was calculated by Hammond especially for high-test molasses. In order to have 0.3 gram total sugars in the 50 ml. used in the test, 0.4 gram of molasses must be taken (since the high test is always close to 75 per cent total sugars), and this may be arrived at by weighing 4.0 gram of molasses in a 500-ml. flask, making to the mark, filtering through kieselguhr, and taking 50 ml. for the test. Then from the milligrams of copper found, the milligrams of invert sugar (in the "0.3 gm. total sugars" column) may be obtained, and this divided by 0.4 gram gives the percentage of reducing sugars as invert sugar. For example, the determination shows 355 mg. of copper which equals 188.9 mg. of invert sugar in the table. Then $188.9 \div 0.4 = 47.23$ per cent invert sugar.

Amino acids may be determined (though not as routine procedure) by the ninhydrin method of Ambler.[11] The method is also given in detail in the *11th Conf. of the Cuban Sugar Technologists* (1937), p. 51.

DETERMINATION OF SATURATION TEMPERATURES FOR MASSECUITES AND MOLASSES

668. The Saturascope. Reference has already been made in Chapter 11 on pan boiling to the determination of saturation temperatures of sugar solutions. Also the need for determining the temperature to which the massecuites may be heated without dissolving the crystals is referred to in the section "Crystallization in Motion" by Davies (p. 221). Several articles have been published on apparatus and methods for use in these determinations,[12, 13] and such methods are in general use in Australia and Hawaii. The apparatus used in Queensland[14] is called a saturation cell, but the Hawaiian cell is called a saturascope, and this name is general in the literature. This apparatus, which is routine equipment for low-grade control in most Hawaiian factories, is described as follows.[15]

[10] Zerban, Mull, and Martin, *J. Assoc. Official Agr. Chem.*, August, 1942, p. 763.

[11] *Intern. Sugar J.*, November, 1927, p. 382; December, 1937, p. 437.

[12] R. H. Harman, "Saturation Temperature of Sugar Solutions," *Intern. Soc. Sugar Cane Tech., Bull. 98*, Puerto Rico, 1932. Also *Intern. Sugar J.*, July, 1933, p. 261; August, 1933, p. 323.

[13] McCleery, *Rept. Hawaiian Sugar Tech.*, 1934, p. 133; *Intern. Sugar J.*, July, 1933, p. 279.

[14] *Laboratory Manual for Queensland (Australia) Sugar Mills*, p. 98.

[15] *Intern. Sugar J.*, May, 1936, p. 189.

The complete equipment consists of: (1) The "Saturascope" itself, which is an electrically heated air cell constructed of micarta or bakelite (Fig. 182), about 4 in. outside diameter and 1 in. deep, with glass cover and a glass-covered opening in the bottom. In the centre is a brass stage. The heating element is a single loop of nicrome wire, No. 28, and the temperature is controlled by a special rheostat to give a very close control.

(2) A thermometer: 0–220° F. in whole degrees.

(3) A rheostat: of special design.

(4) A microscope substage lamp with red glass light filter.

(5) Microscope cover-glasses No. 2, 22 mm. diameter.

(6) A compound microscope, using 30X eyepiece and 10X (16 mm.) objective, giving 300 diameters magnification.

The thermometer is inserted in the side of the "Saturascope" cell between the binding post terminals of the heating element, with the bulb tip almost touching the brass stage. The cell is placed on the stage of the microscope, and the microscope mirror is swung out of position with the microscope lamp placed directly below the microscope stage. The rheostat and the "Saturascope" are connected in series in a 110-volt circuit.

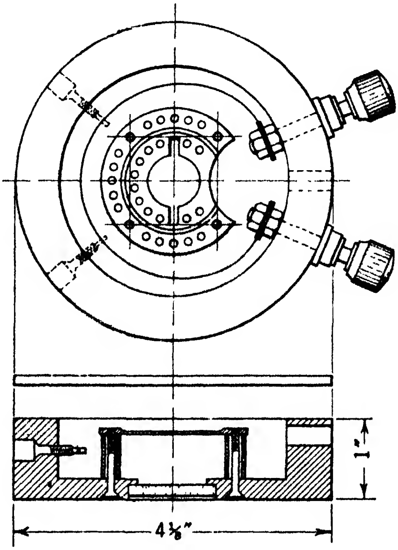

Fig. 182. Saturascope.

669. Operation. *Massecuites.* Place a small quantity of the massecuite between two of the three micro cover glasses and squeeze them together gently until it spreads out into a thin, uniform layer. Remove the glass cover of the saturascope and place the micro cover glasses containing the sample in the center of the brass stage. Replace the glass cover and plug in the substage lamp and the rheostat making sure that all the resistance of the rheostat is in the circuit.

Focus the microscope and move the saturascope slightly until a sharp edge or a broken section of a crystal with sharp points is brought into view, and adjust the focus until the part of the crystal chosen for observation is sharply defined.

Raise the temperature in the saturascope about 1° F. per minute by gradually cutting out the resistance in the rheostat until the crystal under observation starts to melt and note the temperature. The temperature at which the crystal begins to dissolve is the saturation temperature. Evidence that the saturation point has been reached can be indicated by various changes taking place in the appearance of portions of the crystal. If a sharp edge is chosen for observation, it becomes wavy or pitted. If a ragged edge on a broken portion of the crystal is being studied, the sharp points will round off and the edge smooth out.

After the saturation temperature has been determined as outlined above, repeat the test on a second portion of the sample. This time increase the temperature rapidly to within about 5° of the saturation temperature found in the first test. Then increase the temperature very slowly, about 1° every 3 minutes, until the crystal starts to melt. Record the temperature at which melting starts in this test as the saturation temperature. There should not be more than 1° to 2° difference in the two determinations.

Do not spread the material between the micro cover glasses so thin that there is not enough mother liquor in contact with the sugar crystals for them to start dissolving when the saturation temperature is reached. Spread the massecuite just thin enough so that a crystal may be selected and a sharp definition of its outline obtained.

Molasses. If the molasses contains a few stray grains that have passed through the centrifugal screens, proceed as with massecuites. If there are no sugar crystals present, add approximately 1 gram of a freshly dried low-grade sugar to about 100 grams of the molasses to be tested and mix in thoroughly. Place a small sample of this mixture between the micro cover glasses and proceed in the regular way. The temperature at which a crystal begins to dissolve will be the saturation temperature.

If secondary sugar crystals have come into the molasses on standing, it will first be necessary to dissolve these crystals. Place a small amount, 50 or 100 grams, of the molasses in a test tube or beaker and heat slowly to about 150° F. until all secondary crystals have dissolved. Cool rapidly to room temperature and add about 1 gram of freshly dried low-grade sugar. After thoroughly mixing, follow the regular procedure.

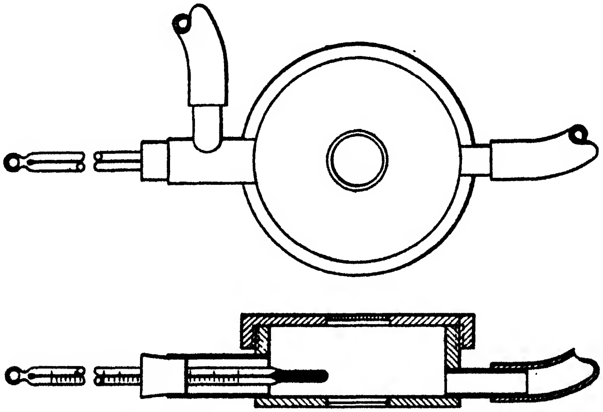

FIG. 183. Australian Saturation Cell.

670. Australian Saturation Cell. This apparatus consists simply of a flat hollow chamber through which water can be circulated, and provided with plane glass windows in the upper and lower faces. That illustrated in Fig. 183 is made of brass, and has been found to give very satisfactory results.

A small quantity of the molasses under examination is placed on the upper glass window, after which the procedure is much the same as that followed with the Hawaiian apparatus. The instructions note that precautions must be taken against evaporation in the case of hot samples and absorption of moisture from the atmosphere in the case of cold samples, by working quickly until the sample is safely under the cover glass. Furthermore, since high-grade products crystallize rapidly, the time between the preliminary and final determination should not be unduly prolonged by starting at a temperature too far below the saturation temperature.

CHAPTER 29

ANALYSIS OF SUGARS

671. Introductory. The analysis of raw sugars is a matter of great commercial and economic importance, and for this reason the methods have been standardized by the customs authorities of different countries and the bodies governing the buying and selling of sugars. In describing the methods that follow, accepted methods will be given without references, but where different methods are advocated by various authorities these will generally be noted.

672. Polarization. Weigh the normal weight of the sugar in a nickel capsule. Add sufficient water to moisten the sugar, waiting a moment for the water to penetrate the mass. The moist sugar may usually be poured slowly into a narrow-neck 100-ml. flask without difficulty. A little practice is necessary to accomplish this expeditiously. If difficulty is experienced, a special funnel of nickel (see p. 379) should be inserted and extend just into the body of the flask. The sugar may be readily washed through the funnel. The capsule, funnel, and neck of the flask must be washed with a jet of water. Care should be observed not to use more than about 60 ml. of water in these operations. The flask should be well cleaned before use (see p. 383) to prevent water from adhering to the neck. Dissolve the sugar by imparting a rotary motion to the flask. Hold the flask above the level of the eye occasionally to see whether all the crystals are in solution. It is essential that no sugar be left undissolved before proceeding to the clarification. A mechanical dissolver should be part of the equipment of all laboratories doing many polarizations. The Multer dissolver shown on p. 380 serves very well.

Having dissolved the sugar, add from 0 to 8 ml. of lead subacetate (54.3°), the quantity depending upon the grade of the sugar. White sugar requires no lead, but should usually receive a little alumina cream to facilitate filtration. High-grade centrifugals require from 1 to 2 ml. and low sugars, according to their grade, up to about 8 ml. of the lead solution. After mixing the sugar and lead solutions add about 2 ml. of alumina cream and complete the volume to 100 ml., washing down the neck of the flask. If foam interferes with this operation, it should be broken down with a drop of ether. The water should be of the temperature of the polariscope room, and the flask should be held by the upper part of the neck during the manipulations, to prevent warming the solution. If drops of water adhere to the neck of the flask they should be absorbed by a strip of filter paper.

Having finished these operations, cover the mouth of the flask with the thumb and mix the solution thoroughly by violent shaking. Pour the entire contents of the flask upon a folded filter in a stemless funnel. Rinse the receiver with the first portions of the filtrate and in all reject fully 25 ml. of the filtrate.

Hardin and Zerban[1] have shown that with perfectly dry filter paper (which is prescribed by the International Commission for Uniform Methods of Sugar Analysis) there is an adsorption of water from the sugar solution which may cause an increase in polarization of more than 0.1° V. By rejecting a full 25 ml. of the filtrate, this adsorption effect is largely, if not entirely, avoided. If very moist paper is used, the results may be lowered by the water given up by the paper to the solution. Most official methods now prescribe air-dried paper. The rejection of the first quarter of the filtrate is mandatory in the Official Methods of the Association of Official Agricultural Chemists. If the solution is not running clear after 25 ml. have passed through the paper, the test should be discarded. The filtration will be rapid and the filtrate bright if the proper amount of lead subacetate has been used. Should the filtrate be cloudy, the whole operation should be repeated, changing the quantity of lead rather than attempt to clear the solution by refiltration. The funnel should be so large that the paper will not project above it, and during the filtration funnel and paper should be covered with a clock glass. The effect of evaporation, if the funnel is not covered, is given in detail by Bates in *Bureau of Standards Bull. C 440,* p. 117.

The polarization is made as usual. The polariscope should be tested with a standardized quartz plate before and after use, and the reading should be corrected if need be. The cover glasses should be carefully selected and be free of flaws and scratches. The sections 477, 479, relative to the influence of clarifying agents and temperature, should be consulted.

The polarization is preferably made at 20° C. but as this is hardly possible in routine work and particularly in tropical countries, the reading must be corrected for temperature by the table given on p. 396. In this writer's experience the use of the temperature correction formula for raw sugars under Louisiana conditions has given results closely checking the polarizations of the New York Sugar Trade Laboratory at 20° C.

673. Dry Lead Clarification. Extended reference has already been made to the reasons for the use of Horne's dry lead subacetate for clarification (p. 390). Its use in polarizing raw sugars was made optional by the International Commission for Uniform Methods (Amsterdam, 1932). The London session (1936), however, modified this ruling (see p. 371) so that if the Ventzke scale, which is 0.1° lower than the so-called international scale (p. 370), is used then lead subacetate solution must be used. If the International scale is used, dry lead defecation should be employed. Put in another way, if the 26-gram normal weight is used on a polariscope with the Ventzke scale, lead solution is to be used (as described above), and if the normal weight of 26.026-gram is used, the dry salt is required. The use of the lead solution is

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[1] *The Planter,* **78,** No. 20 (1924).

generally easier, since the quantity required can be measured from a buret, so for the average laboratory the 26-gram weight with wet lead is recommended. Reference should be made to Sec. 455 on the subject of the correct normal weight and the correction of the polariscope scale.

If the dry lead is used (with the 26.026-gram normal weight as explained) sufficient should be added for clarification, avoiding an excess. One gram of the dry salt equals 3 ml. of the 54.3 Brix solution.

Also add a little dry sand to break up particles of lead precipitate that might occlude sugar solution. Close the flask with the thumb and shake it vigorously to mix its contents. Filter and polarize as has been described.

674. True Sucrose by Clerget's Method. Dissolve 52 grams in a 200-ml. flask. Add 4 to 6 ml. of lead subacetate solution (54° Brix) and 1 to 2 ml. of alumina cream, make to the mark, shake thoroughly, and filter. Pipet two 50-ml. portions into each of two 100-ml. flasks, and proceed with the Jackson-Gillis method IV (Sec. 488), which is recommended for sugar products. The deleading of the filtrate with oxalate may be dispensed with, the 50-ml. portions being pipetted from the lead-clarified filtrate. Calculate the Clerget sucrose according to the following formula:

$$S = \frac{100(P - P')}{132.63 - 0.0794(m - 13) - 0.53(T - 20)}$$

P' and P' are the direct and indirect readings multiplied by 2 to bring them to normality. Since the dry substance in a raw sugar is so close to 100 the concentration $0.0794(m - 13)$ can be disregarded. The temperature correction $0.53(T - 20)$ in which T is the temperature (to tenths of a degree) at which the readings were made may be calculated or taken from Table 49, p. 796. The formula therefore becomes

$$S = \frac{100(P - P')}{132.63 - 0.53(T - 20)}$$

If inversion is carried out at room temperature (see p. 404) as is preferable if time permits, the basis factor is 132.66 instead of 132.63.

675. Relationship between Polarization and Sucrose (Clerget). The relationship between the Clerget sucrose and the direct (commercial) polarization of a raw sugar depends upon the amount and character of the reducing sugars present. If the levulose and dextrose are present in about equal quantities, that is, if the reducing sugars are in the form of invert sugar, the Clerget will be higher than the direct polarization by about one-third of the percentage of reducing sugars.[2] Expressed in symbols the ratio known as the polarizing constant is

$$\frac{S - P}{I} = 0.333$$

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[2] Browne, *The Planter*, **61**, **202** (**1918**).

If the levulose is in less proportion, the ratio will be lowered, and the Clerget and commercial polarization will be closer together. If the levulose is reduced still farther, the Clerget may be actually lower than the direct polarization. This occurs when the dextrose-levulose proportion is above 64 to 36.[3]

Zerban and Hardin[4] showed that there is a seasonal variation of the $(S - P)/I$ ratio in the raw sugars received at the New York Sugar Trade Laboratory. Composite samples of all sugars received during each week were analyzed; commercial polarization, sucrose by invertase and by Jackson-Gillis method IV, and reducing sugars were determined. In calculating the $(S - P)/I$ ratio S was taken as sucrose by the invertase method and P the direct reading on the *deleaded* sample for the invertase method. (The direct reading in the Jackson-Gillis method IV is always low because of the depressing effect of the added sodium chloride.) The results indicated three different periods in the year, viz., (1) January to April when fresh normal sugars were received and the ratio was 0.291, (2) May, June, and July when the ratio was low, probably due to the presence of levulose-destroying torulae, and (3) August to December when the ratio rose again, due to inversion of the raws in storage by mold fungi and bacteria. A summary of the relationship for the three periods follows:

PERIOD	COMMERCIAL POLARIZATION	DIRECT READING FOR INVERTASE P	SUCROSE BY INVERTASE S	SUCROSE J. & G.	I	$(S - P)/I$
Jan. to Apr.	96.37	96.24	96.56	96.65	1.11	0.291
May, June, and July	96.25	96.10	96.25	96.32	1.18	0.139
Aug. to Dec.	95.95	95.75	96.05	96.09	1.36	0.230
Year, 1925	96.16	96.00	96.28	96.34	1.23	0.230

In the 52 weekly analyses, 42 showed sucrose by invertase higher than the commercial polarization; in one case the two figures were the same, and in the nine others the Clerget by invertase was lower than the ordinary polarization.

Zerban[5] studied two different methods for determining dextrose and levulose in the presence of large amounts of sucrose and worked out the polarizing constants of various raw sugars by both methods. His work should be consulted by interested students. Methods for cane molasses have been described by Zerban and Erb.[6]

676. Determination of Reducing Sugars. For the ordinary run of raw sugars containing less than 1.5 per cent of glucose, use Herzfeld's gravimetric method II (Sec. 503). Forty grams of sugar is weighed into a 200-ml. flask,

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[3] Zerban, *Ind. Eng. Chem., Anal. Ed.,* **8,** 321 (1936).

[4] *The Planter,* **77,** No. 6 (1926).

[5] *Loc. cit.*

[6] *Ind. Eng. Chem., Anal. Ed.,* **10,** No. 5, 246 (1938).

dissolved in water and made to the mark. After mixing well add dry kieselguhr and 0.25 gram (approx.) of dry sodium oxalate (the latter to remove lime salts), and filter; 50 ml. of the filtrate is used for the determination. Clarification may be effected with neutral lead subacetate (about 3 ml.) then filtering, after which the mixed solution of disodium phosphate and potassium oxalate (7 grams + 3 grams: 100 ml.) according to Cook and McAllep (p. 411) is used in an aliquot portion of the filtrate to decalcify and delead. The volumetric method of Eynon and Lane (Sec. 506) may be selected, using either method of clarification.

If the sugar is of lower test and contains more than 1.5 per cent and less than 3.5 per cent invert sugar weigh 20 grams to 200 ml., if more than 3.5 per cent invert sugar, 10 grams to 200 ml. For calculating the results with the gravimetric method (Herzfeld No. I) use Rice's tables, p. 785. The above concentrations of solution serve for the Eynon-Lane method also.

677. Estimation of the Moisture. The method of drying sugars in an ordinary oven is described in Sec. 522. As a rule a fixed drying period (3–4 hours) is used in sugar laboratories, rather than attempting to dry to constant weight. A vacuum oven with temperature not over 70° C. is recommended for accurate work.

The Spencer electric oven has proved both rapid and accurate for sugar moisture determinations. It is particularly valuable in controlling the percentage of moisture in sugars as produced in order to insure good keeping quality of the raws in storage (see "Factor of Safety," p. 270). The method of drying sugars in the Spencer oven is described in Sec. 524. The Spencer oven is approved for routine moisture determinations by the Committee on Uniformity of the International Society of Sugar Cane Technologists.

678. Drying of Sugars under Vacuum. A simple and inexpensive oven for the determination of dry substance in sugars has been devised by Gonzalo R. Serbia.[7] It combines the Spencer method of drying the sample in a current of heated air with the official vacuum method. An electric drying oven with thermostatic temperature control provides the heated air at the desired temperature (Fig. 184). The circulation of air inside the oven is such that all the air entering it passes over the heating elements, thermometer, thermostat, and thence through the sample of sugar in a perforated capsule. The capsule for the sample is contained in an airtight chamber provided with an easily removable cover. The rate of flow of air can be regulated from the outside, to adjust the desired pressure. A connection is provided for a manometer. It is possible to dry 10-gram samples of sugar in thirty minutes at 75° C. with a vacuum of 25-in. Hg (factory system), after which the loss in weight is less than 1 mg. in one hour. With the use of a high vacuum laboratory pump or ejector, still lower temperature and pressure can be used, as prescribed in the official methods, thus completing the operation in a small fraction of the time required with an ordinary vacuum oven.

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[7] *Sugar*, August, 1943, p. 19.

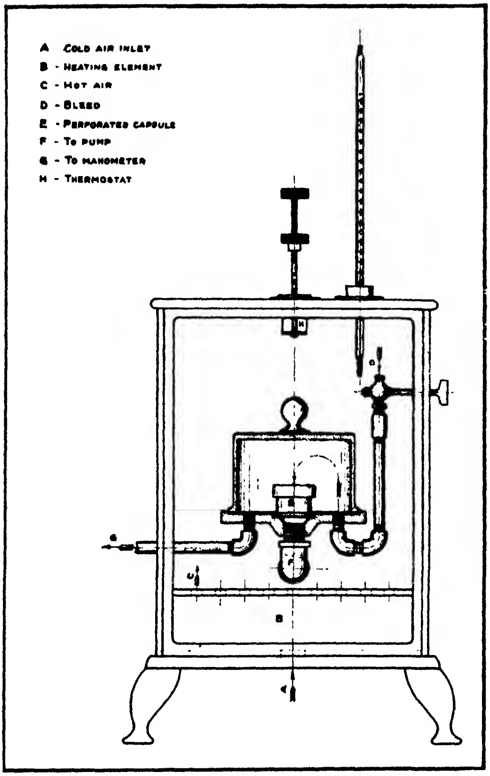

Fig. 184. Serbia's Vacuum Oven.

679. Determination of the Ash. The sulfated ash method as described in Sec. 538 is generally used in both factory and commercial testing. Weigh 5 grams of sugar in a platinum or fused quartz dish, add 0.5 ml. of concentrated sulfuric acid, ignite gently until fully carbonized, then at low red heat in a muffle, add a few drops more of acid, ignite to constant weight, and weigh. Express the result, without deduction, as sulfate ash or deduct one-tenth, depending on the practice employed. (See p. 448 concerning deduction.) The ash may be determined electrometrically as given in Sec. 544 and Sec. 545.

680. Determination of Gums. The percentage of crude gums of a raw sugar is known to affect its working qualities in the refinery, though the determination is not generally included in routine procedure. The results of the determination are dependent upon many factors, and strict adherence to the technic and method is essential for comparable results.

Ruff and Withrow[8] specified the following details after a careful study of the various methods in the literature. Dissolve the sugar in an equal weight

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[8] *Ind. Eng. Chem.*, 14, No. 12 (1922).

of water. If the sample is a solution, e.g., juice or sirup, concentrate or dilute to about 50 per cent of solids. With materials of a high gum content use 5 grams of solids (10-ml. sample); with those of low gum content use 10 grams (20-ml. sample). Sodium benzoate (0.5 per cent) may be used to keep sirup for gum analysis. (Suspended matter in ordinary raws may be disregarded. For juices containing much suspended matter filter through asbestos.)

Add to the sugar solution 0.5 ml. of concentrated hydrochloric acid for the 10-ml. sample, and 1.0 ml. of acid for the 20-ml. sample; then add 50 ml. of 93 to 96 per cent (by volume) alcohol to the 10-ml. sample, and 100 ml. of alcohol to the 20-ml. sample. This precipitation is conducted preferably in beakers of 250-ml. capacity, the alcohol being added from a rather slow delivery pipet, while the mixture is thoroughly and vigorously agitated. The precipitate is allowed to settle for fifteen minutes before filtering, to avoid slow filtering.

The filtration is done through an asbestos mat Gooch crucible, this type of crucible having been found to be superior to alundum for this work. The mat should contain at least 0.20 gram of dry asbestos. It is unnecessary to wash the mat free from fine fibers or to weigh the crucible before filtering the gums. The vessel containing the precipitate should be drained before rinsing, and the rinsings of wash alcohol should not be added before the last drop of original liquor has passed through the mat. The precipitate is then washed with alcohol of the same strength as that used for the precipitation.

After washing, the crucible is dried at 100° to 105° C. to constant weight, one hour usually being sufficient. The crucible is cooled in a desiccator and accurately weighed on an analytical balance. After drying and weighing, the crucible is ignited to complete combustion of the carbon. The crucible is again cooled and weighed. The difference between the weight after drying and the weight after ignition represents the dried gums. For very accurate work, or where the precipitate to be weighed is very small, a correction must be applied for the loss in weight of the dried crucible and mat during ignition. This correction may be determined once for all for any crucible and mat. The mats are nearly constant in weight if prepared by using the same volume of a standard stock solution of asbestos thoroughly shaken in water for each determination.

The alcohol used for precipitating purposes must be from 93 to 96 per cent by volume. Either pure ethyl alcohol, or alcohol denatured with methyl alcohol alone, is preferable for gum precipitation. All alcohol should be filtered before use. It is advisable to use the same strength alcohol for washing as was used for precipitating the gums. Occasionally a little acid in the wash alcohol will speed up the filtration.

681. Dye Number. This is a method of estimating the relative amount of colloids in a sugar and is described in detail in Sec. 353. It is used as an indication of the filterability of raw sugars in the refinery.

682. Iron in Sugars. Sulfide Colorimetric Method.[9] This test is rarely used except in special work. Prepare a stock solution containing 10 grams of pure crystallized ferrous sulfate, $FeSO_4 \cdot 7H_2O$ dissolved in a 50 to 60 per cent sucrose solution, with the addition of a few drops of sulfuric acid, and dilute to 1000 ml. with the sugar solution. The acid should be very largely diluted before adding it to the sugar solution.

Dilute this stock solution with distilled water, from time to time as required, e.g., 10 ml. to 100 ml. and 50 ml. of this solution to 500 ml.

The tests are made in Nessler's cylinders, a number of which of the same diameter and height should be provided. Into a series of these cylinders measure increasing amounts of the diluted stock solution, noting the quantity of iron in each, and dilute each to 100 ml. Add a milliliter of recently prepared monosulfide of ammonia to each and stir. Dissolve 3 to 10 grams of the sugar in a Nessler's cylinder, dilute the solution to 100 ml. and add 2 ml. of the sulfide solution. Let the cylinders stand ten minutes and then match the color of that including the sample with one of those containing the stock solution. Both then contain the same quantity of iron, i.e., the quantity of iron in the sugar used. The cylinders should stand on white paper in making the comparisons.

The sulfide is prepared by saturating ammonium hydroxide with hydrogen sulfide and then adding an equal volume of ammonium hydroxide. For dark sugars, incinerate the sugar, with the addition of iron-free sulfuric acid, burning at the lowest possible temperature. Dissolve the ash in a minimum quantity of iron-free hydrochloric acid and proceed with this solution as has been described.

683. Color of Sugars. The color of raw sugars has become an important figure both for the producer and the refiner. It may be determined in any of the several ways described in the chapter on color determinations, p. 470. The two simplest instruments for routine laboratory use are the Hess-Ives tint photometer, Sec. 583, or some form of Duboscq colorimeter, Sec. 579. The Duboscq modified by Brewster is also recommended. Prepare the solution as follows.

Weigh 125 grams of sugar into a tared Erlenmeyer flask, including stirring rod, then add boiling water a few milliliters at a time, stirring vigorously and constantly until solution is complete, being careful that the total weight of sugar and water does not exceed 200 grams. The Brix of the solution must at all times be 60° or above to avoid coagulation of colloids (see p. 471). The solution is allowed to cool, and the necessary amount of water is added to complete the weight to exactly 200 grams. Add a weighed quantity of washed and dried kieselguhr, Sec. 575, and filter repeatedly until the solution is free from turbidity as evidenced by a slanting beam of light against a dark background.

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[9] Eastick, Ogilvie, and Linfield, *Intern. Sugar J.*, **14**, 428.

For the Hess-Ives instrument, place this solution in a cell of suitable thickness (3 mm. or 6 mm., depending on color of solution), read through "560" screen, recording as Meade-Harris units (p. 481), and calculate to 1 cm. thickness. Color units are proportional to cell thickness. For the Duboscq instruments, place the clear solution in one of the tubes and a standard caramel-in-glycerin solution in the other, set the standard at any convenient figure, say 20 mm., and compare the length of the test column solution required to match the standard. Calculate the color as described on p. 476. The Brewster instrument will give $-\log \mathbf{t}$ values by the method of calculation given on p. 478.

If dilution of color is necessary, this is accomplished by the addition of the requisite amount of a 60° Brix colorless sugar solution (see Sec. 571), or preferably by the addition of pure dry white sugar in known amount before dissolving. The methods given above should give results as accurate as are required by the raw sugar producer for his purposes. For special work in conjunction with refinery char filter studies a complete color analysis by the spectrophotometer is of great value. (See p. 486.)

684. The Dutch Color Standards. Foreign sugars, on entering certain countries, pay duty according to their polarization and color. If their color is No. 16 Dutch standard, e.g., or lighter, they pay a higher rate of duty than if darker than this standard. The importance of these color standards is no longer as great as formerly (see Sec. 300).

685. Rendment. The rendment is the estimated yield of refined sugar that a raw sugar will produce based on refining experience. With the almost complete disappearance of low-grade sugars from the raw sugar market, the polarization and certain refining qualities (see p. 275) are sufficient to determine the percentage of yield to be expected. Formerly, American refineries deducted five times the percentage of ash from the polarization of the raw sugar to obtain the percentage rendment or the "analysis" of the sugar. This practice has now become obsolete.

CHAPTER 30

EXAMINATION OF REFINED SUGAR

686. Introductory. Chemical analysis of high-grade refined sugar is not general in the United States since the product polarizes so close to 100° (corrected for temperature) that the difference is negligible for most purposes. Most of the routine tests are of a physical nature and are carried out to maintain the product at the standard required by the trade. Soft brown sugars are regularly analyzed in the routine control of the refinery by the methods described for the analysis of raw sugars (Chapter 29). In selecting the amount of the sample to be taken, allowance must naturally be made for the greater quantity of reducing sugars, ash, water, and color present in the soft sugars than in raw sugars.

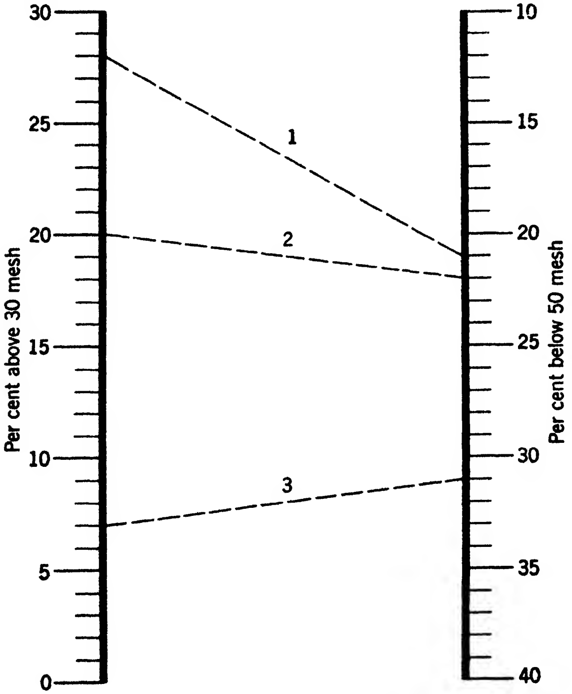

Fig. 185. Grist Test Diagram.

687. Grist Test. Grain analysis is useful in controlling the pan work and keeping the commercial product within reasonable limits as to size. Mechanical screening devices, such as the Ro-Tap, are convenient and give tests which check well with each other. A simple graphic method of reporting screen tests is shown in Fig. 185. Study of results of many tests of ordinary granulated showed that the significant figures were the amount of sugar staying on a 30-mesh screen (directly proportional to the coarseness of grain) and the amount of sugar going through a 50-mesh screen (inversely proportional to coarseness of grain). By representing two vertical scales as shown, one direct and the other inverse, results may be reported as shown by the dotted lines.

The advantage of this graphic method is that it not only shows at a glance the relative coarseness or fineness of grain but also the evenness of the crystals is indicated by the *slope* of the line. The greater the slope *downward* from left to right, the more irregular is the sugar; the greater the slope *upward* from left to right, the more regular is the sugar. In practice a horizontal line or one sloping slightly upward from left to right is about the best that can be obtained without special screening.

Referring to the diagram, the sugar represented by line 1 has 28 per cent above 30 and 21 per cent below 50, and is a sugar too coarse for ordinary granulated and irregular in grain. Line 2 with 20 and 22 per cent respectively on the two scales is a regular sugar but somewhat coarse for ordinary granulated, whereas sugar 3 with 7 and 31 per cent respectively is a very regular sugar of the fineness generally called extra fine. Examination of the grain of refined sugar by the projectoscope described in Sec. 340 is frequently of value.

688. Color of Refined Sugar. The determination of color in white sugars is far from a standardized procedure; in fact, it is in a more confused state than is the determination of color in other sugar products (see Chapter 24). The amount of color present in high-grade sugars is so slight that differences are evident only in solutions of considerable depth, whereas the colorimeters in ordinary use do not permit of reading in more than a few centimeters in depth (or thickness) so that they are valueless for white sugar solutions.

Color by Reflectance. Directions for determining the color (or more strictly speaking the reflectance) of refined sugars in the solid state by photoelectric methods have been given by Keane and Brice,[1] but the value of such determinations is doubtful. Variations in grain size affect the results, and the arrangement of the sample requires extreme care in which the personal element would seem to be a factor. Finally, Keane and Brice found (1) that the appearance of the sugars as judged by several observers was rather closely correlated with reflectance determinations, and (2) that the reflectance and the apparent color in solution (transmittancy) were in some instances widely at variance.

More recently Gillett and Holven have reported on the photoelectric measurement of reflected color of both white and soft sugars.[2] Their photoreflectometer is reported as giving reproducible results of much higher accuracy than is possible with visual comparison of soft sugars.

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[1] *Ind. Eng. Chem., Anal. Ed.*, **9**, No. 6, 258 (1937).

[2] Presented before American Chemical Society, Memphis, April 23, 1942.

Reflection measurements of solid sugars are discussed at length by Zerban,[3] and the theory and practice of the various methods advanced by different observers are given. The practical difficulties are evident from a study of this discussion. The accurate measurement of reflectance of white sugars may be of value in avoiding the personal element, but the fact still remains that it is only a measure of the *appearance* of the sugar and that the true color must be determined in solution. The practice of judging with the eye by comparison with other sugars of similar grain size under proper light conditions would seem to be almost as valuable for practical routine purposes as the quantitative determination. If the sugars are placed in two small piles side by side and then flattened down by a piece of paper until they come together, the line of demarcation will show otherwise imperceptible differences.

Color of Sugars in Solution. Qualitative Method. For routine checking of the color of refined sugars in solution the so-called blowup test is useful. It consists of making a heavy sirup from two parts of the sugar and one part of distilled water, the mixture being brought to a boil in a glass or aluminum vessel to expel air bubbles. The sirup is then examined for color and turbidity in a long test tube (1 in. by 10 in. is convenient) against a milk-glass background. A high-grade refined sugar of the specialty type, i.e., any of the large-grained sugars described on p. 341 should give a water-white sirup, whereas ordinary granulated tested in this way should show only a faint golden yellow color with no tendency to brownish, grayish, or greenish tints. No granulated sugar of standard grade should show any haziness or turbidity in the sirup in this test.

Quantitative Methods. Many color standards and methods of color determination for refined sugars have been devised but unfortunately most of them are based on arbitrary units, or if determined by the usual spectrophotometric methods (see Chapter 24) or by colorimeters not requiring a comparison standard the depth of solution examined may not be great enough to show significant differences between high-grade sugars. Keane and Brice[4] and Zerban and Sattler[5] have published methods of determining color and turbidity on unfiltered solutions. These methods can hardly be considered suitable for routine tests. The most recent study on the measurement of color and turbidity of white granulated sugar by Morse and McGinnis[6] uses a photoelectric colorimeter[7] altered to take 25-cm. absorption cells. The method involves measurement in unfiltered solutions and gives both color and turbidity indices as per cent absorbencies. About seven minutes is required for each analysis.

[3] Browne-Zerban, *Sugar Analysis,* New York, 1941, pp. 611–631.

[4] *Loc. cit.*

[5] *Ind. Eng. Chem., Anal. Ed.,* May, 1937, p. 229.

[6] *Ind. Eng. Chem., Anal. Ed.,* March, 1942.

[7] Lumetron Model 402-E Photovolt Corp., New York.

Methods using color standards for white sugar solutions have many variations. Ambler and Byall[8] have suggested the use of the caramel-glycerin standards recommended by Balch[9] for grading maple sirups. Balch's No. 5 standard is diluted 10 ml. to 100 ml. with water. This solution is compared with a 60 per cent solution of the sugar (clarified if necessary with standard Filter-Cel) in a Duboscq or similar type colorimeter (Sec. 579). The Balch standards have the advantage of having a known spectrophotometric standard of reference, but in the experience of this writer caramel-glycerin solutions do not have the correct color for comparison with refined sugar solutions or char-filtered liquors; in fact, the difference in tone is generally so great as to make accurate comparison impossible. Furthermore, in laboratories not having means of checking the caramel standard spectrophotometrically, the Balch standards are not reproducible.

Brewster's colorimeter (Sec. 581) may be used with white sugar solutions with a 460-mμ filter instead of the 560-mμ filter used for other products.

Standards made with metallic salts, of the type long used in water analysis, have been recommended[10] and have the advantage of being reproducible in any laboratory, and the color tone can be regulated to conform with the sugar solutions to be examined. A practical standard of this type for refined sugars in solution and for light-colored char-filtered liquors was developed by A. L. Holven at the California and Hawaiian Refinery and has been adopted by several refineries and large sugar buyers. The solutions are prepared as follows:

Solution *A*. 5 grams cryst. $K_2Cr_2O_7$ made to 1 liter with distilled water.
Solution *B*. 10 ml. of solution *A* diluted to 1 liter with distilled water.
Solution *C*. 5 grams $CoCl_2 \cdot 6H_2O + 50$ ml. of 0.1 N HCl made to 1 liter with distilled water.
Solution *D*. 150 ml. of solution $C + 50$ ml. of 0.1 N HCl to 1 liter with distilled water.

The mineral color standard (= 300 color) is made as follows: 150 ml. of solution $B + 120$ ml. of solution $D + 50$ ml. of 0.1 N HCl diluted to 1 liter with distilled water.

The advantages of this standard are many. (1) The standard closely matches the color tone of refined sugars and near-white char-filtered liquors. (It is valueless for colors before char filtration, e.g., washed sugar liquor.) (2) It is readily reproducible. (3) By using a long column of liquid (10–12 in.) very small differences in color may be recorded. (4) The scale is such that it shows small differences without the use of fractions. (5) The standard may be translated to absolute units if desired.

Examples of the "spread" of the scale follow. A very high-grade specialty sugar from selected liquor may test below 200 on the scale; a sugar including

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[8] *Ind. Eng. Chem., Anal. Ed.*, April 15, 1931, p. 135.
[9] *Ind. Eng. Chem.*, **22**, 255 (1930).
[10] Brewster, *Intern. Sugar J.*, April, 1932, p. 154.

one or two in-boilings may show 400, ordinary granulated from 400 to 600, and off sugars 1000 or higher.

The main disadvantage of the California and Hawaiian standard is that the diluted standard which is used for the tests fades rather rapidly and must be made fresh every two or three days (Holven recommends renewal daily). René Baus has overcome this difficulty by using a mixture of Wesson oil and white mineral oil which closely matches the color tone and can be standardized against the California and Hawaiian standard. The oil color standards keep for several months. Baus has devised a convenient colorimeter for routine work, which makes use of these oil standards, similar to the Nesslerimeter shown in Fig. 186, and by means of which any laboratory can check sugar colors within 25 units on the California and Hawaiian scale. This is about as close as can be obtained by two independent observers using a Duboscq type colorimeter with the California and Hawaiian mineral standard.

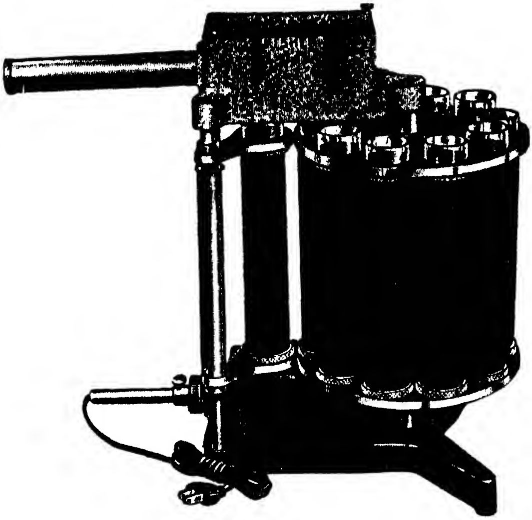

FIG. 186. Colorimeter for White Sugar Solutions.

689. Turbidity in Refined Sugars and Liquors. As has been noted in describing the blowup test, no appreciable haze or turbidity should appear in a solution of a good-grade granulated sugar. Methods for determining turbidity in white sugar solutions by spectrophotometry (expressed in $-\log t$ values) have been reported by Keane and Brice[11] and Zerban and Sattler,[12] and more recently by Morse and McGinnis.[13] Cummins and Badollet[14] have

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[11] *Loc. cit.*

[12] *Loc. cit.*

[13] *Loc. cit.*

[14] *Ind. Eng. Chem., Anal. Ed.,* Sept. 15, 1933, p. 328.

described a Tyndall-metric examination method for filtered liquors which expresses the strength of the emergent Tyndall beam in foot-candles. These methods are for research or investigation purposes, and interested students may refer to the original articles for details. For routine turbidity observations the turbidiscope of Horne and Rice may be used (see Sec. 597), but this test is only qualitative.

690. Candy Test. "Barley Candy Test." This test was devised many years ago by Dr. Samuel Hooker to approximate the conditions of making hard candies. Interest in it has been revived by various investigators in the U.S. Bureau of Chemistry and Soils who have used it in studies of the impurities in white sugars. Paine, Badollet, and Keane[15] determined polarization, pH, surface tension, and reducing sugars in several beet and cane sugars and their corresponding candies but found no very convincing findings as to the relationship between the various factors. More recently Ambler and Byall studied the effects of some impurities in white sugars on the candy test.[16] They showed that the candy test must be used with caution to obtain concordant results and that the test must always be carried out in the same manner with the same quantities of materials and under the same conditions of heating, etc., down to the smallest detail. As will be shown later the Bureau of Standards has greatly elaborated the test to eliminate errors.

It is obvious that the test may prove not only valueless but also definitely misleading in the hands of an ordinary operator who uses it only occasionally. The test is not recommended except in cases where sufficient repetitions with the same sugar can be made to prove that the operator can obtain consistent results. In fact if the test is to be used at all it would probably be best to adopt the Bureau of Standards procedure described later. Directions for the original method follow.

Apparatus for Candy Test. A copper casserole is beaten out of 1/16-in. stock. It is 4 9/16 in. in diameter at the top, 2¼ in. in diameter at the bottom, and 2 9/16 in. deep. The sides are fairly straight, slightly rounded toward the bottom. Riveted on one side is a handle made of copper tube, slightly tapering toward the bowl, ⅞ in. in diameter at the larger, outer end, and about 2 in. long, fitted with an oak or ash handle about 7 in. long.

For pouring and cooling the specimen, a clean polished copper slab 14 in. by 14 in. by ¼ in. thick is used.

Procedure for Test. To 227 grams of the sugar add 90 ml. of distilled water in the copper casserole. Heat over a naked flame so regulated that the total time of heating to 175° C. will be twenty-one to twenty-five minutes. The mixture is constantly stirred until the sugar is dissolved; then the stirring rod is removed. The solution should start to boil in five to five and a half minutes, and the casserole should then be covered with a watch glass. If the sugar tends to foam excessively on boiling, the casserole may be lifted

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[15] *Ind. Eng. Chem.*, **16**, 1252 (1924).

[16] *Ind. Eng. Chem., Anal. Ed.*, May 15, 1935, p. 168.

from the flame momentarily until the foam subsides. Such excessive foaming should be noted as a characteristic of the sugar.

Fifteen minutes after the casserole was first placed over the flame, remove the watch glass, insert a thermometer, and stir constantly with the thermometer until the temperature reaches 175° C. Remove the casserole from flame immediately, and pour the contents rapidly on the cold copper slab. After the candy is brittle it may be broken up and analyzed.

691. Bureau of Standards Method for Candy Test.[17] The Bureau of Standards recommends a test of much more elaborate scope. Every detail of the apparatus is rigidly prescribed: the vessel is of glass; the thermometer must follow strict specification; the thermometer bulb is accurately placed in a fixed position; a special gas burner with gas-pressure regulation is used, and the whole assembly is especially designed. The description of the apparatus and the method of test, together with the results attainable, covers twenty pages and cannot be given here for reasons of space. A high degree of reproducibility is reported. The conclusion is reached that "the dependability and increased convenience of the new procedure, once the equipment is installed, and as compared with what heretofore has been available, should place the candy test on a much surer footing."

Fig. 187. Sediment Tester.

The Bureau investigators point out that the loss in polarization of the candies, which was originally the only criterion on which Hooker based his test, is not necessarily a measure of the grade of the sugar; in fact, pure sucrose will show a much greater drop than an ordinary granulated. The degree of caramelization is of greater value in judging the character of the sugar, together with other characteristics such as foam number, and the spread of the plaque of candy on the cooling slab (which is water-cooled at 25° C.).

692. Test for Foreign Matter. A test which has been found of value in controlling the quality of refined sugar is made with the Wisconsin sediment tester originally designed for milk. Minute particles of foreign matter such as scale or dust which may get into refined sugar from various sources can be readily detected by means of this device.

The sediment tester (Fig. 187) consists of a heavy copper tank with air-tight cover fitted with a rubber aspirating bulb which can be used to force any liquid through a special cotton filtering disk held in the bottom of the

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[17] See "Polarimetry, Saccharimetry and the Sugars," *Bur. Standards Circ. C 440*, p. 371.

apparatus. In making the test, 200 grams of sugar are dissolved in about half a liter of hot distilled water which has previously been put through the apparatus to make certain that the water contains no sediment or specks. Any foreign matter present in the sugar solution is collected on the filter disk which may then be inspected with a small lens.

This apparatus is also valuable for checking the origin in the refinery of any foreign matter which may occur in the sugar. Liquors and sirups may be filtered through the sediment tester, and the character and origin of the foreign matter may be determined. The apparatus shown is intended for portable use, but it has been found convenient to fix it to a support for routine laboratory tests.

693. Reducing Sugars (Invert Sugar) in Refined Sugars. It is not customary to make this test in refineries in the United States, but Lyle[18] reports that it is routine practice in England to determine the invert sugar in the refined sugar output and to control the quality by maintaining the invert and ash content within certain limits. The maximum allowable invert is 0.01 per cent with an actual average of 0.008 per cent. American granulated sugars tested for comparison showed 0.033 per cent invert.[19]

The amount of invert sugar present is so small as to be difficult to determine by the usual means described in Chapter 20. The method of Kraisy (see Sec. 511) has been found accurate and effective in the New York Sugar Trade Laboratory. The colorimetric method devised by deWhalley described in Sec. 512 is especially suited for routine analyses of high-grade refined sugars because it is rapid. It is recommended if this test is to be made regularly.

694. Ash Determination. The ash in refined sugars is not generally determined by United States refiners; but as noted in the discussion of invert sugar it is routine procedure in English refineries. The upper limit for ash according to Lyle is 0.009 per cent with 0.006 per cent as the average for the output of Plaistow Wharf Refinery (London). American granulated sugars analyzed for comparison averaged 0.009 per cent ash.

Chemical Ash. The sulfate ash may be determined with a solution of the sugar by the dropwise method of Valdez and Camps-Campins described in Sec. 538. At least 25 grams of sugar should be taken in order to insure a weighable quantity of ash.

Conductimetric Ash. The best and simplest way of determining the ash in refined sugars is the electrometric method using the simple C ratio (Sec. 542) for the calculation. Zerban and Sattler[20] give the following directions. Dissolve 25 grams of the sugar in 100 ml. conductivity water, determine the specific conductance of the solution at 20° C., correct for the conductivity of the water as explained below, and multiply by the factor 530 (the average C ratio found by Zerban and Sattler).

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[18] *Technology for Sugar Refinery Workers,* London, 1941, p. 317.

[19] *Op. cit.,* p. 254.

[20] *Ind. Eng. Chem., Anal. Ed.,* Jan. 15, 1931, p. 41.

The sucrose content of the solution depresses the conductance of the salts in the sugar and also that of the electrolytes in the water used in dissolving it. When refined sugars containing minute quantities of ash are measured the conductance of the water represents a large proportion of the conductance of the solution and may even exceed it. To correct for the depressing effect of the sucrose when 25 grams of refined sugar is dissolved in 100 ml. of water, the conductance of the water should be multiplied by 0.6 and this product deducted from the conductance of the solution of the sample tested.

It should be emphasized that extreme cleanliness is essential in determining conductrimetric ash of refined sugars. Contaminating influences of any sort may completely vitiate the value of the determinations.

IMPURITIES IN WHITE SUGARS

695. Amount and Importance. Refined sugar is the purest organic substance produced in volume and is among the purest of any commercial products, organic or inorganic. The percentage of sucrose is above 99.9, and the non-sugar impurities are in minute quantities, in the order of parts per 100,000. The polarization is of little or no value in checking the quality of a white sugar since the difference between the polarization of a highest-grade specialty sugar and that of a relatively poor grade of ordinary granulated is within the limits of polarization errors. The impurities, particularly color and colloids causing haziness in solution, are of great importance in many processes such as the making of fine candies, condensed milk, water-white sirups, and soft drinks. For most practical purposes the measure of the impurities present is the color and appearance of a solution of the sugar; a sugar giving a clear and colorless, or nearly colorless, blowup test (see p. 551) is of the highest grade. A measure of the color is valuable for comparative purposes (p. 552), but quantitative determination of other non-sugar impurities is generally of value only for research.

Extensive studies of the impurities in white sugars, their methods of determination, distribution in the crystal, and effects on the decomposition of sucrose at high temperature have been made by the Carbohydrate Division of the U.S. Bureau of Chemistry and Soils, and a summary of the findings of these investigations is given below.

696. Distribution of Impurities in the Crystal. It has long been a matter of practical knowledge that the major portion of the impurities of a white sugar is on or near the surface. Keane, Ambler, and Byall of the Bureau of Chemistry and Soils made quantitative studies by mingling sugar crystals screened to uniform size with sugar solutions of different densities before saturation so as to dissolve from 4.3 to 30.0 per cent of the crystal mass.[21]

Certain general conclusions were reached in line with practical knowledge. (1) The higher the purity of the liquor from which the sugar is crystallized,

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[21] *Ind. Eng. Chem.*, January, 1935, p. 30.

the freer it will be of impurities. (2) The separation of the crystals from the mother liquor must be as complete as possible, and the adherent film of mother liquor must be removed as thoroughly as possible by washing. (3) Small-grained sugars and those of crystals of uneven size are more difficult to free of the mother liquor. (4) For several reasons washing in a centrifugal is neither complete nor uniform. These investigators showed that mingling crystals with a saturated solution of sugar and repurging will completely remove the film of mother liquor, resulting in an improvement in color and ash, but that further pronounced improvement in color, purity, and quality of the sugar is obtained when the outer shell of the crystal is also removed. (See Sec. 290 on double purging.) This is to be expected since the exterior layers of the crystal are formed from liquor in which the non-sugar content is becoming progressively higher, whereas the center portions were formed while liquor was at or near its original purity.

Analyses of the original sugars and those resulting from the treatment with sugar solutions of different densities below saturation showed that in general over 50 per cent of the ash, sulfates, chlorides, sodium, potassium, and total nitrogen is located in the outer 5 per cent of the crystal, i.e., in the surface layer, whereas color, calcium, and sulfites are more uniformly distributed through the crystal. The candy tests (see Sec. 690) made from the sugars when the outer layer of the crystal has been removed were much better in color.

697. Effects of Impurities on Decomposition of Sucrose at High Temperatures. Ambler and Byall[22] studied the effects of salts and organic substances which may occur in white sugars, by making the candy test on a very pure sugar to which the non-sugar was added in known proportions. (The difficulties in making the candy test which were brought out by this study have already been referred to in Sec. 690.) It was found that the non-sugars fall into three groups. Group I is composed of those which inhibit color formation while increasing the amount of reducing sugars. This group includes the neutral salts: chlorides, nitrates, and sulfates of sodium, potassium, and calcium; and those which have an acid reaction, like potassium acid phosphate and tartrate. Group II includes the non-sugars which increase color and decrease the amount of reducing sugars, and salts which have an alkaline reaction in solution (such as sodium carbonate and bicarbonate and potassium carbonate), and salts of strong bases with volatile or unstable acids (such as sodium nitrite and sulfite, disodium phosphate, calcium gluconate, and sodium citrate). Group III is made up of those non-sugars which increase both the color and the reducing substances, which include ammonium salts: amino acids and amides and iron salts. It will be noted that this third group includes those substances which have long been recognized in practice as detrimental to sugar quality.

The work of these investigators also emphasized the acidic nature of sucrose, which is greatly enhanced at the high temperatures studied, causing

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[22] *Ind. Eng. Chem., Anal. Ed.,* May, 1935.

the so-called auto-inversion of pure sucrose by heat. They showed that sugars with less than 0.010 per cent ash if heated dry at 170° C. for fifteen minutes[23] (Pucherna's caramelization test) may show inversion as high as 35 per cent with little increase in color, whereas in sugars with more than 0.010 per cent ash the inversion will be less than 1 per cent. They conclude that this ash of 0.010 per cent in sugars of usual composition is just sufficient to act as a buffer against the increased liberation of hydrogen ions from the sucrose at the high temperature of the test.

698. Determination of Impurities in White Sugar. As has been stated above, the color and appearance are the only determinations usually made for practical work with white sugars. For research or other studies the methods developed by J. A. Ambler and others may be used for the determination of pH, sulfates, sulfites, phosphorus, silica, chlorides, calcium, sodium, and potassium oxides; total nitrogen, protein, ammonia, nitrates, nitrites, amino acids, and related compounds. All these methods have been published[24] and have recently been compiled into a booklet[25] giving the complete methods in detail.

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[23] *Z. Zuckerind čechoslovak. Rep.*, 55–144, 663 (1930–1931).

[24] *Ind. Eng. Chem., Anal. Ed.*, 1930, 1931, 1932.

[25] "Methods for Determining the Uniformity of Quality of White Sugar." J. A. Ambler, May, 1940, Bur. Agr. Chem. Eng., Washington, D. C.

CHAPTER 31

ANALYSIS OF THE FILTER PRESS CAKE

699. Preparation of the Sample. The sample of press cake obtained as directed in Sec. 615 should be reduced to small fragments and mixed with a spatula. If the cake is very soft, it may be necessary to rub it to a paste in a large mortar, then subsample it. If moisture is to be determined, the mixing and subsampling should be rapid to avoid evaporation.

700. Moisture Determination. Moisture in press cake is not usually included in the daily routine tests as the appearance of the cake is sufficient guide to the factory superintendents and the pressmen.

The Committee on Uniformity of the International Society of Sugar Cane Technologists recommends that as large a sample as can be conveniently handled should be used. The Hawaiian methods specify 25 grams. Drying to constant weight in a water-jacketed oven at approximately 100° C. is the simplest method if special drying apparatus is not at hand.

Loss in weight × 100 ÷ weight of sample taken = per cent moisture

The sample should be partly dried at a low temperature before heating the oven to 100° C., otherwise the surfaces of the fragments of press cake may be covered with a glazed coating which would prevent the escape of moisture.

Moisture may be determined on a large sample (100 grams) in the Spencer electric bagasse oven (p. 564) or in a smaller amount (5 grams) in the oven used for sugar moistures (p. 439). The bottom of the capsule should be covered with asbestos to prevent dust being pulled through the wire mesh by the air current.

701. Sucrose or Polarization. The true sucrose in the press cake (by double polarization) is never determined, as the difference between sucrose and polarization is so slight as to be negligible. The International Society of Sugar Cane Technologists Committee on Uniformity says, "Sucrose (in filter cake) is by common consent considered to be equal to pol." Transfer 25 grams of filter press cake to a small mortar. Add boiling hot water to the sample and rub it to a smooth cream with the pestle. Wash the material into a 100-ml. flask with hot water, cool, add 6 ml. lead subacetate solution (54.3° Brix), complete the volume to 100 ml., mix the contents of the flask thoroughly, filter, and polarize. The polariscope reading is the polarization (pol) in the press cake.

It is usually more convenient to use 50 grams of the sample, and add the lead subacetate while rubbing the material to a cream and wash all into a

200-ml. flask. This facilitates the removal of the last portions of the press cake from the mortar. A flask with the neck enlarged above the graduation is more convenient in this analysis than an ordinary sugar flask. (Fig. 188.)

The International Society of Sugar Cane Technologists recommends the addition of 1 or 2 grams of Horne's dry lead after the solution is made to the mark instead of the wet lead specified above.

The object in using 25 grams of the material in this analysis instead of the normal weight is to correct for the volume of the lead precipitate and that of the insoluble matter.

Parallel experiments on a sample of filter press cake, by the method described, and also modified by adding acetic acid after cooling to decompose saccharates of lime and precipitate levulose, gave in each 9.6 pol. A third portion of this sample was rubbed to a cream with hot water and defecated with lead subacetate, then washed onto a filter. The washing was continued with very hot water to nearly 200 ml. of filtrate. The filtrate was cooled and diluted to 200 ml. and polarized, giving a reading of 4.6. The residue was washed into a sugar flask and diluted to 100 ml. and filtered. The filtrate polarized 0.5. The sum of $4.6 \times 2 + 0.5$ is 9.7, or nearly the same as in the other two experiments, showing that 25 grams is approximately the correct amount of average material to use instead of the normal weight. Many experiments by Spencer have given results similar to those described.

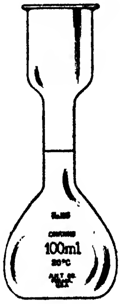

FIG. 188. Kohlrausch Flask.

A routine method devised by Spencer uses an ordinary milk-shake shaker to insure disintegration of the sample. Fifty-two grams of the finely divided press cake is placed in the tumbler of the shaking machine, then 162 ml. of distilled water (room temperature), using an automatic buret. The proportions of press cake and water are now as above. About 2 grams of Horne's dry lead is added, the tumbler is covered, and the machine put in motion to agitate the mass violently. Filter and polarize in a 200-mm. tube.

CHAPTER 32

ANALYSIS OF THE BAGASSE

702. General Considerations. The analysis of the bagasse presents some problems which are not present in the analysis of the other products in the factory control such as juice, sugar, and molasses. The large proportion of insoluble matter as compared to the soluble constituents, as well as the lack of homogeneity in the different sized particles, make the sampling and analysis more difficult. Many investigations of methods have been carried out, and the subject is still being studied.

The Committee on Uniformity of the International Society of Sugar Cane Technologists and most of the published methods prescribe that for control purposes the bagasse from the last mill be analyzed as sampled, i.e., without further cutting or chopping. In the methods given in this chapter it is assumed that the bagasse sampled as it leaves the last mill is not given further treatment after it is sampled and subsampled.

Obviously if special tests are to be made of the bagasse from earlier mills in the tandem, such samples will have to be further disintegrated before analysis, particularly for the polarization.

703. Preparation of the Samples. After securing the sample as described in Sec. 603, it should be rapidly and thoroughly mixed and subsampled. Modern milling in long trains and under high pressures disintegrates the bagasse to such an extent that it is general practice, as stated above, to make both the polarization and moisture tests on the sample in the condition that it comes from the mill. Some few laboratories continue the use of choppers or shredders, but this is not recommended.

The most recent and elaborate study of the sampling and analysis of bagasse was made by Behne. He found[1] that with reasonable care in sampling and mixing (by methods given in Sec. 603) accurate polarization and moisture results can be attained by the methods given in the following sections.

For those special analyses of bagasse from the earlier mills (which are made only occasionally) preparation of the sample, at least for the polarization test, may be indicated.

Slow preparation of the sample may result in a large error in the moisture test. This may be avoided by drying the bagasse in the condition that it comes from the mills (even the earlier mills in the tandem), and if large enough samples are used for drying this is to be recommended.

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[1] *Intern. Sugar J.*, October, 1937, p. 390.

The Warmoth disintegrator, already described on p. 508 for use in the analysis of cane, is the best and most rapid method of preparing these special bagasse samples for analysis. Experience has shown that about 1 per cent of moisture is lost during sampling, storing, and disintegration in this machine. By using 99 grams of the bagasse sample thus prepared both for the moisture and polarization determinations, instead of 100 grams, this evaporation is compensated for, and furthermore the relationship between moisture, polarization, and fiber is strictly comparable since the tests are made on the same sample. This latter point is equally true if both the polarization and moisture are made on the sample in the condition that it leaves the mills. It is not so certain if the moisture is made on the unprepared sample and the polarization on a chopped or disintegrated portion.

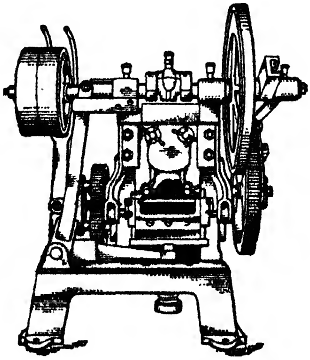

FIG. 189. Bagasse Chopper (Boot and Krantz).

The Boot and Krantz[2] chopper, Fig. 189, is also suitable for preparing the sample. The knife is adjustable to cut the bagasse to any desired fineness. The machine may be driven by belt or by hand power. Another device is the Cutex cane fibrator made in England. Other types of choppers (such as meat choppers) have been used but they are not so efficient or rapid as the disintegrators described above.

DETERMINATION OF THE MOISTURE

704. Size of Sample. As has been indicated in the preceding section, it is important to dry a large sample on account of the lack of uniform distribution of the moisture. India and Java prescribe samples of 1000 grams and the Committee on Uniformity of the International Society of Sugar Cane Technologists recommends "much larger samples than the usual 100 grams, i.e., at least 500 grams." However, Behne in an extended study of moisture determinations[3] found that 100-gram samples will give results that are quite satisfactory for control work, but he recommends larger samples (1000 grams) for special work where importance is attached to results of single determinations. Therefore for routine tests the 100-gram sample may be continued as heretofore, but smaller amounts than 100 grams should not be used.

Experiments upon both a manufacturing scale and in the laboratory have shown that bagasse may be heated to high temperatures without appreciable decomposition. Such tests were made in drying shredded cane at Preston, Cuba, where the temperature employed was very much higher than is here

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[2] *Intern. Sugar J.*, December, 1933, p. 480.

[3] *Intern. Sugar J.*, September, 1937, p. 348.

suggested for laboratory work. Drying in vacuum ovens at low temperatures is not dependable. The Committee on Uniformity of the International Society of Sugar Cane Technologists recommends heating to 130° C.

705. Drying in a Current of Heated Air. *Spencer Electric Oven, Large Size.* This oven, especially designed for the determination of moisture in bagasse, is designated as Type B to distinguish it from the smaller oven used for sugars, juices, etc. (Sec. 524). The bagasse oven is essentially the same in principle as the smaller size. The air first passes over an electric heating element, and a large volume of heated air is drawn through the sample to be dried. The suction is best applied by connecting with the factory vacuum system. The capsules are 10 cm. in diameter by 20 cm. high fitted with a bottom of metal filter cloth to allow free passage of the air. The test is made on 100 grams of the bagasse as it comes from the mills, i.e., without chopping or otherwise comminuting. This sample, in a tared capsule, is placed in the oven, the suction is started, and current to the heating element is regulated to give a temperature of 135° C., which is continued for forty-five minutes. The capsule is then cooled and weighed. A simple method of avoiding all calculations is to place the tare and a 100-gram weight on one pan of the balance and the capsule containing the dried sample on the other. Weights are then added to the pans containing the sample, this weight in grams representing the percentage of moisture in the bagasse.

706. Special Hot-Air Oven. The Spencer electric oven is excellent for control tests which require only a 100-gram sample, but if larger samples (500 to 2000 grams) are to be dried, an oven of another type must be used. This oven, also designed by Spencer, was the forerunner of the electric oven just described. It has the advantage that it may be constructed in the factory machine shop, largely of old materials. As shown in Fig. 190 it is a cylindrical cast-iron vessel, *C*, provided with a removable cover, *D*. A rubber gasket, *I*, and the clamps, *E*, insure an airtight joint between the body of the oven and the cover. The clamp may not be necessary if the door is heavy and the joints are well ground. A steam ejector, *F*, draws a very strong current of air through a heater, the pipe, *G*, and the basket, *A*. The air is heated by passing it over a steam coil. It may receive a preliminary drying by drawing it over quicklime, followed by filtration, but this does not appear to be necessary. The pipes should be covered, and a very large volume of air must be used.

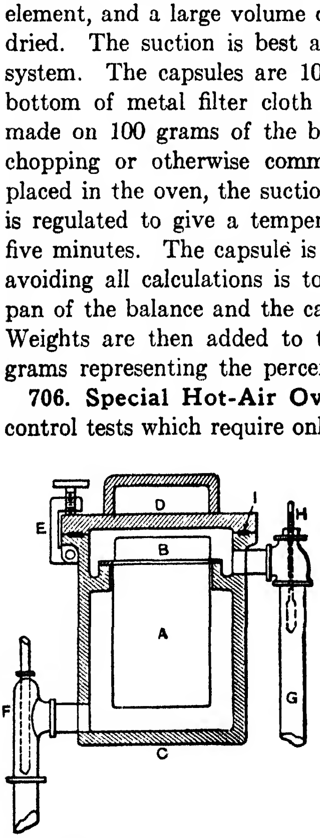

Fig. 190. Bagasse Oven with Induced Draft.

A cylindrical bagasse basket of very thin sheet brass, *A*, fits in the oven as is indicated. Two or more baskets should be provided and tared if many samples are to be dried. The bottom of the basket is of finely perforated brass plate, such as is used in centrifugals. The top of the basket is open. A narrow flange supports the basket and makes a joint with the iron castings. The weight of the basket and bagasse and the pressure of the air are usually sufficient to insure a good joint, but if need be, a gasket of steam packing may be used. The thermometer, *H*, indicates the temperature of the air current. If the oven forms one of a battery, a stop valve should be provided in the pipe, *G*. A valve is also convenient in regulating the current of air. If it is desired to leave the steam turned on the ejector, *F*, when opening the oven, it is necessary to provide an aircock in the cover or elsewhere for use in breaking the vacuum.

This oven may be constructed of any convenient size. Those constructed with a basket 8 in. in diameter and 12 in. deep will hold 1 kg. of loose bagasse or 2 kg. if lightly packed. The drying period is extended a few minutes by the packing. A basket of this size may be used with the 5-kg. balance shown in Fig. 135.

The method of drying is as follows. Reduce the sample to about 2 kg. by subsampling; fill the tared basket with 2 kg. of bagasse, packing it lightly as may be necessary. Insert the basket in the oven, replace the cover, and turn sufficient high-pressure steam into the ejector to produce a very strong current of air. The air should be heated to any convenient temperature above 110° C. and preferably to 130° C. The air pipe should be well covered.

The drying period varies with the temperature of the air and the condition of the bagasse. At 110° C. the period is usually about ninety minutes. At the close of the drying period, which after a little experience with the oven may be arbitrarily fixed, remove the basket by the bail, *B*, and place it in a desiccator to cool. A large earthenware jar will serve as a desiccator. After cooling, ascertain the weight in grams of the dry bagasse and divide this number by 20, to arrive at the per cent of dry matter. (With such a large sample weighing hot is permissible.) The per cent moisture is 100 minus the per cent of dry matter.

The oven as originally designed[4] is self-contained. The lower section contains the heating-coils, and in the upper are six bagasse drying-tubes of 200 grams capacity each. The oven illustrated is less expensive and is more accessible for repairs than the older model.

Khainovsky's Method. This is described by Behne[5] as follows:

> The apparatus is shown in Fig. 191. The fan blows air over the heating element in the duct and then up through the containers which hold the samples of bagasse. These containers fit one above the other and are lowered as the bottom one is removed, so that each one has to spend a certain period in the lowest position. A gauze lid fits neatly over the top to prevent fine particles from being carried away.
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> In the apparatus actually used the containers themselves were too heavy for accurate weighing, so light copper, gauze-bottomed sleeves were fitted in-

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[4] Spencer, *J. Ind. Eng. Chem.*, **2**, No. 6 (June, 1910).

[5] *Loc. cit.*

side each. The sample (1 kg.) was weighed into one of these sleeves, the whole then slipped into one of the heavy containers which was placed in the dryer. Drying was continued till constant weight, the temperature of the air being maintained between 110° and 130° C. Usually 1 to 1¼ hours was sufficient. The dried sample was weighed hot.

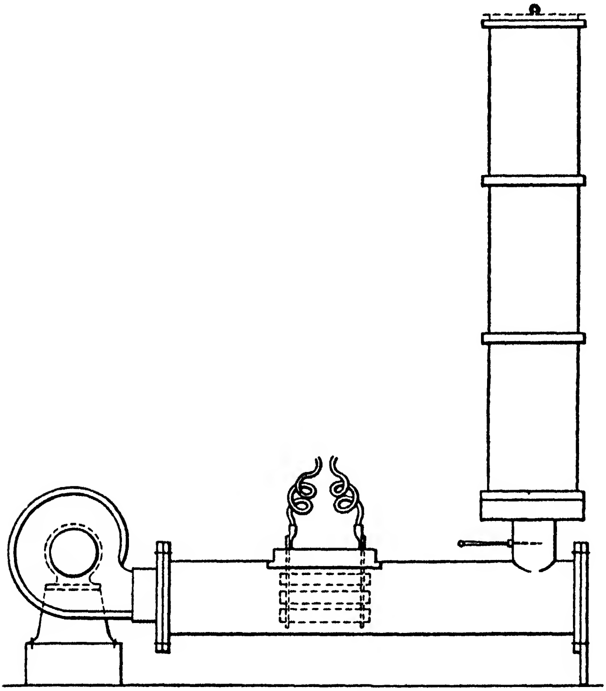

FIG. 191. Khainovsky Bagasse Drier.

The chief difference between the two methods (apart from size of sample) lies in the fact that in the Khainovsky apparatus the air is blown through the bagasse, whilst in the Spencer type suction is used.

707. Drying in Metal Trays. Shallow metal trays may be used as containers for the bagasse in conjunction with the oven described in the next paragraph. A suitable size for use on the sugar balance and holding 50 grams of bagasse is 4 in. by 8 in. by 1.25 in. deep. The drying progresses faster if the bottom of the tray is of wire gauze or perforated sheet brass. The temperature of the oven should be at least 110° C. and preferably 130° C. This is the method prescribed in Hawaii.[6] Larger trays may be used if a balance of sufficient size is at hand.

708. Steam Drying Oven. A steam drying oven is shown in Fig. 192. It is most conveniently constructed of 2-in. planks of well-seasoned lumber.

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[6] *Chemical Control*, 1931, p. 37.

Two steam manifolds, or coils, of iron pipe are used with live steam to heat the oven. Half-inch iron pipe is suitable for making the coils. The use of two coils, instead of one, facilitates regulating the temperature.

Holes in the door of the oven admit air, and the moist warm air escapes at the top. A thermometer can readily be inserted in the oven by boring a hole in the wall near the shelf. This shelf or tray is made of heavy wire screen. The mesh should be large. The door and other parts should be protected from warping by the usual carpenter's expedients. There should be globe valves on each of the inlet and tail pipes, to each coil, to regulate the steam and the discharge of the condensation water.

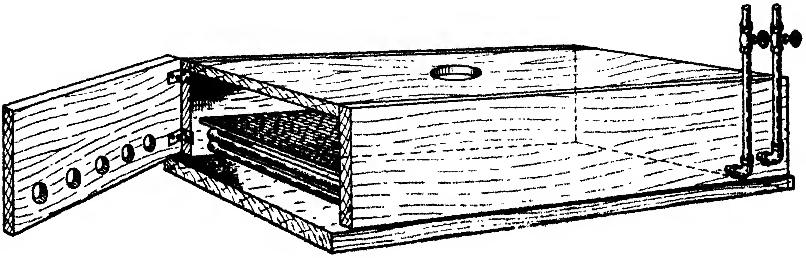

FIG. 192. Bagasse Oven.

709. Conductivity Method for Determining Water in Bagasse. G. Gundu Rao[7] has suggested a method for determining moisture in bagasse by electrical conductivity but this has not had general acceptance. When a definite quantity of the bagasse is digested with a solution of common salt of known electrical conductivity, the salt solution will be diluted, and the depression in its conductivity will be a measure of the amount of water present in the bagasse.

The conductivity imparted to the salt solution by the electrolytes in the bagasse cannot be ignored, and a correction must be applied to allow for the conductivity of the "residual juice" in the sample. This, in fact, limits the method to final bagasse, since the presence of larger quantities of residual juice than are present in that product would render the results inaccurate. Where the operating conditions are not varied, it is generally found that the residual juice correction can once and for all be determined, and its value used subsequently without appreciable error.

DETERMINATION OF THE SUCROSE (POLARIZATION)

710. Sucrose and Polarization. True sucrose is never determined in bagasse; the polarization, by general agreement, is accepted as the sucrose figure and is used in true sucrose balances. However, as the polarization is the figure determined, it is so referred to although such expressions as "sucrose

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[7] *Intern. Sugar J.*, February, 1934, p. 78; February, 1938, p. 68.

in bagasse," "sucrose lost per cent fiber" are commonly and correctly used. The polarization determination is almost invariably made by single digestion methods, and extraction methods are rarely used. The calculation by the digestion method involves the use of percentage of fiber in the bagasse, but as this is determined daily in the regular control by the indirect method described later (Sec. 716) no additional work is entailed.

The Committee on Uniformity of the International Society of Sugar Cane Technologists recommends that samples of at least 500 grams and preferably 1000 grams be used for the polarization test, but Behne[8] in a study of polarization of bagasse reached a similar conclusion to the one cited above for moisture determinations, viz., that the 100-gram sample gives satisfactory results for control figures.

711. Polarization. Single Digestion Method. This consists, in general, of digesting a weighed quantity of finely divided bagasse (100 grams usually) in a measured quantity of water (say 900 or 1000 ml.) at or near the boiling temperature for one hour in a tared container. Suitable means for agitating the solution and bagasse during the digestion period are usually provided. Five milliliters of 5 per cent sodium carbonate solution is included if the bagasse sample is fresh or was preserved with formaldehyde. If the ammonia-chloroform mixture recommended in Sec. 603 is used as a preservative the soda is omitted. After the digestion period the container is cooled and weighed, the solution drained off, clarified with dry lead, and polarized. The calculation of the polarization is explained in an example given later.

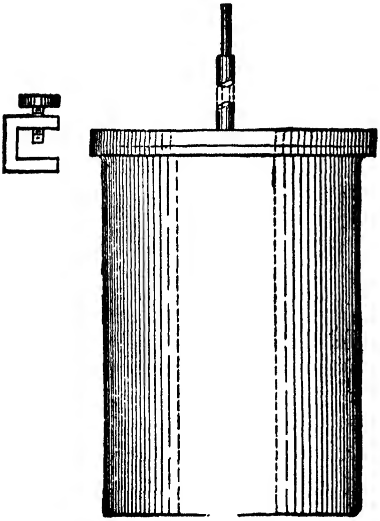

FIG. 193. Bagasse Digester.

A convenient digester, Fig. 193, is of copper about 4 in. in diameter and 6 in. deep, provided with a brass cover and clamp for making a tight joint. A brass tube attached to the cover serves as a condenser. A brass rod, carrying a small disk for mixing purposes, should pass through and extend above the tube. The rod should be moved up and down occasionally either by hand or mechanically. A Kodak developing tank, fitted with a condensing tube, may conveniently be used as a digester.

No water should be added to the bagasse after starting the digestion. The boiling should be very gentle or, preferably, the liquid should just reach the boiling point.

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[8] *Intern. Sugar J.*, August, 1937, p. 304.

The test is conducted as follows. Tare the dried digester, add 100 grams of bagasse, then 1000 grams of boiling water (0.25 gram of dry sodium carbonate should be added to the water if ammonia has not been used as a preservative). Close the digester and place in a steam or water bath and heat at the temperature of boiling water for one hour, during which the contents of the container are agitated by means of the rod as described.

Cool and weigh, drain off some of the liquid (about 150 ml.), place in a cylinder, and add sufficient dry lead subacetate to clarify. A little dry kieselguhr may be used to insure a clear filtrate. Shake vigorously, filter, and polarize in a 400-mm. tube.

The following calculation illustrates the method of arriving at the percentage of sucrose (pol), but this may be dispensed with in practice by using the table on p. 797.

Weight of bagasse used	100 grams
Fiber in bagasse	48 per cent

Weight of digester, bagasse, water	1275.5 grams	
Weight of digester	175.5	
Weight of bagasse, water	1100 grams	
Weight of fiber	48	
Weight of extract	1052 grams	
Polariscope reading in 400-mm. tube	3.2	
From Schmitz's Table 3.2	= 0.83	
0.83 ÷ 2	= 0.415	per cent sucrose (pol) in thin solution
1052 × 0.415 = 4.37 grams	= 4.37	per cent sucrose (pol) in bagasse

By the table on p. 797 opposite 3.2 and under 1050 (*W*) is found 4.37. By interpolation for 1052 the per cent sucrose (pol) is 4.38 per cent corresponding with the above. The figures for tenths in Schmitz's table are averages for a range of degrees Brix and polarizations, and this accounts for the difference of 0.01 per cent in the results.

712. Spencer Rotary Digester. This apparatus was invented by Spencer to obviate some of the difficulties involved in the use of the ordinary digester. It consists (Fig. 194) of a cylindrical steam bath in which three cylinders are rotated at 5 rpm. by means of a small motor and reducing gear. Hand operation is also provided for. The cylinders for the bagasse samples are similar to the digester previously described, being 4½ in. by 8, with tight covers held in place by screw clamps. Vacuum breaks are provided in the covers. These cylinders revolve endwise, the slow motion of the shaft causing the bagasse to fall from end to end in the solution, promoting maceration and diffusion. Steam and cold water connections to the casing provide efficient means of heating and subsequent cooling. The test is conducted, and the calculation is made in the same way as has been previously described.

Norris' Method.[9] Dr. R. S. Norris, noting that one of the weak points in the sucrose test is the small polariscope reading, devised the following modification of the single-digestion method. A special digester, Fig. 195, is used. This is like the double cooker used in the kitchen for cereals, etc. A tamp, *C*, made of a heavy perforated metal disk and provided with a substantial handle, is fitted rather snugly into the inner vessel, *B*, and serves to press the bagasse

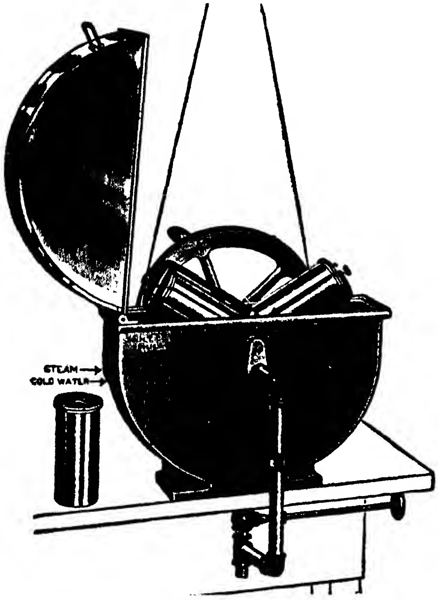

FIG. 194. Spencer Bagasse Digester.

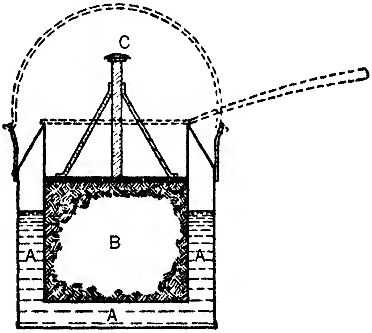

FIG. 195. Norris Bagasse Digester.

and also as a cover. The inner vessel should be 4.25 in. deep and 4.25 in. in diameter.

Weigh 100 grams of bagasse in the tared inner vessel. The tamp should be tared with the vessel. There should be no pieces of bagasse larger than 6 mm. in diameter. Add 500 ml. of hot water containing 5 ml. of a 5 per cent solution of sodium carbonate. Place the vessel in the boiler and digest for one hour, pressing the bagasse and mixing the solution every fifteen minutes. The tamp should be fastened at the top of the inner vessel when not in use, and should serve as a cover. It should not be removed until after the completion of the digestion and weighing. At the conclusion of the digestion period, remove and wipe the inner vessel and set it aside to cool and weigh, or it may be cooled by placing it in cold water. The calculation of the per cent sucrose (pol) is made as in the single-digestion method, p. 569.

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[9] *Assoc. Hawaiian Sugar Tech., Chemical Control,* 1931, p. 38.

Norris states that the shape and dimensions of the vessel apparently influence the results. The vessel should not be too deep. The dimensions given are those decided upon by Norris after many experiments.

713. Khainovsky Method for Polarization. This permits the use of samples of 1000 grams and extraction with ten liters of water. Figure 196 illustrates a setup described by Behne,[10] consisting of a single apparatus. It is usual to mount the apparatus in pairs with a common steam pipe.

One kilogram of bagasse with 10 liters of water is placed in the boiling vessel 1; gauze screen 2 is placed on top to keep the bagasse completely submerged, and the counterpoised cover 3 is lowered and clamped in position. Steam is turned on by means of the valve 4, entering through hollow trunnion 5 into the domed steam chamber 6 and exhausted through trunnion 7 and valve 8 on the opposite side of the boiling vessel. Placed on top of, and supported by the cover, is a water-cooled reflux condenser, consisting of two conical-bottomed cylindrical chambers 9 and 10, connected at the top by small pipe 11, and vented to atmosphere at 12.

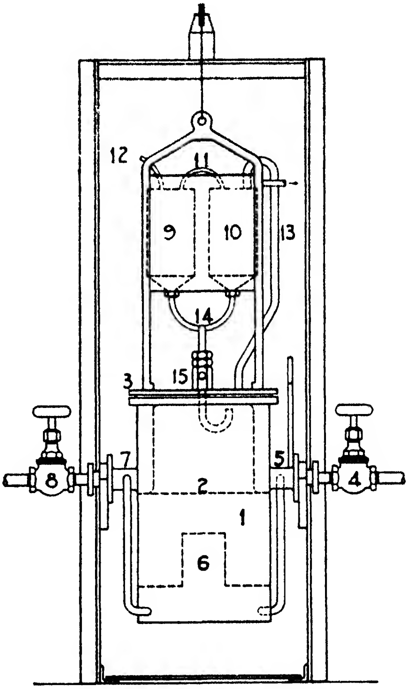

Fig. 196. Khainovsky Bagasse Extractor.

The bent pipe 13 takes the vapor from the boiling vessel to the top of the condenser, and the condensate runs away from the bottom through the Y-pipe 14 back to the boiling vessel. Boiling is regulated till a small, steady reflux is observed through the sight glass 15, and is maintained for one hour. Then the steam is turned off, the cover is raised, and the extract is drained off into a large vessel by inverting the boiler which swings about the trunnions. The extract is mixed, subsampled, cooled, and polarized, using the Schmitz method with a 400-mm. tube. The polarization may be calculated from the table on p. 755 in the same way as for the 100-gram sample.

714. Repeated Digestion or Extraction. As has been stated previously this method is rarely, if ever, used in control work. For reference purposes it is given here. In a suitable dish, preferably a porcelain casserole, cover 100

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[10] *Intern. Sugar J.*, August, 1937, p. 304.

grams of finely divided bagasse with water and boil it about ten minutes. Drain off the liquid, pressing the bagasse with an iron spoon. Repeat this digestion with water and the decantation in all eight times. Press the residue of the bagasse in a powerful hydraulic or other press and unite the liquid expressed to those portions already drained off. Cool the liquid to the ordinary temperature and measure it, adding, if need be, a little water to bring it to an easily measurable volume. Clarify the solution by Horne's dry-lead method. The degree Brix of this very dilute solution may be neglected. Polarize the solution, using a 400-mm. observation tube, and calculate the sucrose with the aid of Schmitz's table on p. 755, dividing by 2 to compensate for the tube length. This per cent pol is that of the extract. Consider the cubic centimeters of the extract to be grams and multiply this number by the pol to ascertain the pol in the bagasse. About 0.5 gram of sodium carbonate should be added to the water used in making the first extraction.

715. Comparison of Various Methods for Polarization. The methods described differ from each other in three important particulars: (1) whether the water is actively boiled or not, (2) size of sample taken, and (3) ratio of water to bagasse. The question of boiling or not boiling has been quite extensively studied.

Geerligs[11] states that long digestion with boiling water dissolves a dextrorotatory gum from the fiber of the bagasse and makes the polarization too high. De Haan extracted this gum and found that it is precipitated by the lead subacetate.[12] Norris failed to find this dextrorotatory substance in extracts from Hawaiian cane.[13]

Behne in the work already cited did not find any great difference between the Queensland method (which uses a digester similar to that on p. 568 but heated in a water bath) and the Khainovsky method that boils the water vigorously. He also found that the 100-gram sample for control work in which averages are to be used was sufficiently accurate although 1000-gram samples are recommended for single tests. He found that the 1 to 5 ratio of bagasse to water gave low results as compared with the Khainovsky method, and he concluded that the Hawaiian method, i.e., the 1 to 5 ratio but with the Queensland digester, should not be used. It was later shown that if the Norris digester as described above is used with finely divided bagasse the results are comparable to those of the Khainovsky method.[14] With coarse bagasse (as from earlier mills or with poor grinding) the results will be considerably lower than with the Khainovsky method.

Another point brought out in the Behne investigation is that the use of alkali (Na_2CO_3) is not necessary, but this is not in accord with the work of other investigators. Haddon[15] concluded that the use of 1 gram of soda ash

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[11] *Intern. Sugar J.*, **11, 56.**

[12] *Hawaiian Sugar Planters' Expt. Sta., Bull. 32,* p. 12.

[13] *Hawaiian Sugar Planters' Expt. Sta., Bull. 32,* p. 18.

[14] *Intern. Sugar J.*, July, 1939, p. 277.

[15] *Rev. agr.*, 1934, p. 168.

is not sufficient and that soon after boiling starts the alkalinity disappears. He recommends that boiling should be in the presence of barium hydrate, but this has not been accepted by other investigators. Dymond[16] shows that fiber is quite soluble in water with extended boiling and that the solubility differs with different varieties.

From the above it is evident that any of the methods of single digestion outlined in this chapter will give results suitable for control work. Points to be avoided are samples less than 100 grams, extended boiling periods (more than 1 or 2 hours), and ratios of bagasse to water less than 1 to 10, except where the Norris digester is used with finely divided bagasse.

716. Determination of the Fiber. The fiber may be determined directly as follows if the Spencer oven has been used for the moisture determination; otherwise the method described for the analysis of cane (Sec. 627) must be employed. Cover the capsule with a small mesh screen and wash the dried bagasse sample with hot pure water (condensation water) until wash water is free of sugar by alpha naphthol test. Then wash the fiber with two or three liters of boiling distilled water; dry in the Spencer bagasse oven as for moisture determination and weigh. Weight of fiber in grams equals per cent fiber in bagasse.

The indirect method is much preferable, and direct fiber determinations are now rarely made. This method was adopted by Spencer after several thousand comparative tests in the Cuban-American Sugar Company's laboratories. The required data are obtained in the mill control.

Let S = the dry matter in the bagasse;
P = the per cent sucrose (pol) in the bagasse;
C = the coefficient of purity of the residual juice (see Sec. 717);
x = per cent fiber in the bagasse.

Then

$$x = S - (100P/C)$$

Steuerwald, of the Java Experiment Station, in an investigation of the various methods of fiber determination, arrived at the conclusion that the indirect method as above gives the most reliable results. He considered the claims of the water- and alcohol-extraction methods and concluded that the aqueous methods give high figures and the alcoholic extraction, even correcting for the separation of saccharetin from the fiber, gives low figures.

Much more recently Dymond[17] in South Africa made a series of experiments indicating the solubility of fiber in hot water. With continued boiling for eight hours the fiber decreased each hour until the loss was 15 per cent of the fiber found at the end of one hour's boiling. The direct determination was further complicated by the discovery that the difference between fiber

[16] *Intern. Sugar J.*, November, 1941, p. 342.
[17] *Intern. Sugar J.*, November, 1941, p. 342.

by boiling and the fiber by the indirect method increases with the fiber content of the cane.

717. Purity of the Residual Juice. The residual juice, i.e., the juice remaining in the final bagasse, is considered to correspond in purity to that of the juice extracted by the last pair of rolls of the mill train (last expressed juice).

Studies in Java have demonstrated that the true residual juice is of much lower purity than that of the last expressed juice but as "no practical method for the routine determination is available" the International Society of Sugar Cane Technologists recommends the continuance of the use of the purity and Brix of the last expressed juice. The coefficient of purity of this juice is used in calculating the fiber. The analysis is made as for other juices.

Australian Method. According to E. R. Behne[18] the analysis of residual juice is determined in Australia on the extract obtained by the Khainovsky method for polarization (Sec. 713), using at least 500 grams of bagasse and ten times the weight of water. The specific gravity of the extract is then determined by pycnometer, using Erlees' tables[19] to obtain corresponding Brix and temperature correction. It is important that correction be made for specific gravity of the water used by boiling a blank and determining the specific gravity as for the extract. Polarization is determined in the usual way, but in calculating the Brix of the bagasse from the Brix of the extract correction must be made for the hygroscopic water content of the fiber. Values of 10 to 20 per cent have been found by different investigators, and the Australian method uses 15 per cent as a compromise. No sodium carbonate or bicarbonate should be used in making the extraction. Extreme care is needed in making the pycnometric determination of specific gravity.

Figures given by Behne show that the purity of the residual juice as determined on the extract is 10 to 12 per cent lower than the last expressed juice, but he adds, "The influence on pol extraction is of course very small being of the order of 0.05%, thus to a certain extent justifying the assumption of equality of the two purities for routine purposes."

The elaborate pycnometric determination on the extract therefore seems to be an unnecessary refinement in the control work. Possibly, a few determinations might be made to determine the average difference and a correction applied to convert last expressed juice purity to residual juice purity, but this has not been recommended by any governing body.

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[18] Personal communication to E. M. Copp.

[19] *Java Handbook.*

CHAPTER 33

CHECKING FACTORY WASTES AND BOILER FEED WATER FOR SUGAR

718. Sugar in Factory Wastes. A small percentage of sugar in the condenser waters from the pans and evaporators and in other factory wastes may result in large monetary losses. It is therefore necessary to check all outflowing waste waters regularly and systematically in the factory and in the refinery, and when appreciable amounts of sugar are found, the source of the loss should be determined immediately by following up the individual effluents which make up the combined flow. The methods given in this chapter for detecting small amounts of sugar in wastes serve also for tracing sugar to the boiler feed water.

719. Sugar in the Boiler Feed Water. The feed water for the steam boilers in a cane sugar factory is largely derived from that evaporated from the juice and sirup. The water from the steam condensed in the calandria of the first vessel of the multiple effect, the calandrias and coils of the vacuum pans, and the coils and tubes of the various heaters forms a very important source of water supply for the boilers. Sugar may enter these waters through entrainment with the vapors from the juice and sirup and through defects that develop in the heating surfaces.

Sugar causes the water to foam in the boilers and may lead to accidents. Further, though sugar may not be present in sufficient quantity to endanger the boilers through foaming, it is decomposed by the heat into products that are very detrimental to the tubes and shells of the boilers, causing pitting and overheating. The sugar is first hydrolyzed, after which the dextrose and levulose decompose. The dextrose produces levulins, formic and acetic acids, and the levulose, humic and formic acids, and insoluble humic compounds. Both the acids and the insoluble humic compounds are injurious to the boiler plates. Humic compounds form only when the water contains ammonia or soda. Except for its action on levulose in forming humic compounds, ammonia does not appear to injure the plates.

The plates may also be attacked by the acidity in the water derived from sulfited juices. This may be prevented by the addition of soda to the water or by reducing the acidity of the juices.

It is evident that a thorough control of the feed water is the best safeguard against sugar. As soon as sugar appears in the water it should be turned to the sewer and the boilers should be thoroughly blown down. The alpha-naphthol test as described below should be used at frequent intervals in testing the water and in tracing the sources of sugar in it. The odor of the steam is very pronounced when the water contains sugar.

720. Alpha-Naphthol Test for Traces of Sugar in Water. This test is widely used to determine the presence of sugar in condenser waters, boiler feed waters, factory sewer outflows, and other waters where the presence of sugar may be detrimental or represent a preventable loss. It is roughly quantitative if carried out under similar conditions and compared with tests on standard solutions. Add five drops of a 20 per cent alcoholic solution of alpha-naphthol to 2 ml. of water (cooled) in a test tube, then by means of a pipet slowly pour down the side of the tube 5 ml. of concentrated sulfuric acid. In the presence of sucrose a violet zone or ring appears at the juncture of the two liquids, the intensity of the color depending on the sucrose present. The acid used must be chemically pure and the alpha-naphthol should be of good quality. The solution of this reagent darkens on exposure to light and should be freshly prepared from time to time.

The test is extremely delicate. When the solution contains as little as one part of sucrose per million parts of water a very pale lilac tint is visible, whereas one part in ten thousand gives almost a black ring because of charring of the sugar by the acid. The colors between these limits range from various shades of lilac through a deep reddish purple. The chemist can best familiarize himself with the color variations by making tests on water of the type to be tested to which has been added known amounts of sugar. The colors darken on standing.

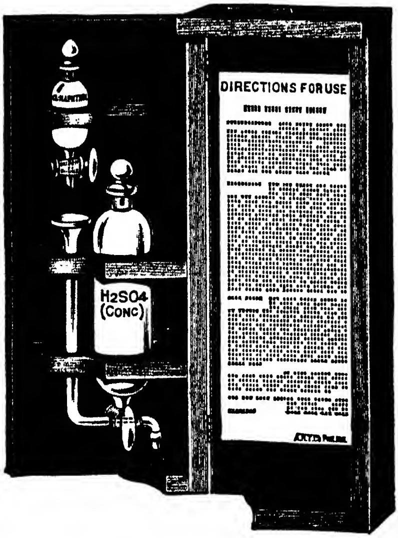

FIG. 197. Spencer Alpha-Naphthol Apparatus.

721. Spencer Alpha-Naphthol Apparatus.[1] This was devised to expedite the routine tests for sucrose in condenser waters, etc., doing away with the possibility of contamination by contact with the hands or laboratory apparatus. The funnel, Fig. 197, is first washed with repeated portions of plain water, then rinsed with the solution to be tested. After draining it is again filled with the sample and allowed to flow out until only the horizontal portion is filled. Close the three-way cock, add two or three drops of alpha-naphthol solution from the small container, allow to stand a moment, then cautiously open the three-way cock to the acid reservoir and allow several

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[1] Spencer, *Ind. Eng. Chem.*, **14**, 593 (1923).

milliliters of acid to back up in the funnel. Note the color reaction as in a test tube. Then drain off the acid and water and rinse with fresh water.

722. Quantitative Alpha-Naphthol Test. A quantitative method using the Helige comparator (Sec. 561) has been devised[2] with a special color disk showing 10, 20, 30, · · · 100 parts per million of sucrose under the conditions of the test. Five drops of alpha-naphthol and 5 ml. of sulfuric acid are added to 2 ml. of water (filtered and cooled if necessary), as described above, and the mixture is shaken; the color is allowed to develop at room temperature for three minutes and is immersed in cold water for two minutes. The sample is placed in the comparator and allowed to stand another two minutes and then the reading is taken. Any natural color in the water tested is compensated for by having 2 ml. of the water mixed with 5 ml. of distilled water in the other opening of the comparator.

723. Photoelectric Alpha-Naphthol Apparatus. A rather elaborate apparatus for making automatic tests of refinery waste waters has been patented in England.[3] The apparatus adds a fixed volume of sulfuric acid-alpha-naphthol mixture to a reaction tube to which a fixed volume of waste water is added at stated intervals. The test solution then syphons over into a cell, and depending on the amount of light which can pass through the colored solution a photoelectric cell actuates a mechanism which gives an alarm if there is excess sugar.

724. Substitutes for Alpha-Naphthol. Thymol (U.S.P.) in alcohol may be substituted for alpha-naphthol in the above tests. Lysol (concentrated commercial diluted with five parts distilled water) may also be used. A solution of 150 ml. of pure cresol made up to a liter with water containing 60 grams of Castile soap is another reagent that may be used, ten drops to the test. The substitutes are not so sensitive as alpha-naphthol, but they are advocated by various authors because the solutions do not deteriorate on standing.

A test using ammonium molybdate was described by Matthews.[4] It is not so sensitive as alpha-naphthol but it offers some advantages through the use of permanent standards for quantitative tests. In practice this method has not been found so valuable as the alpha-naphthol test.

Five milliliters of the liquid to be tested is placed in a clean test tube, three drops of concentrated HCl and 3 ml. of a 4 per cent ammonium molybdate solution are added, and the tube is placed in a boiling-water bath for exactly six minutes. If sugar is present a blue color develops, which is, however, not permanent. For quantitative work, standards are prepared by diluting a solution containing 1 gram pure sucrose per liter to convenient concentrations, and treating these solutions exactly as described for the test. Permanent standards may be made by diluting blue-black ink to match the freshly prepared primary standards. For concentrations below 0.0125 per cent of sucrose, dilute Fehling solution must be used instead of ink to get a perfect color match.

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[2] *Intern. Sugar J.*, March, 1937, p. 108.

[3] *Intern. Sugar J.*, September, 1937, p. 342.

[4] *Maryland Acad. Sci. Bull.*, 7, No. 3, 35 (1928).

These secondary standards keep for six months. The conditions of the test must be strictly adhered to in quantitative work, because even slight variations in detail may cause large errors.

725. Automatic Alarm for Sugar in the Boiler Feed Water. This alarm is based upon the change in density of the water in the presence of sugar. It is composed of two communicating tubes (communicating vessels) one within the other. The water is stagnant in the inner tube and flows through the outer at a constant level. A change of level in the inner tube causes a float to rise and close an electrical circuit and ring a bell. Since the water is of the same temperature in both tubes, the density is automatically corrected. The columns are 1.5 meters high, therefore a solution of 1.001 sp. gr. will lift the float 1 mm.

It is evident that the sensibility of the instrument may be increased by adjusting the contact. The instrument should be placed in a convenient location as free as possible of vibrations. A bell should be placed near the feed-water pump and another in the laboratory.

CHAPTER 34

DEFINITIONS AND EXPRESSIONS USED IN SUGAR HOUSE WORK

726. Uniformity in Reporting. Much confusion has resulted from the use of different terms and definitions and different methods of calculating and arriving at control figures throughout the cane sugar industry. The International Society of Sugar Cane Technologists working through its Committee on Uniformity in Reporting Factory Data has made great advances toward standardization of terms, definitions, calculations, and control methods. The findings of this committee edited by F. W. Zerban, the Chairman, have been recently published by the Society[1] in book form. Throughout this chapter and those immediately following, the recommendations of the Committee on Uniformity are given preference, but it is to be noted that the Committee's recommendations frequently conform with terminology and practice in the eastern hemisphere—Hawaii, Java, and Australia. Therefore, it has not seemed advisable to discard or disregard expressions which are in common use in most of the rest of the world. Where confusion or difference in usage has existed the expressions and definitions recommended by the Committee are marked (ISSCT), and the Committee definitions are in quotation marks. Where no confusion or difference exists and terms are universally recognized, the definitions given conform to those of the Society but are not necessarily in the same wording.

727. Absolute Juice (ISSCT). "All the dissolved solids in the cane plus the total water of the cane; cane minus fiber." The Committee decided to abandon entirely the term "normal juice" because it has several different meanings and is therefore confusing. In most of the western hemisphere, "normal juice" is used in the same sense as "undiluted juice" defined below, whereas the original meaning of the term was that of the juice as it actually exists in the cane.

The cane is known to contain water that is free of sugar. This is termed "undetermined water" or "colloidal water." If a piece of cane is passed between the rolls of a mill a part of this water exudes and drips from the end of the stalk. Because of the presence of this water it becomes difficult to define the juice as it exists in the cane in the light of factory requirements. For calculations based upon the whole juice of the cane, it may be well to consider this the water-soluble constituents—juice solids dissolved in all the

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[1] *System of Cane Sugar Control of the International Society of Sugar Cane Technologists* (New York, 1942). Published by *Sugar*.

water contained in the cane, that is, the "absolute juice" as defined above. This assumes that all the cells may be broken down and the solids may be distributed in their liquid content. The cells are never all ruptured in milling, therefore the juice extracted in dry milling can only approximate the "absolute juice."

728. Undiluted Juice (ISSCT) (Normal Juice). "The juice expressed by the mills or retained in the bagasse corrected for imbibition water. For purposes of calculation it has the Brix of the primary juice" (defined below). This corresponds to the term "normal juice" which was used in previous editions of this book and which is still generally used in Cuba, Puerto Rico, and Louisiana.

The basic difference between the ISSCT control and the older methods lies in the definition of absolute juice as "cane minus fiber," and the calculation of mill control figures on the basis of "absolute juice" instead of "undiluted" (or "normal") juice. The older methods calculate the Brix of the undiluted juice from the Brix of the first expressed juice by means of a "dry milling factor." This factor is obtained by periodically operating the mills for a short while without imbibition water, determining the Brix of first expressed juice and of mixed juice so obtained (see Sec. 613). For example, Brix first expressed juice = 20.0; Brix mixed juice (dry milling) = 19.4; dry milling factor = 19.4 ÷ 20 = 0.97. It is then assumed that this factor will be the relationship between the first expressed juice and the undiluted juice when imbibition water is used, under which conditions mixed juice will be of much lower density. For example, if the Brix of the first expressed juice is 19.21 in regular milling, the undiluted juice Brix will be 19.21 × 0.97 = 18.63. (See example, p. 599.)

E. M. Copp[2] recommends determining the absolute milling factor with "juice imbibition" instead of "dry milling." Juice imbibition replaces the imbibition water with a part of the mixed juice, operating the mills as usual, with this exception, and deferring the test until the juice from the last mill approximates that of the mixed juice in density. Copp has shown that the factor so found (about 0.95) is much lower than with dry milling, and he suggests[3] that the Brix of mixed juice found by this test will probably be the same as the absolute Brix as calculated in the ISSCT control since neither brings into account the juice in the unbroken cells. The objections to milling factors are discussed in Sec. 784.

At first glance, the ISSCT statement that "Undiluted Juice for purposes of calculation has the Brix of primary juice" would appear to be an unwarranted simplification in that it assumes a milling factor of 1.00. Actually the undiluted juice enters into only one calculation (undiluted juice in bagasse per cent fiber, p. 605) which is of little importance, since an analogous and preferable figure of absolute juice in bagasse per cent fiber is also calculated.

[2] *Facts About Sugar,* March 24, 1928, p. 280.

[3] Personal communication.

729. First Expressed Juice (ISSCT). "The juice expressed by the first two rollers of the tandem." This is juice to which no water has been added. It was formerly defined as either the crusher juice alone or the combined crusher and first mill juice, or when there is no crusher the juice from the first mill. The ISSCT definition is shorter and more exact.

730. Primary Juice (ISSCT). "All the juice expressed before dilution begins." In most mills this is the combined crusher and first mill juice.

731. Secondary Juice (ISSCT). "The diluted juice which, together with the primary juice, forms the mixed juice."

732. Last Mill Juice (ISSCT). "The juice expressed by the last mill of the tandem."

733. Last Expressed Juice (ISSCT). "The juice expressed by the last two rollers of the tandem."

734. Residual Juice (ISSCT). "The juice left in the bagasse: bagasse minus fiber." The bagasse may be regarded as a sponge that absorbs and retains a part of the juice. The juice so retained, the residue of that in the cane, is the "residual" juice. The true residual juice can only be approximated, and in the analysis only the coefficient of purity of an assumed residual juice is determined. This number is used in calculating the percentage of fiber in the bagasse and cane. In practice the juice flowing from the last mill of the train, "last mill juice," or the last roll of that mill, "last expressed juice," is considered to have the same coefficient of purity as the true residual juice. The juice from the discharge roll of the last mill is recommended for this test (last expressed).

Investigations in Java and Australia have definitely shown that the purity of the juice in the bagasse is lower than that of the "last expressed" or "last mill juice" (see Sec. 717), but the Committee on Uniformity decided that until a more practical method is found of determining the true residual juice the last expressed juice is to be used in the calculation of fiber.

735. Mixed Juice (ISSCT) (Dilute Juice). "The juice sent from the crushing plant to the boiling house."

736. Bagasse (ISSCT). "The residue obtained from crushing cane in in one or more mills. Known respectively as 'first mill bagasse,' 'second mill bagasse,' etc., and as 'last mill bagasse' or 'final bagasse,' or simply 'bagasse,' when the material from the last mill is referred to." In general, the term "bagasse" means that from the last mill unless otherwise described.

737. Fiber (ISSCT). "The dry water-insoluble matter in the cane." The true fiber or cellulose is not determined in the factory control.

738. Imbibition (ISSCT) (Saturation, Maceration). "The process in which water or juice is put on the bagasse to mix with and dilute the juice present in the latter. The water so used is termed imbibition water."

The above definition and the one following for maceration sharply define these terms, but in Cuba and Louisiana and some other sections of the western hemisphere as well, the term "maceration" is used to mean imbibition as above defined.

739. Maceration (ISSCT). "The process in which the bagasse is steeped in an excess of water or juice, generally at high temperature. The water so used is termed maceration water." By the above definitions maceration is a special kind of imbibition.

Because of the fact that maceration, as here defined, is not practiced to any extent in the western hemisphere, the terms "maceration," "imbibition," and "saturation" are used interchangeably to mean imbibition in many countries. The exact differentiation of these terms is desirable, but this is one of the cases where established usage may be hard to change. In this edition of this book the terms "imbibition" and "saturation" are used interchangeably, but "maceration" is limited to the special meaning given above.

740. Dilution Water (ISSCT). "That portion of imbibition or maceration water present in the mixed juice. Note: If the term 'dilution' is used it should always be further characterized by giving the basis to which it refers." (See p. 601 for further discussion of this point.)

741. Extraction. Mill Extraction. *Pol Extraction* (*ISSCT*). "Pol in mixed juice percent pol in cane."

Sucrose Extraction (*ISSCT*). "Sucrose in mixed juice percent sucrose in cane."

Dilute Extraction. This is the percentage of mixed juice on cane.

"Normal Extraction." Similarly, this is the percentage of undiluted juice (normal juice) on cane. This figure has been recognized as having little significance and has been dispensed with in most factory controls.

A discussion of these various extraction figures will be found in Sec. 782.

742. Java Ratio. This arbitrary milling ratio was first used in Java, but it is not in use any more in that country. It is the relationship between sucrose (pol) in the first expressed juice (generally the crusher juice) and the sucrose (pol) in the cane.

$$\text{Java ratio} = \frac{\text{Per cent sucrose (pol) in cane}}{\text{Per cent sucrose (pol) in first expressed juice}} \times 100$$

This relationship was formerly thought to be fairly constant for any given mill equipment irrespective of normal variations in the fiber content of the cane. If it is calculated for any given sugar factory over a sufficiently long period, it may have some value as an indication of the accuracy of cane weights and juice weights, as well as the sampling and analysis of the juices and bagasses although this is now questioned by many authorities. It does not afford any comparison between the work of one factory and another, and a radical change in milling equipment may alter the ratio quite appreciably for any given factory.

The Java ratio has been used in Natal and other countries to figure the sucrose in the cane as delivered by the grower, from the sucrose (pol) in the crusher juice of that cane.[4] This factor varies in Cuba from about 84 down

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[4] Bechard, *So. African Sugar J.*, **12**, 177 (1928).

to 77, the average being close to 81 for all the factories in the Cuba Sugar Club; the heavier the crushing the higher the ratio, of course. Figures as high as 90 or above have been reported by Hawaiian mills using shredders.[5]

Deerr[6] found factors varying from a minimum of 81 to a maximum of 84.8 and an average of 82.5. According to Pellet the factor in Egypt is usually from 83 to 84 and may be as low as 82 to 80.

Moberley found that the ratio was greatly affected by water adhering to the cane during rainy weather.[7] R. H. King in the Philippines found considerable variation in the ratio in the same mill even when other conditions were fairly constant.[8] Copp[9] also reports that the Java ratio varies with the fiber content of the cane. In general the Java ratio has not the importance which it was thought to have when the figure was first introduced.

743. Milling Loss (ISSCT). Sucrose (Pol) in Bagasse Per Cent Fiber. This is the relationship between the sucrose (pol) in the bagasse and the fiber. Since it takes into account the inert fiber which serves as a vehicle for the sugar lost in bagasse, it is considered a more valuable measure of milling efficiency than the per cent sucrose in bagasse alone. It is in common use in Cuba and Louisiana on a polarization basis, although always referred to as "sucrose in bagasse % fiber." This ratio does not take into consideration the sucrose in the cane.

744. Extraction Ratio (ISSCT). "The percentage ratio of unextracted sucrose (pol) to fiber in cane." (100 − sucrose (pol) extraction divided by fiber in cane) × 100. This figure is in use in Hawaii.

745. Clarified Juice (Defecated Juice). The dilute juice after it has passed through the clarification system.

746. Sirup. The sirup is the concentrated juice of the cane from which no sugar has been extracted. This is the "meladura" of the Spanish-American factories. The word sirup has an opposite meaning in sugar refineries where it is applied to solutions from which sugar has been removed by crystallization.

747. Massecuite. The massecuite is the concentrated sirup or molasses in which the sugar has been crystallized or the material has been concentrated to a point where it will crystallize. Massecuites are designated by names, numbers, or letters, indicating their purity or the number of crops of crystals of sugar that are to be removed.

748. Molasses. When a massecuite is spun in a centrifugal machine the sugar crystals are separated from the mother liquor. This liquid is termed "molasses," and is designated by names, numbers, or letters corresponding with the massecuites. The final or true molasses is the liquid residue from

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[5] *The Planter*, **68**, No. 2 (1922).

[6] *Intern. Sugar J.*, **13**, 15 (1911).

[7] *Intern. Sugar J.*, August, 1930, p. 431 (abs.).

[8] *Sugar News*, 1933, No. 5, p. 237.

[9] Personal communication.

which no more sugar can be removed, either on account of factory equipment or for commercial reasons. This is termed "barrel sirup" in the refineries.

749. Circulating Water. Condenser Water. This is the water used in condensing the vapors in the evaporation of the juice and sirup. After leaving the condensers, this water (in the tropics where water is scarce), together with that derived from the vapors, is usually passed over a cooling tower to reduce its temperature, from which it is returned to the condenser, thus recirculating through this apparatus. This water is often termed "cooling-tower water."

750. Sweet Water. With the older types of apparatus, the vapors condensed in the calandrias of the evaporator often contained sugar, carried into it by entrainment. The water resulting from these vapors was called sweet water and still retains this name, though with modern apparatus it contains no sugar. In refinery practice any very dilute sugar solution that is returned to the process is termed sweet water.

751. Entrainment. When sugar is carried off with the vapors from the evaporators and vacuum pans, this is called entrainment.

752. Coefficient of Purity. The purity of a sugar product, generally speaking, is the percentage of sucrose (pol) in the solids or Brix. The purity will obviously vary, depending on whether sucrose or polarization or true solids or Brix are used, and it is necessary to differentiate the purities as determined.

The commonest and most useful in ordinary control work is the polarization divided by the Brix which has long been called the apparent purity or simply the purity and is so termed by the ISSCT.

The true purity is the percentage of (true) sucrose in the dry substance or sucrose per cent solids.

The gravity purity is the percentage of (true) sucrose in the Brix (or gravity solids).

The apparent purity or (more commonly) purity as defined above has also been called the quotient of purity, exponent of purity, or in some sections the quotient or exponent. Its wide usefulness in all branches of the sugar industry (raw, refined, and beet) has given it general acceptance although it is recognized that its value is for comparative purposes. The test may be made in a few minutes by trained bench men.

The true purity is of greater value in comparing the work of different factories and in research work, but obviously it cannot be employed as a routine control figure in the factory, because of the time required for the determination of sucrose (Clerget) and solids by drying.

The gravity purity as defined above is not used to an appreciable extent in the West Indies or continental United States; in fact this concept is not understood by many sugar chemists in the western hemisphere.

Its use in Java and Hawaii is fairly common. The ISSCT has adopted the gravity purity as standard for comparative control; this follows from the fact that Brix (not dry substance) and sucrose are standard for all but routine control. (*Cane Sugar Factory Control,* pp. 20–21.)

A fourth type of purity may be added if the density of the solution is determined by refractometer. This has been termed the "refractive purity," but so far it is not in general use in cane house work.

It is evident that the term "purity" should be used only for "apparent purity" and that "true purity" and "gravity purity" should always be used to designate the coefficient as determined according to the correct definitions.

753. Sucrose. Polarization. These terms are frequently used synonymously in cane factory work to designate the direct polarization. The Clerget polarization is called true sucrose or the Clerget to differentiate it from the direct polarization figure. This practice of confusing sucrose and polarization has been condemned by the ISSCT and sucrose is designated as "the disaccharide known in chemistry as saccharose or cane sugar $C_{12}H_{22}O_{11}$," without regard to the method of determination; polarization is designated as "pol" and defined as "the value determined by direct or single polarization of the normal weight solution in a saccharimeter. The term is used in calculations as if it were a real substance."

Throughout this edition of this book this distinction is adhered to, but it is recognized that the previous practice of using "sucrose" to mean "polarization" (or pol) has become so ingrained in common parlance in the United States and the West Indies as to be difficult to eradicate. Such expressions as "sucrose extraction," "sucrose losses," and "purchase of cane on sucrose," when polarization is meant, will probably continue in use for many years even among scientific sugar men.

754. Glucose Coefficient. Glucose Ratio. This number is calculated as follows:

$$\frac{\text{Per cent reducing sugars} \times 100}{\text{Per cent sucrose (pol)}} = \text{Glucose ratio}$$

This coefficient is useful in detecting inversion of sucrose in the manufacture. Provided no sucrose has been separated from the material and no reducing sugars have been removed or destroyed, an increase in the glucose ratio indicates inversion.

It is possible that by the destruction of both sucrose and reducing sugars the relations between the two sugars might remain the same and yet inversion have occurred. (See Sec. 826.)

755. Saline Coefficient. Ash Ratio. Sucrose (Pol)—Ash Ratio. The saline coefficient is the quantity of sucrose (pol) per unit of ash.

$$\frac{\text{Per cent sucrose (pol)}}{\text{Per cent ash}} = \text{Ash ratio}$$

756. Molasses Factor. This is the ratio between the sucrose (pol) lost in molasses and the non-sucrose (non-pol) entering the house in the juice.

Molasses factor =

$$\frac{100 \times \text{Sucrose (pol) in molasses \% cane}}{(\text{Bx mixed juice} - \text{Sucrose (pol) mixed juice}) \times \text{Mixed juice \% cane}}$$

or expressed in another way,

$$\frac{100 \times \text{Sucrose (pol) in molasses \% cane}}{\dfrac{100 \times \text{Sucrose (pol) Extracted \% cane}}{\text{Purity mixed juice}} - \text{Sucrose (pol) Extracted \% cane}}$$

It is evident that in the latter arrangement of the formula the purity of the mixed juice will be apparent purity if pol figures are used, or gravity purity if Brix and sucrose are used. The ISSCT recommends reporting the molasses factor calculated with sucrose figures and gravity purities, but factories that work on a polarization basis may continue to use this factor as heretofore.

This is a valuable figure in comparing the molasses work of different factories since it takes into account the percentage of molasses made and the sucrose (pol) content of that molasses, in relation to the amount of molasses-making material (non-sugar) entering the factory. It is quite evident that all these factors must be considered for a true comparison.

What the molasses factor really represents is the pounds of sucrose (pol) going to final molasses for each 100 lb. of soluble non-sucrose (non-pol) entering the boiling house; the lower the figure, the better the work. For Cuba, figures from 30 to 40 are common. The molasses factor fails to take into account the impurities going into the raw sugar. For this reason, the factor gives strictly comparable figures only where raws of the same test are turned out. Obviously a factory making raws of 97° test will make more molasses than one turning out 96° test raws (all other conditions being equal).

757. Recoveries and Yields. The ISSCT Committee calls attention to the fact that the subject of recoveries and yields is in a state of confusion both as to terms used and methods of calculation in various countries. In the separation of these terms they have clarified the situation somewhat, but as will be discussed below the selection of terms seems of doubtful value, particularly the elimination of the term retention which is so widely understood and accepted in the western hemisphere.

758. Yield. The yield is defined by the ISSCT as the quantity of sucrose (pol) or standard sugar based directly on the cane. This is the generally accepted definition in Louisiana and Cuba as well as elsewhere.

759. Boiling House Recovery (ISSCT). Retention. This is the percentage of sucrose (pol) in the mixed juice that is recovered (or retained) in the commercial sugars.

This figure is almost universally known as retention in the United States and the West Indies. Formerly, the term "recovery" was used by some factories to mean the same as retention, but this practice is now quite rare in this hemisphere and "recovery" is understood to mean the same as overall recovery defined below.

760. Overall (Total) Recovery (Sucrose or Pol) ISSCT. This is the sucrose (pol) recovered in commercial sugars per cent sucrose (pol) in cane. It is therefore the boiling house recovery (retention) multiplied by sucrose (pol) extraction.

761. Discussion of Terms. It is to be regretted that the ISSCT Committee discarded the term "retention" (which has a definite and unequivocal meaning) and substituted the more cumbersome "boiling house recovery" when "recovery" has a well-recognized different meaning in at least half of the world. It may be argued that "boiling house recovery" and "overall recovery" cannot be misunderstood if the full terms are used, but the substitution of expressions of two or three words for single words is not likely to meet with general favor. (Translated into Spanish the newer terms become still more complicated.) "Retention" and "recovery" are therefore likely to continue in use in the Americas in the same sense that they have been used heretofore. It is of interest that Noël Deerr in personal correspondence with E. M. Copp accepts the terms "retention" and "recovery" as preferable to his own terms "boiling house recovery" and "overall recovery" which the Committee on Uniformity recommends. In the following example of a sucrose (pol) balance 92.32 is the boiling house recovery according to ISSCT definition, or retention as it is termed in Louisiana and the West Indies:

Sucrose (pol) in the mixed juice, per cent		100
Sucrose (pol) in the sugar per cent sucrose (pol) in the extracted juice	92.32	
Sucrose (pol) in the molasses per cent sucrose (pol) in the extracted juice	6.58	
Sucrose (pol) in the press cake per cent sucrose (pol) in the extracted juice	0.44	
Sucrose (pol) undetermined per cent sucrose (pol) in the extracted juice	0.66	
		100

CHAPTER 35

CHEMICAL CONTROL[1]

762. Factory Organization. The organization of most raw sugar factories consists of a manager, who has general supervision and responsibility for the entire property, plant, and process; a chief engineer and assistant engineers, who are responsible for the milling of the cane, the boiler and power plants, and the mechanical equipment throughout the factory; a superintendent and assistant superintendents, who are responsible for the sugar making from the time the juice leaves the mills; and a chief chemist and assistant chemists, who have charge of the chemical and technical control of the entire factory. The chemists and laboratory are generally under the immediate direction of the superintendent, but the laboratory carries out the mill control as well as that of the rest of the factory.

Since the operation of the factory is usually on a twenty-four-hour basis, the assistant engineers, assistant superintendents, and assistant chemists must work in shifts and are sometimes known as shift engineers, etc. Two detailed articles have appeared on the duties of these men directly in charge of operations, one by Murdoch[2] on the duties of the shift engineer, and the second by Waddell[3] on the duties of the assistant superintendent (called shift chemists in this article, the laboratory assistants being called bench chemists). These articles are worthy of study by men engaged in this type of work.

763. Duties of the Chemist. The chemist in the factory corresponds to the auditor in the accounting department. He charges the superintendent with the sucrose entering the factory in the raw material and credits him with that leaving it in the products, by-products, and losses. It is his duty to trace the travel of the sucrose and locate losses.

The chemist is also the statistician of the factory. He reports the quantity of the raw materials, the analysis of the materials in process of manufacture and of the products and by-products. The chemist prepares statistics that have a bearing upon the control and economy of the manufacture and the business of the establishment.

Research and investigation work in connection with the improvements in the equipment or processes is often called for, and this necessitates both technical and chemical training on the part of the chemist.

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[1] Thanks are due Mr. Earle M. Copp of Petree & Dorr Engineers, Inc., for valuable suggestions in the preparation of this chapter. (G. P. M.)

[2] *Intern. Sugar J.*, January, 1932, p. 22.

[3] *Intern. Sugar J.*, September, 1932, p. 342.

The laboratory should be a part of the training school for future superintendents. The chemist becomes acquainted with all the methods, problems, and difficulties of the manufacture. His control of the work should familiarize him with the details of the processes. The chemist's training unfortunately lacks the opportunity for practice in directing and controlling labor. This must come to him before he can leave the school of the superintendent. A good working knowledge of pan boiling is also desirable.

A lesson that must be learned early in factory control is that samples must be representative of the materials, and their integrity must be unquestionable. Apparatus and methods must be adapted to the work in hand. Where the highest feasible accuracy is required, as in the testing of the raw materials and the products, no detail should be omitted or labor spared that may lead to more dependable results.

The chemist must judge from the equipment of the factory what control work may be omitted without decreasing the efficiency of the sugar house. He must under all conditions determine when he may properly, to some degree, sacrifice accuracy for the sake of promptly obtaining approximate figures for immediate use.

It is just as important that the necessary chemical control be complete and the laboratory records well arranged as that the financial accounts of the various departments be full and accurate.

764. Chemical Control. The chemical and technical control of the sugar factory has a threefold purpose: (1) to guide the operations as they are being carried out in such a way as to insure the best practical results, (2) to provide figures which will indicate the extent of the losses in the factory and to aid in detecting these losses, and (3) to accumulate data which will allow the work of the factory during one period to be compared with that of the other periods, either day, week, month, or year. An extension of this third purpose is to provide figures for comparison with other factories owned by the same company, or with other factories in the same country, or with factories throughout the world.

The first of these purposes may be termed the routine or running control, and the methods used must be simple and rapid. The accuracy of the methods need only be such as to give comparative or indicative results satisfactory to those directing the operations controlled. The second purpose requires methods of such accuracy that they will not give misleading results, either one way or the other, but here again the results need only be accurate from the comparative standpoint and to the satisfaction of the management. The third purpose, particularly if comparison between groups of factories is required or if internationally comparable figures are desired, requires that the work be done by accepted and uniform methods. For this reason, most sugar-producing countries have published methods of chemical control and more recently the International Society of Sugar Cane Technologists has issued their *System of Cane Sugar Factory Control,* edited by Zerban (New York, 1942), which is intended to systematize the control methods for all countries. These methods are largely based on those used in the eastern hemisphere and their adoption by western hemisphere factories may be delayed because of this.

765. Basis of Factory Control (Pol or Sucrose). The routine or running control figures are always based on polarization and Brix, and in many factories these are the only figures that are determined. For all published control figures for comparative purposes the International Society of Sugar Cane Technologists recommends sucrose ("true sucrose," Clerget) and Brix (not solids by drying). Spencer had the factories under his control determine both apparent and true figures, i.e., polarization and Brix, and sucrose and solids by drying. It is essential that control figures be correctly designated as to whether they are based on polarization or sucrose determinations.

By far the greater number of sugar factories in Louisiana and the West Indies (and possibly generally) depend upon polarization figures for their entire control. Nothing which is said here or elsewhere in this chapter is to be taken to mean that such a control, accurately and carefully carried out, is not of great value and dependability. Also, it should be emphasized that any system of control or any control figures may be on the polarization basis even though the International Society of Sugar Cane Technologists specifies sucrose. This point will be referred to again when specific instances arise.

The advantages and disadvantages of the use of polarization or sucrose figures have been discussed fully by Noël Deerr,[4] and his views coincide with those of the present writer. Polarization figures have the advantage of simplicity and rapidity, but they do not give as true a balance as do sucrose figures, which should be used for comparative control purposes where trained chemists are available to carry out the more complicated and difficult double polarization determinations accurately. The polarization figures are always determined and therefore the sucrose control figures are in addition to the pol figures and not a substitute for them. Under no circumstances should the control be on a mixed basis, i.e., using polarization in juice and sucrose in molasses. This latter method (advocated to some extent in Java) is apparently based on the idea that the difference between polarization and sucrose is small in the analysis of the juice, but it is large in the analysis of the molasses. Obviously, when the weights of the pol and sucrose in the juice and in the molasses are considered it will be found that the difference is as appreciable in the larger volume of the juice as in the smaller volume of the molasses.

766. Outline of Factory Control. The control is separated into two sets of figures, the milling control and the boiling house control. Relatively few basic figures are required. The International Society of Sugar Cane Technologists[5] gives the fundamental equation for mill control as "cane plus water equals mixed juice plus bagasse," and the ideal system of control would deter-

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[4] *Intern. Sugar J.*, August, 1936, p. 301.

[5] *Factory Control*, p. 9.

mine the weights of these four materials directly. However, no economical method for weighing the bagasse on a full factory scale has been developed, although reports of progress in this direction are made from time to time.

In most modern factories the other three components of the fundamental equation, cane, imbibition water, and mixed juice are all weighed directly, and the weight of bagasse is calculated from the equation by difference.

In some countries, notably Hawaii, where the cane cannot be weighed directly because of fluming or for other reasons, an inferential method is used, the bagasse per cent cane being calculated from the fiber in cane (directly determined) and the fiber in bagasse, and the weight of cane then is calculated.

Where settlings are returned to the mills (Sec. 112) or any part of the clarification juices recirculated (as in compound clarification, Sec. 110) the cold mixed juice cannot readily be weighed so the weighing is done on the hot clarified juice. From the analysis and weight of the mixed juice (or clarified juice in the special cases noted) and the analysis and (calculated) weight of the bagasse, the analysis of the cane in most systems of control is calculated. Direct analysis of the cane (except for fiber in the inferential method) is not generally carried out.

For the control of the boiling house, the basic figures are the weight and analysis of the mixed juice (or of the clarified juice if the mixed juice cannot be weighed), the weight and analysis of all sugars turned out, the weight and analysis of the final molasses, and the weight and polarization of the press cake. The juice weights and analysis also form a part of the mill control.

The analysis of the sirup or concentrated cane juice, the "meladura" of the Spanish-American factories, is the control of the purification of the juice and its evaporation, and is a guide in the sugar boiling. To obtain satisfactory results in pan boiling and to bring this work to as nearly a scientific basis as is possible, the analysis of the massecuites and molasses is necessary. Massecuites are usually boiled to specified purities to conform with certain boiling systems (p. 206), and careful control of this work is essential to a systematic grading of the materials for the most profitable extraction of the sugar, considering the limitations of the factory. Frequent control tests are also required in the conduct of the crystallizers. The products, sugar and molasses, are controlled both as a check upon the manufacture and to meet market conditions.

The filter press cake is one by-product whose weight may be readily ascertained. Its analysis is usually limited to the determination of the polarization, though occasionally the solids must be determined for controlling the efficiency of the pressing and the quantity of water used in sweetening off, or in reducing the cake for refiltration.

A loss of sugar may occur through entrainment in the multiple effects and vacuum pans, and a check on the condenser waters and sweet waters is necessary in controlling this loss.

WEIGHTS AND MEASURES

767. General Considerations in Regard to Weights and Measures. It is quite essential that all weights and measures should be of the same system or be reduced to the same system. This remark applies especially to Spanish America, where it is not unusual to find English and Spanish units used indiscriminately.

The system of weights and measures adopted should be applied as accurately as possible. A checking system should be devised to meet the conditions of the factory and in so far as may be possible to eliminate errors in the calculations.

The International Society of Sugar Cane Technologists recommends the metric system for all international comparisons, but it recognizes the difficulties in the way of general adoption. The simplification of calculations by the use of the metric system needs no argument.

No allowance or correction for cane trash should be made in the control figures, even though deductions may be made in payments to cane growers.

768. Weight of the Juice. Most of the factories in Cuba, Puerto Rico, and Louisiana weigh the mixed juice in tank scales, hand operated. Weighing is recommended by the International Society of Sugar Cane Technologists and is becoming more general throughout the world. These scales are balanced and counterpoised tanks with conical bottoms to facilitate washing, arranged in pairs so that one is emptying while the other is filling. Inlet and outlet valves are so interlocked that they cannot be open at the same time. Counters, printing counterpoises, and other control devices are used with these scales. Recording devices, showing the number of times the weighing tanks have been filled and emptied, are an added precaution in quite general use. A factory grinding about 2000 tons of cane per day requires two tank scales, each holding about five tons of juice.

On the accuracy of the weight of the mixed juice depends the entire control of the cane factory and extreme care should be taken at this station to avoid even small errors, particularly those which may tend to become cumulative. With those clarification processes which recirculate part of the juice or muds the juice weights must start with the clarified juice (see Sec. 770).

Automatic Juice Measurement. The Foxboro automatic juice measurement system (Fig. 198) does not actually weigh juice in pounds, but it controls, measures, and records the head of juice in a calibrated tank, directing the flow from the mills to the measuring tanks, automatically filling them and dumping them, and making a chart record of each tankful. Since the level of the juice in the tank at time of emptying is regulated by the head, differences in density are compensated for, and the tank delivers the same weight of juice for a given setting of the instrument. The weight of this standard tankful has previously been determined, so that no record-keeping is involved except to multiply this known weight by the number of tankfuls shown on the chart each day. If there are several mills a separate system for each unit and its dependent tanks is usually desirable.

The airflow or bubble pipe principle of measurement is employed. A constant stream of air delivered at constant pressure to an open-end pipe immersed in the juice meets an increasing back pressure as the juice level rises, until at the predetermined level the back pressure causes control action and the operation of the controlled valves. Operation is continuous and completely automatic. Davies, Yearwood, and Tivy[6] found the Foxboro equipment to give very close agreement with actual weighing at the Experimental Factory of the Imperial College of Agriculture in Trinidad. Many installations are in use in Louisiana and are giving satisfactory results.

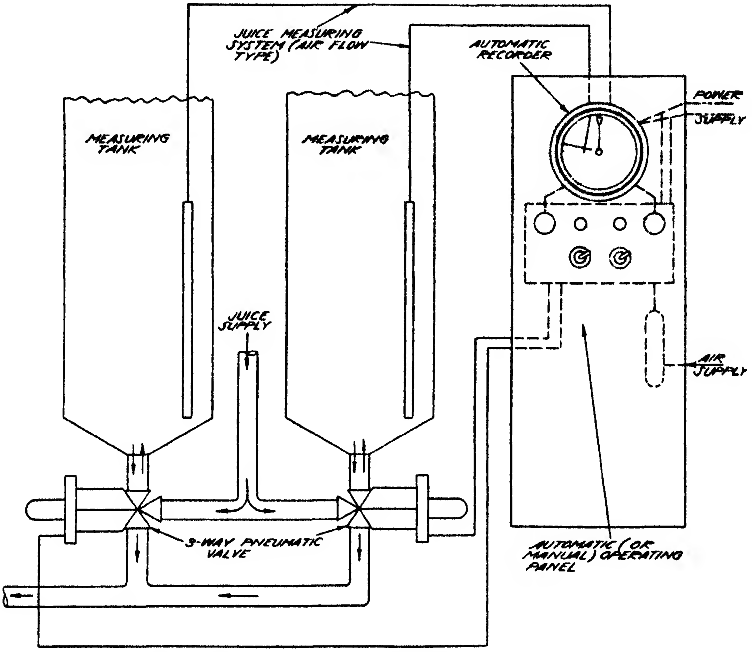

FIG. 198. Foxboro Juice-Measuring Device.

769. Measurement of the Juice and Calculation of Its Weight. The measurement may be made with a fair degree of accuracy in factories using separate liming tanks, though the results are never as satisfactory as with juice scales, and the practice should be avoided wherever scales can be installed. At least three tanks are required, and these should be connected with a much deeper tank, serving as a pump tank. The pump tank must be deep enough to admit of complete drainage of the liming tanks. The tank

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[6] *Intern. Sugar J.*, January, 1943, p. 23.

valves should be well ground to prevent juice from leaking from a full to an empty tank. All the tanks should have conical bottoms and spray pipes for washing. An overflow pipe should connect each tank with a trunk line leading back to the mills. The overflow pipe of each tank should extend into the tank a short distance horizontally, ending in a T with the cross bar of the T in the vertical position. The upper outlet of the T should be extended by nipples well above the foam on the surface of the juice and the lower outlet should extend a few inches below the surface. This arrangement provides a free overflow for the juice, without interference by foam. A 3-in. T is a suitable size.

The measuring tanks should be calibrated with water under service conditions. Corrections must be applied to the measured volume for temperature, for milk of lime added and for air entrained with the juice. The allowance for air should be determined experimentally. It varies with milling and pumping conditions. A tank should be filled to the overflow with juice and its temperature be noted. After a few hours the temperature and shrinkage of the juice should be noted. A factor should then be figured from these data, making allowance for change due to temperature. It is advisable to add formaldehyde to the juice to insure its preservation and to make the period of rest as long as is practicable. Occasionally factories warm the juice moderately on its way to the liming tanks. This reduces the error due to entrainment of air. The method of calculating the weight of the juice from its volume is given on p. 641.

770. Weighing Clarified Juice. It has already been noted that with compound clarification or the return of muds to the mills the cold mixed juice cannot be weighed readily. The clarified juice should be weighed where these conditions prevail, and this can best be done by tank scales similar to those described for the weighing of cold juice. The automatic device of Foxboro (Sec. 768) has been successful in this type of weighing.

771. Weighing Imbibition (Saturation, Maceration) Water. Tank scales should be used for this purpose, similar to those used for weighing juice, and the same precautions should be observed to insure accurate weights. The weight of imbibition water is one of the components of the fundamental equation of mill control and the weight must be accurate if the mill control is to be exact. Care should also be exercised to see that no water other than that weighed as imbibition water enters the juice during milling. It is evident that any unweighed water (such as water to cool mill journals, wash water on mills, etc.) that enters the juice will increase the weight of mixed juice and give a false decrease in the weight of bagasse, which is calculated from cane weight plus water minus juice weight.

772. Weight of Bagasse—Calculated versus Actual. There is not available a ready method of weighing the bagasse of the factory although such weights are recognized as very desirable. Methods have been suggested from time to time but these have not proved practicable. The usual method of calculating the weight of bagasse is from the fundamental equation of milling, i.e., cane plus water equals juice plus bagasse. Three of the components of this formula are ordinarily weighed (or the weights of the liquids calculated from the volumes), and the bagasse obtained by difference, but there are recognized objections to this procedure. There is unquestionably loss of water by evaporation, especially in tropical countries and where hot water is used. Furthermore, unweighed water used in cooling journals and in washing down mills may enter the juice at the mills. Of course, these two errors (evaporation and added water) are counterbalancing, but the second is to a great extent avoidable, whereas the evaporation is not.

Experimental weighing of all four components has been reported recently by Davies, Yearwood, and Tivy[7] at the Experimental Factory of the Imperial College of Agriculture in Trinidad. They found a much greater loss in weight (using cold water imbibition) than they had expected. The figures show 11.43 per cent more bagasse calculated (from the fundamental equation by difference) than was found as bagasse weighed. These figures are such as to cast doubt on the accuracy of the ordinary control methods where the bagasse is weighed.

As against these figures we have the following from the largest factory in Louisiana for a year in which all bagasse was sold to the Celotex Company and weighed as produced. Cane, juice, and imbibition water were also all weighed.[8]

	Bagasse
Tons of cane ground 92,982	
Mill weights (cane plus water minus juice)	25,757 tons
Weight on Celotex track scale	25,357 "
Difference	400 "
Difference % of factory weights	1.55%

Obviously these figures are within the limits of error for factory measurements, and the indication is that the mill equation serves for control purposes, but this may be due to a counterbalancing of errors as mentioned above. The Louisiana crop is ground during the colder months which may have an appreciable effect on evaporation, as compared to that under tropical conditions.

773. Measurement of Water. Where conditions will not permit weighing the water for imbibition, it may be measured and the weight calculated, allowance being made for the temperature of the water. Measurement is not recommended by the International Society of Sugar Cane Technologists. Water meters are not sufficiently reliable for the work.

774. Measurement and Weight of the Sirup. The weight of the sirup is not usually required, except in taking account of the stock of the material in process. The sirup must be measured occasionally in taking account of the sugar in process for the run reports. For this purpose the tanks should

[7] *Intern. Sugar J.*, January, 1943, p. 79.

[8] Personal communication, W E. McFarland, Reserve, La.

be gaged, and the volume per inch of depth should be tabulated. In these measurements it is more convenient to note the "inches out" and figure the sirup in the tank by difference.

775. Measurement and Weight of the Massecuites. The measurements in the mixer and especially in the crystallizers should be made immediately on striking the massecuite, on account of its increase in volume after stirring or further crystallization. The measurement or weight of the massecuite is usually required only at the end of a run or period for calculating the quantity of sugar in process of manufacture.

A sample of the massecuites should be taken as discharged for analysis for use in making the necessary calculations and for the guidance of the pan boiler in the conduct of the pan work.

776. Sugar Weights. The accurate weighing of the raw sugar is of equal importance with the juice and cane weights, and this phase of the technical control should be under constant supervision both by the laboratory and the factory superintendent.

Dependable automatic scales, of a type similar to those that have long been used in the weighing of refined sugar, are now generally employed for weighing raws. These use a mechanical arrangement to regulate the flow of the sugar into the weighing hopper and to adjust the dribble, or last small quantity. One type of these scales will accurately weigh more than 100 bags (325 lb.) of raw sugar per hour. Because of the sticky nature of raw sugar the scales should be cleaned frequently and the weight delivered by the automatic scales should be checked frequently by reweighing on ordinary platform scales. The International Society of Sugar Cane Technologists advises checking the weight of sugar by weighing the loaded sugar cars where this can be done.

777. Measurement of the Molasses. The measurement of the molasses is accompanied by difficulties arising from the nature of the material itself. Molasses is very viscous and drains slowly from the containers, making successive measurements in a tank uncertain. Further, heavy molasses occludes considerable quantities of air. This occlusion of air raises the question of a definition of the commercial gallon of final molasses. (See Sec. 657.)

778. Weight of Final Molasses. Final molasses should be weighed in tank cars when possible, on account of its importance in the factory control. A "wantage" table is given on p. 690 for the measurement of the molasses in horizontal cylindrical tanks (tank cars). The amount of air occluded varies with the container, the method of filling it, and the period that elapses between filling and measuring. As has been stated (Sec. 658) a molasses may weigh less than 11 lb. per U.S. gallon, measured immediately after filling a tank car, whereas the air-free weight of the same molasses is 12 lb. This indicates the importance of experimental data as a basis for calculating the weight of the molasses in meeting local conditions. A float measurement, using a copper float, may be employed for molasses stored in very large tanks.

The International Society of Sugar Cane Technologists recommends weighing final molasses in scale tanks of large capacity, tare weights being taken after each weighing. Measurement of molasses for control purposes is not advisable. The Foxboro equipment described in Sec. 768 is being successfully used in Louisiana for molasses weights.

Pneumercator Attachment for Molasses Tanks. The Pneumercator is a patented device for weighing the material in a tank by means of the pressure on a delicately adjusted air chamber communicating with a mercury gage calibrated for each particular tank. The gage is marked to register in any convenient unit; pounds or tons being the usual markings. The hydrostatic pressure of the molasses on the air chamber is the actuating force so that all question of foam and allowance for occluded air is eliminated. Many checks on this device have been made against actual weighings of Cuban molasses, and the agreement is highly satisfactory. The Pneumercator has eliminated the difficulties enumerated above concerning the measurement of molasses.

This device has been found valuable for weighing molasses in refinery control work. The degree of accuracy is dependent on the size of the tank; the smaller the diameter, the greater the accuracy.

MILL CONTROL FIGURES

779. Fundamental Equation. As has already been pointed out, the basis of the mill control is the fundamental equation, cane plus imbibition water equals mixed juice plus bagasse, but the actual control is by no means as simple as this relationship would imply. In fact, there is wide difference of opinion on the methods to be employed, and no matter which methods are chosen some assumptions must be made which are open to question.

The fundamental equation offers the difficulty that the bagasse cannot be weighed and must be obtained by difference where the other three constituents are weighed. But undoubtedly water evaporates from the bagasse in its course through the mills, and water used for other purposes than imbibition (to cool bearings, wash down mills, etc.) may enter the juice without being weighed. This point has been considered in detail in Sec. 772.

780. Inferential Methods. The inferential method, used where the cane is not weighed, depends on the direct determination of the fiber in cane, which involves cane sampling, an operation of doubtful accuracy at best, particularly if canes of different varieties and from different localities are being ground (see Sec. 602).

781. Composition of Juices; Colloidal Water. The subject of great controversy and the one on which assumptions must be made is the composition of the juice as it exists in the cane, or the composition of the extracted juice, excluding that portion of the imbibition water which enters into and dilutes the mixed juice. The presence of colloidal water or uncombined or undetermined water in the cane is generally recognized, although the percentage is variously estimated.

The International Society of Sugar Cane Technologists control methods ignore the undetermined water entirely and consider the juice in the cane, under dry milling conditions, to be the difference between the weight of cane and the weight of dry fiber. This "does away with any assumptions regarding the undetermined water and its behavior during the milling process" and greatly simplifies the milling control. It also does away with the need for a milling factor which is used in the older methods of control generally employed in the West Indies and Louisiana, the use of which is explained below.

CUSTOMARY METHODS OF CONTROL IN THE WESTERN HEMISPHERE

782. Mill Extraction. The quantity of juice or sugar removed from the cane by the mills, i.e., the mill extraction, is usually expressed in two ways in the older methods of control.

a. Extraction in Weight Terms of the Cane and Undiluted Juice (*Juice Extraction*). Divide the weight of mixed juice (see p. 592) by the weight of the cane and point off percentage to ascertain the per cent dilute extraction. Calculate the dilution per cent cane (Sec. 783) and subtract this number from the per cent dilute extraction. The remainder is the extraction of (undiluted) juice per cent cane. This figure, the so-called normal extraction, is now rarely used as it is recognized that it has little value or significance.

b. Extraction in Percentage Terms of the Weight of Sucrose in the Cane and in the Extracted Juice (*Sucrose Extraction*). Calculate the weights of sucrose (pol) in the mixed juice and in the bagasse. The weight of the bagasse is ascertained by subtracting the weight of the mixed juice from the sum of the weights of the cane and imbibition water. The sum of the weights of sucrose (pol) in the extracted juice and the bagasse is the weight of sucrose (pol) in the cane. (Pol in bagasse is accepted as the same as sucrose.) One hundred times the sucrose (pol) in the extracted juice divided by the weight of sucrose (pol) in the cane gives the extraction number in terms of the sucrose (pol) in the extracted juice per cent sucrose (pol) in the cane.

This sucrose extraction, or more simply the extraction, is generally on a polarization basis. The International Society of Sugar Cane Technologists recommends that extraction be on a sucrose basis for international comparison. The figure calculated from polarization should be reported as "Extraction (Pol)."

The extraction is still looked upon by many as a measure of mill performance, but it is now recognized that the figure is greatly influenced by the composition of the cane, particularly as to sucrose and fiber content. For this reason other figures, based on the sucrose (pol) left in the bagasse per cent fiber and other similar relationships, have been developed and will be discussed later. (See Sec. 792 *et seq.*)

783. Use of Milling Factor. In the method of calculating extraction outlined above it is necessary to establish a relationship between the Brix of the crusher juice (first expressed juice) and the undiluted juice extracted from the cane. This is done by assuming that the undiluted juice and the mixed juice extracted by dry milling, i.e., operating the mills without adding any imbibition water, have the same composition. The method of sampling is given in Sec. 613. In the example there given the crusher juice had a Brix of 20, the mixed juice by dry milling a Brix of 19.4; therefore, the milling factor is 19.4 divided by 20, or 0.97. In the example for calculating the juice extraction the crusher juice has Brix of 19.21, and when this factor of 0.97 is applied the Brix of the undiluted juice is 19.21 × 0.97 = 18.63. Before discussing the objections to the milling factor we will give an example of the calculations for extractions by the above method.

Example of calculating the extraction.

Tons of cane ground	3254	
Tons of mixed juice extracted	3450	
Tons of imbibition water applied to the bagasse	877	
Degree Brix of the crusher (first expressed juice)	19.21	
Factor for reducing the Brix of the first expressed juice to that of the undiluted juice (see above)	0.97	
Degree Brix of the mixed juice	14.52	
Per cent sucrose (pol) in the mixed juice	11.98	
Per cent sucrose (pol) in the bagasse	2.90	
1. Brix of the undiluted juice = 19.21 × 0.97		18.63
Dilution per cent mixed juice = 100(18.63 − 14.52) ÷ 18.63 (see Sec. 787)		22.06
Mixed juice extraction = 3450 ÷ 3254 × 100		106.02
Dilution per cent cane = 106.02 × 0.2206		23.39
Mill extraction and the diluted juice per cent cane = 106.02 − 23.39		82.63
2. Weight of bagasse = (3254 + 877) − 3450		681 tons
Sucrose (pol) in the bagasse = 681 × 0.029		19.749 tons
Sucrose (pol) in the extracted juice = 3450 × 0.1198		413.31 tons
Sucrose (pol) in the cane = 413.31 + 19.749		433.059 tons
Mill extraction, sucrose (pol) extracted per cent sucrose (pol) in cane 413.31 ÷ 433.059 × 100		95.44
Résumé:		
Mixed juice extraction per cent cane		106.02
Dilution per cent cane		23.39
Extraction, undiluted (normal) juice per cent cane		82.63
Extraction, sucrose (pol) extracted per cent sucrose (pol) in the cane		95.44

784. Objections to Milling Factor. The milling factor is open to several objections: (1) Even admitting that it represents the relationship between first expressed juice and undiluted juice at the time of the determination, it certainly does not represent that relationship when cane and other conditions are changed, e.g., varieties, maturity, and rates of grinding. (2)

The determination cannot be made every time conditions change, and the tendency is to continue to use the same figure under all conditions, i.e., an arbitrary factor is adopted. (3) Dry milling may not give the correct factor at all.

This latter contention is raised and fully argued by E. M. Copp[9] who shows that dry milling extracts little or no juice from the last mills of a tandem. He recommends "juice maceration" (imbibition) to give the true Brix relationship desired. Juice imbibition consists of substituting for the imbibition water a part of the mixed juice, continuing the return of other juices to the mills as in usual practice, until the Brix of the juice from the last roll is approximately the same as that of the mixed juice (undiluted) going to the scales. Copp's figures show that under these conditions there is a much greater difference between the Brix of the first expressed juice and the mixed juice than there is with dry milling. Instead of the usual mill factor of 0.97–0.98 obtained with dry milling, he finds factors close to 0.95 with juice imbibition.

The determination of the factor with juice imbibition does not dispose of the other objections, viz., that the factor will change with changing conditions and the tendency will be toward a fixed or arbitrary factor. For these reasons, the International Society of Sugar Cane Technologists control which does away with the need for any milling factor has definite advantages over the older methods.

785. Dry Milling Extraction Number. (*For Mill Adjustments.*) Considerable information of value concerning the performance of the various mills in a tandem may be obtained from the analysis of the bagasse as it passes from one mill to the next without imbibition. A section of the bagasse blanket is marked in any convenient way (a boundary of dark oil, colored stain, lime, or the like), and samples are taken from this section as it emerges from the different mills. The bagasse from each mill is analyzed. The extraction of each mill may be calculated from the fiber as follows:

$$\text{Juice extracted \% cane} = \frac{\text{\% fiber in bagasse} - \text{\% fiber in cane}}{\text{\% fiber in bagasse}} \times 100$$

This calculation gives the total amount of juice extracted up to and including the mill unit under observation. The juice extraction for each mill may be found by subtraction.

Milling with juice imbibition, as described by Copp would seem to approximate true milling conditions more closely and would therefore be a more reliable guide in this type of work.

786. Imbibition (Saturation, Maceration). The quantity of water used in saturating the bagasse can only be determined with certainty by weighing or by accurate measurement. An estimation of the water is sometimes made by an inferential method similar to that used in estimating the weight of the

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[9] *Facts About Sugar,* March 24, 1928, p. 280.

cane. Inferential methods for the water are open to the same objections as those for the weight of the cane.

The water should be accurately weighed and its percentage on the cane calculated, or (if weighing cannot be done) it should be measured and calculated to weight. The percentage should be in terms of the weight of the cane.

$$\text{Imbibition \% cane} = \frac{\text{Weight of water}}{\text{Weight of cane}} \times 100$$

787. Per Cent Dilution. The dilution water has already been defined as that part of the imbibition water that goes into the mixed juice, but the per cent dilution differs with different methods of control, depending on the basis to which it refers. It is therefore essential to state the basis on which the percentage dilution is figured, e.g., Dilution per cent cane or Dilution per cent mixed juice.

In the older methods of control now under discussion the dilution per cent cane is reported, and this is the figure in general use in Cuba, Puerto Rico, and Louisiana. It is calculated from the Brix of the undiluted (normal) juice (as obtained from the first expressed juice by the milling factor) and the Brix of the mixed juice (dilute juice) as follows:

$$\frac{\text{Brix undiluted juice} - \text{Brix mixed juice}}{\text{Brix undiluted juice}} \times 100 = \text{Dilution \% mixed juice}$$

$$\text{Dilution \% mixed juice} \times \text{Mixed juice \% cane} = \text{Dilution \% cane}$$

These calculations are shown in the example given in Sec. 783 and the derivation of the formula is given in Sec. 832.

As will be shown later the International Society of Sugar Cane Technologists reports the "dilution % absolute juice." This is the Hawaiian practice, whereas Queensland (Australia) calculates "dilution % undiluted juice," in the same manner as "dilution % mixed juice" is calculated above.

788. Inferential Methods. Where the cane cannot be weighed for various reasons, an inferential method of arriving at the weight has been used. This method involves the direct determination of fiber in cane, which as has already been pointed out (Sec. 602) is of doubtful accuracy because of the difficulty of obtaining a representative sample of cane. Also, the weight of imbibition water may be calculated by the inferential method, assuming that the cane is weighed. This latter method is given by the International Society of Sugar Cane Technologists as an alternative to weighing the imbibition water and the calculations are as follows:

$$\text{Bagasse \% cane} = \frac{100 \text{ Fiber \% cane}}{\text{Fiber \% bagasse}}$$

The weight of bagasse can then be calculated from the weight of cane. The weight of imbibition water (from the fundamental equation) is the difference between the weight of cane and the combined weight of mixed juice and bagasse. From there on, the control calculations are the same as in the methods already given.

The inferential method used in Hawaii, wherever cane weights cannot be taken because the cane is flumed to the mill, is based on the weight of the mixed juice only, and direct determination of fiber in cane. The bagasse per cent cane is calculated as above from fiber per cent cane and fiber per cent bagasse. A milling factor from dry milling is used to obtain Brix absolute juice. The steps to obtain the weight of cane are as follows.[10]

$$\text{Juice extraction \% juice in cane} = 100 - 100 \times \frac{\text{Bagasse \% cane} \times \text{Brix \% bagasse}}{\text{Brix absolute juice } (100 - \text{fiber \% cane})}$$

$$\text{Purity absolute juice} = \frac{\left(\begin{array}{l}(\text{Juice extraction \% juice in cane} \times \text{Purity mixed juice}) + (100 \\ -\text{Juice extraction \% juice in cane}) \times \text{Purity last expressed juice}\end{array}\right)}{100}$$

$$\text{Pol \% absolute juice} = \frac{\text{Brix absolute juice} \times \text{Purity absolute juice}}{100}$$

$$\text{Pol \% cane} = \frac{\text{Pol \% absolute juice} \times (100 - \text{Fiber \% cane})}{100}$$

$$\text{Extraction \% cane} = \text{Pol \% cane} - \text{Pol in bagasse \% cane}$$

$$\text{Extraction (pol)} = \frac{\text{Extraction \% cane} \times 100}{\text{Pol \% cane}}$$

$$\text{Weight of pol in mixed juice} = \frac{\text{Weight mixed juice} \times \text{Pol \% mixed juice}}{100}$$

$$\text{Weight of pol in cane} = \frac{\text{Weight of pol in mixed juice} \times 100}{\text{Extraction (pol)}}$$

$$\text{Weight of cane} = \frac{\text{Weight of pol in cane} \times 100}{\text{Pol \% cane}}$$

Other inferential methods in which neither cane weights nor juice weight are available are also given in the Hawaiian *Methods of Chemical Control* (pp. 75–76) and may be consulted by those interested. Obviously, such methods cannot have the validity of those based on direct weighing of the cane, water, and juice.

ISSCT METHODS OF MILL CONTROL

789. Figures Required. Reference has already been made (Sec. 726) to the published *System of Cane Sugar Factory Control* of the International

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[10] *Methods of Chemical Control,* Hawaii, 1931, pp. 74–75.

Society of Sugar Cane Technologists. The Society recommends the following figures for judging milling results: extraction, milling loss, absolute juice in bagasse per cent fiber, reduced extraction, and undiluted juice in bagasse per cent fiber. Many of these figures have been in use in the older methods of control, although some are new to the western hemisphere. It is generally agreed that no single figure can be used as a measure of milling results. The International Society of Sugar Cane Technologists says that the number of criteria figures now specified may be reduced later if experience shows this to be advisable.

790. Extraction. There is no difference in the method of calculation of the extraction (sucrose or pol) from that already described (Sec. 782). The International Society of Sugar Cane Technologists recommends that the extraction be on a sucrose basis, for figures for international comparison, but recognizes that many factories do not determine true sucrose in the various products and so must report on a pol basis. The basis should be stated, e.g., "Extraction (pol)."

791. Fiber in Bagasse. The percentage of fiber in the bagasse enters into the milling figures which follow. The International Society of Sugar Cane Technologists continues to use the method in general use (Sec. 716). The last expressed juice is considered as the residual juice in the bagasse (although this is known to be in error) and the Brix of the bagasse is calculated as follows:

$$\text{Brix \% bagasse} = \frac{\text{Pol \% bagasse} \times \text{Brix last expressed juice}}{\text{Pol last expressed juice}} \qquad (1)$$

$$\text{Fiber \% bagasse} = \text{Dry substance \% bagasse} - \text{Brix \% bagasse} \qquad (2)$$

It will be seen that (1) may also be written

$$\frac{\text{Pol \% bagasse} \times 100}{\text{Purity last expressed juice}}$$

which is the form usually given in the older methods of control.

792. Milling Loss (ISSCT). This figure, in quite general use in Louisiana and the West Indies as "Sucrose (pol) in bagasse % fiber," is what the name implies: the percentage ratio between the sucrose (pol) per cent bagasse and fiber per cent bagasse. It does not take into consideration the sucrose in the cane and therefore it is not considered as good a criterion as the figures which follow.

793. Extraction Ratio. This figure in use in Hawaii and Queensland is calculcated as follows:

$$\frac{100 - \text{Sucrose (pol) extraction}}{\text{Fiber \% cane}} \times 100$$

As with all other figures the basis (whether pol or sucrose) should be stated in reporting.

794. Reduced Extraction. It is obvious that the extraction is greatly influenced by the amount of fiber in the cane. To eliminate the influence of variations in fiber Noël Deerr proposed a formula to reduce the extraction to a common basis of 12.5% fiber in cane.[11]

The effect of variation in fiber is eliminated by calculating the absolute juice in bagasse per cent fiber as follows:

$$\text{Absolute juice in bagasse \% fiber} = \frac{(100 - \text{Sucrose (pol) extraction})(100 - \text{Fiber \% cane})}{\text{Fiber \% cane}}$$

It will be seen that the numerator in this expression is the lost absolute juice or the absolute juice in bagasse per cent cane. By the International Society of Sugar Cane Technologists definitions, 100 – extraction (sucrose or pol) is the sucrose (pol) in bagasse per cent sucrose (pol) in cane; 100 – fiber is the per cent of absolute juice; the product of these two is the absolute juice in bagasse, and this product divided by fiber per cent cane gives absolute juice in bagasse per cent fiber. Solving the above formula for extraction, we obtain

$$\text{Extraction} = 100 - \frac{\text{Absolute juice in bagasse \% fiber} \times \text{Fiber \% cane}}{100 - \text{Fiber \% cane}}$$

Then if fiber equals 12.5 per cent the formula becomes

$$100 - \frac{12.5}{87.5} \times \text{Absolute juice in bagasse \% fiber}$$

Therefore the extraction reduced to the basis of 12.5 per cent fiber in cane

$$\text{Reduced extraction} = 100 - \frac{\text{Absolute juice in bagasse \% fiber}}{7}$$

(Deerr has designated this as

$$e_{m,\,12.5} = 100 - \frac{v}{7}$$

where v equals absolute juice in bagasse per cent fiber, but the use of small letters and subscripts does not make the formula as clear as the use of the terms themselves.) The reduced extraction can therefore be calculated (either on pol or sucrose basis) from the extraction as found, and the fiber in cane.[12]

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[11] *Intern. Sugar J.*, June, 1933, p. 214.

[12] It is evident that the weight of fiber in bagasse and in cane is the same; therefore, when a quantity is calculated to percentage of fiber it is unnecessary to state "fiber in cane" or "in bagasse." However, when the fiber is calculated to per cent cane or per cent bagasse it must be so designated.

795. Absolute Juice in Bagasse, Per Cent Fiber. This figure uses Brix extraction rather than sucrose (pol) extraction as the basis:

$$\text{Absolute juice in bagasse \% fiber} = \frac{(100 - \text{Brix extraction})(100 - \text{Fiber \% cane})}{\text{Fiber \% cane}}$$

This formula is analogous to the reduced extraction formula above, with the Brix extraction substituted for sucrose (pol) extraction. The formula may also be stated in the equivalent form:

$$\text{Absolute juice in bagasse \% fiber} = \frac{10{,}000 \text{ Brix \% bagasse}}{\text{Brix \% absolute juice} \times \text{Fiber \% bagasse}}$$

The calculation for Brix per cent bagasse has already been shown in the method for calculating the fiber in bagasse (Sec. 791). The Brix of the absolute juice is calculated as follows:

$$\text{Brix \% absolute juice} = 100 \times \frac{\text{Weight of Brix in mixed juice} + \text{Weight of Brix in bagasse}}{\text{Weight of absolute juice}}$$

and

$$\text{Weight absolute juice} = \text{Weight cane} - \text{Weight fiber}$$

796. Undiluted Juice in Bagasse, Per Cent Fiber. This figure was added by the International Society of Sugar Cane Technologists because it is used in Java. No milling factor is used as the Brix of the primary juice is taken as the same as the Brix of the undiluted juice (in effect a milling factor of 1.00). The formula then becomes

$$\text{Undiluted juice in bagasse \% fiber} = \frac{10{,}000 \text{ Brix \% bagasse}}{\text{Brix \% primary juice} \times \text{Fiber \% bagasse}}$$

797. Performance Formulas Based on Mechanical Equipment. The figures recommended by the International Society of Sugar Cane Technologists express the results obtained by milling and do not take into account the milling equipment. Various formulas have been advanced for comparing and judging the results obtained according to the equipment used. The most comprehensive of these were proposed by Noël Deerr[13, 14] and may be referred to by the interested student. No formula of this type has had wide acceptance.

798. Other Control Figures in ISSCT Methods. The International Society of Sugar Cane Technologists control for dilution and other attendant figures is based on the concept of absolute juice, which by definition is cane minus fiber. The analysis of the absolute juice is then worked out as follows:

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[13] *Intern. Sugar J.*, September, 1930, p. 471.

[14] *Intern. Sugar J.*, October, 1931, p. 480.

Brix % absolute juice =

$$100 \times \frac{\text{Weight of Brix in mixed juice} + \text{Weight of Brix in bagasse}}{\text{Weight absolute juice}}$$

Sucrose (pol) % absolute juice =

$$100 \times \frac{\text{Weight sucrose (pol) in mixed juice} + \text{Weight pol in bagasse}}{\text{Weight absolute juice}}$$

$$\text{Purity absolute juice} = 100 \times \frac{\text{Sucrose (pol) absolute juice}}{\text{Brix absolute juice}}$$

Absolute juice extracted:

$$\text{Weight of absolute juice extracted} = 100 \times \frac{\text{Weight of Brix in mixed juice}}{\text{Brix \% absolute juice}}$$

$$\text{Absolute juice extraction} = 100 \times \frac{\text{Weight absolute juice extracted}}{\text{Weight of cane}}$$

$$\text{Weight of absolute juice in bagasse} = 100 \times \frac{\text{Weight Brix in bagasse}}{\text{Brix \% absolute juice}}$$

$$= \text{Weight absolute juice} - \text{Weight absolute juice extracted}$$

With the above figures at hand the other control figures are calculated as in the older methods, but absolute juice is substituted for the older normal juice (obtained by the use of the milling factor). The weight of dilution water is the weight of mixed juice minus weight of absolute juice extracted. Dilution per cent cane, dilution per cent absolute juice, and dilution per cent absolute juice extracted are all obtained by dividing the weight of dilution water by the appropriate base. Another figure not generally used in the older methods is imbibition water in bagasse per cent cane. This is obtained by subtracting dilution per cent cane from imbibition per cent cane.

799. Comparison of ISSCT Mill Control with Older Methods. A study of the International Society of Sugar Cane Technologists mill control shows that the differences between it and the older methods in general use in the western hemisphere are not great, and that the differences which do exist are along the line of simplification.

The concept of absolute juice as cane minus fiber admittedly ignores the undetermined water but the amount and distribution of such water is largely a matter of conjecture anyway. Once absolute juice has been defined as cane minus fiber, the need for a milling factor disappears. The objections to and the difficulties of the milling factor have been discussed in Sec. 784.

Some of the various milling criteria which the Society recommends, many of which have not been made use of previously, need be calculated only if international comparison is contemplated. In adopting the ISSCT system individual factories will probably report only those figures which they find useful for their own purposes. Again, the adoption of the system does not require the adoption of sucrose control, although this is recommended by the Society for comparative reports. Few factories in Louisiana and Cuba determine true sucrose on anything except molasses, but this need not be a deterrent in adopting the ISSCT methods.

Possibly the greatest drawback to many will be the unfamiliar terminology. Such expressions as "sucrose extraction," "sucrose basis for cane purchase," "sucrose loss," where polarization is meant, are deeply rooted in Cuba and Louisiana, whereas "pol" is entirely new to all but a few. The system can be adopted without insisting on the new terminology (except in published reports), and in time the newer terms will become familiar.

All things considered, it is hoped that this move toward simplicity and uniformity will be universally adopted as have, for example, the Brix scale in place of Baumé, the 20° standard temperature, and other reforms which at their inception met resistance because of unfamiliarity.

CONTROL OF THE SUGAR BOILING

800. Control of Vacuum Pans and Crystallizers. The control of the vacuum pans and crystallizers requires rapid analytical work of moderate accuracy.

The analysis of the sirup as made in the daily routine work, or in its stead the analysis of the juice, and that of the molasses indicate the quantity of the latter to be drawn into the pan to produce a massecuite of the desired purity. If a cut strike is to be boiled, the purity of the above massecuite and that of the molasses to be boiled-in, supply the data for calculating the quantity of each of these required to produce a massecuite of a certain purity. These calculations are made by the following formula, with sufficient accuracy for the purpose.

Let 100 = total weight of massecuite in the strike;
P = purity of the sirup, or, for a cut strike, that of the massecuite left in the pan;
p = purity molasses to be boiled-in;
M = purity of the required massecuite;
x = percentage by weight of that part of the strike to be formed of molasses;
$100 - x$ = percentage of the strike to be derived from sirup or from a previous boiling.

Therefore

$$x = \frac{100(P - M)}{P - p}$$

The proportions of the materials used in making a mixture of a certain purity may also be quickly calculated by the diagram method, p. 639. It is not feasible in pan work to base the calculations on actual weights. The approximate densities of the massecuite footing, for example, and the molasses to be boiled in should, however, be considered.

A sample of the mixed massecuite should be brought to the laboratory immediately the strike is dropped. A portion of this should be dissolved in water to form a solution of about 15° Brix, and its apparent purity should be determined. A second portion should be purged in a laboratory centrifugal and the purity of the molasses should be determined as above.

The labor of calculating the purity may be avoided by the use of the expanded Horne's table, p. 767.

The purity data of the massecuites and molasses samples should be promptly sent to the superintendent and the pan boiler. All mixed strikes, especially those of the lowest purity, should be controlled in this way. The relation between the purity of the massecuite and the molasses purged from it in the laboratory, immediately after boiling, is a valuable guide in boiling low-purity mixed strikes. A convenient centrifugal is shown in Fig. 199, and a filtering device in Fig. 200. This filter is inexpensive and very efficient. It is a copper funnel, separable at the ground joint, *A*, to facilitate cleaning. The filtering surface is of centrifugal lining sieve having about 625 round holes per square inch. The sieve must be supported by braces. The funnel is used in connection with a vacuum-filtering flask. Connection is made with the vacuum system of the factory.

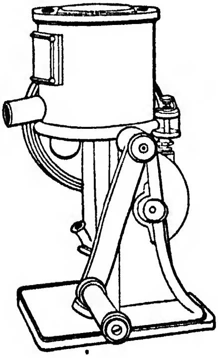

Fig. 199. Laboratory Centrifugal.

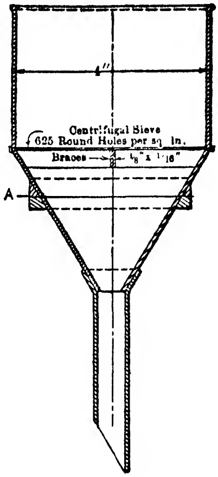

Fig. 200. Massecuite Funnel.

Very often this method of control will indicate whether the pan boiling is good or poor. When a low-purity massecuite yields a high-purity molasses, on immediate purging, it indicates poor boiling. A few days' experience with the pans, following the work with these control tests, will usually indicate whether the sugar boilers are obtaining the best results the pans and material are capable of yielding.

DIVISION OF THE SEASON INTO PERIODS

801. Division of the Manufacturing Season into Runs or Periods. To present the laboratory and manufacturing data properly to the management of the sugar house, the season should be divided into a series of short periods, or runs. In a factory that is not operated on Sundays, the periods may conveniently end with that day, making a run of two weeks. In many factories it is customary to close a period's work when the machinery is necessarily stopped on account of bad weather or a mishap.

Spencer preferred to close a period arbitrarily, after a certain number of days' work, in order that the periods of one crop might be readily compared with those of previous years. A period of two weeks or fifteen days is a convenient one.

If the machinery is idle, it is necessary only to measure the quantity of material in process at the various stations and calculate their commercial sugar value.

If the factory is in operation the following is the mode of procedure. The chemist should prepare slips of paper with the numbers or other designations of the tanks and various pieces of apparatus, conveniently arranged for noting the measurements at the stations.

If the factory day ends at midnight, for example, the chemist, an assistant, and the laboratory helper, provided with sample cups, a measuring rule and a thermometer should at that time go systematically through the factory drawing samples of the juice, etc., and measuring the quantity of material at the various stages of manufacture, at the same time noting the temperature. This stocktaking should begin with the juice and end with the sugar and must include all material in process, even the sugar in the centrifugals. It is usually more convenient to measure the depth of the empty space in the tanks, rather than that of the liquor, i.e., the "inches out." By using a very small measuring cup for sampling, one cupful, for example, may be drawn from a quarter of a tank of sirup, two cupfuls from half of a tank, and so on, thus forming a composite sample that will represent the average composition of the sirup with a fair degree of accuracy. If the tanks are of different sizes, the quantity drawn from each must be varied accordingly.

The volume of juice in the multiple effects should be estimated and may be considered a constant quantity from run to run, when the apparatus is in use. The material in the multiple effect may be measured when liquidated into tanks, or the apparatus may be calibrated with water at the beginning of the season. The density of the sirup in the vessels of the evaporator should be ascertained from time to time as a guide in calculating the sugar value.

By prearrangement with the sugar boiler, certain tanks of sirup (meladura) and molasses may be omitted as such from the stock and may be used to complete strikes of massecuite then in the pans. Thus these massecuites need not be measured until they reach the crystallizers, or if immediately purged, the sugar and molasses may be separately considered. If this arrangement is not feasible, the sugar boilers should, at the whistle signal, note the depth of sirup and molasses in the tanks and indicate the approximate depth of massecuite in the pan by chalk marks. The condition of the massecuite should be noted or, preferably, proof stick samples should be drawn for analysis. The quantity of sugars in the centrifugals, hoppers, and bins should be noted, also the last serial package number.

Stocktaking in a large factory requires but a few minutes, if the above scheme is followed. The results are practically as accurate as if the stock taken were during a shutdown.

When the run report is called for to include a certain date, the work of stocktaking may be facilitated, should it be known that the factory will be shut down a day in advance or a day later for cleaning or other reason. In this event the cane ground before or after the date is either carried as stock and figured to sugar, or its product is deducted as the case may require.

802. Estimation of the Yield of Sugar for Stock in Process. When the samples have been taken as described, they are carried to the laboratory and analyzed for Brix, polarization, and apparent purity. From suitable tables prepared for the various-sized tanks in the factory, the volume of each of the different grades of material is calculated to convenient units (gallons, cubic feet, or metric measurements as preferred). The method for calculating the stock is similar to that employed in refineries, namely, to obtain the total weight of the solids, weight of pol, and the purity of the whole stock, and then calculate the available sugar and molasses from these data.

On a suitable stock sheet form the data as obtained should be entered as follows:

1. Grade of Material.
2. Temperature, as observed in factory (T).
3. Volume of Material.
4. Brix, at Temperature T, obtained as described below.
5. Weight per Unit Volume at Temperature T.
6. Weight of the Material [(3) × (5) = (6)].
7. Corrected Brix at 20° C.
8. Weight of Solids [(6) × (7) = (8)].
9. Pol of Material.
10. Weight of Pol [(8) × (9) = (10)].

The totals for the weight of material (6), solids (8), and pol (10), having been obtained, the average purity of the stock in process is obtained thus:

Then

$$\frac{\text{Weight of pol (10)}}{\text{Weight of solids (8)}} = \text{Average purity of stock in process } (C)$$

Let x = yield of anhydrous commercial sugar in terms of the solids in the stock;

C' = purity of sugar produced in that period;

M = purity of molasses produced in that period.

Then,

$$x = \frac{100(C - M)}{(C' - M)} \quad \text{(See Sec. 836 (}c\text{))}$$

Weight of solids in stock (8) multiplied by x equals weight of dry commercial sugar (11) which, divided by the per cent solids in the sugar for that period equals the weight of commercial sugar in stock (12).

Solids in stock (8) minus solids in sugar (12) equals weight of solids in molasses (13) which, divided by Brix of molasses equals weight of molasses in stock (14) multiplied by pol in molasses equals weight of pol in molasses in stock. The weight of molasses (14) divided by the weight per gallon gives the gallons of molasses in the stock.

Having thus determined the weight of sugar in stock (12) and the weight of molasses in stock (14), these may be added to the sugar and molasses actually made, thus giving sugar made and estimated and molasses made and estimated for the calculations of the various data, retention, yield, losses, etc., for the technical report. The usual practice is to calculate the figures to date and, by subtraction of previous to-date figures, to obtain the figures for the run.

Brix of Materials. The Brix at the factory temperature T is not generally obtained by direct observation but by recorrecting the corrected Brix (7) back to the factory temperature T. For example: Suppose the clarified juice in the factory to have a temperature of 80° C. and a corrected Brix (20° C.) of 15.20. By the table on p. 729, 80° C. has a correction of 6.9. Then $15.20 - 6.9 = 8.3$, is the Brix at 80° C., and the corresponding weight per unit volume may be found on p. 731.

If time permits (as when the factory is not operating) a much simpler procedure is that used in refineries, where stocktaking is invariably done during a shutdown. The Brix of the materials in the tanks is taken at the factory temperature, care being exercised to avoid cooling while the determination is being made. By this means all temperature corrections are dispensed with, and the weight per unit volume may be obtained directly from the table.

AVAILABLE SUGAR

803. Purpose of Formulas. Many formulas are in use for the calculation of available sugar, or the sugar it is assumed that a factory should be able to obtain from juices of a given analysis. Manifestly there are several conditions that control the proportion of sugar that may be considered available in the mill juice, viz., the efficiency of the machinery, the quality of the juice, and the efficiency of operation. The quality of the juice, so far as the recovery of sugar is concerned is dependent not only on the amount of sucrose present but also on the amount and composition of the reducing sugars, ash, and organic non-sugars. Climatic and soil conditions, variety and maturity of canes, and methods of cultivation may all affect the make-up of non-sucrose in the juice. No formulas in general use take these factors into consideration.

In addition to the factors mentioned above the recovery of sugar varies with the purity of the juice and of the final molasses, the purity of the sugar produced, and the losses in manufacture. Most formulas do not include losses, and many of them assume a fixed purity for the final molasses.

The practical applications of available sugar calculations are in the purchase of cane on analysis (Chapter 37); also in estimating return from new investment, in comparing the present work of a factory with that of some previous period, or with that of a factory similarly located as regards soil and climate. Such calculations are of value in the control of several establishments operated under a central organization. Frequently it is desirable to know (1) without stocktaking how nearly a factory is approaching its previously demonstrated efficiency, (2) how much sugar is in process for commercial, operating, or accounting reasons, and (3) whether it is more profitable to reduce the rate of grinding or sacrifice somewhat in juice extraction. For these purposes the available sugar formulas are of value.

804. *SJM* Formula. The basis of practically all available sugar calculations is the widely known *SJM* formula of Noël Deerr.[15] Expressed in words, this formula says that given a juice (or initial material) of J purity and producing a sugar of S purity with a molasses of M purity, the percentage of the total sucrose (or pol) in the original material which will go into the sugar will be

$$\frac{100S(J - M)}{J(S - M)}$$

The formula, from the mathematical development, separates the sucrose (pol) in the original material into two portions, one of which goes to the sugar produced and the remainder to the molasses. No losses of any sort are taken into account in this formula. The figure which is found is the maximum sucrose (pol) in sugar per cent sucrose (pol) in juice under the conditions, i.e., the maximum attainable retention or boiling house recovery with the given juice, sugar, and molasses purities.

In the original exposition of this formula[16] Deerr said that it should be used with true sucrose and true solids, i.e., with true purities. He suggested that the sirup purity should be used for J instead of the juice purity so that the removal of impurities in the clarification and by scale formation in the evaporators might be allowed for. In the later edition of his book (London, 1921) he suggested gravity purities with the Brix of sirup and molasses determined at the approximate Brix of the raw juices. This is the practice in Hawaii.[17]

[15] *Cane Sugar,* London, 1911, p. 509.

[16] *Loc. cit.*

[17] *Methods of Chemical Control,* 1931, p. 6.

The formula has found wide application with apparent purities, and its value is by no means limited to gravity purities, although they may be used where sucrose figures are determined or, if true solids are also determined, true purities may be used. As will be shown in the following sections many modifications have been advocated, usually with a fixed purity for molasses.

In the writer's experience, the *SJM* formula with the apparent purities of the actual juice, sugar, and molasses is the simplest and in many ways the most valuable of the available sugar formulas. The calculated retention, with allowance for factory losses, should check fairly closely the retention found in the factory, if the weights, sampling, and analytical work are correct. This is particularly true for the to-date figures. An example taken from the crop figures of a large Cuban factory follows.

Juice purity		83.81
Sugar pol	97.36	
Sugar moisture	0.54	
Sugar purity		97.89
Molasses purity		32.51

Then,

$$\text{Maximum retention} = \frac{97.89(83.81 - 32.51)}{83.81(97.89 - 32.51)} \times 100 = 91.65$$

The actual retention for the crop was 90.91 and the undetermined loss 1.25. It is obvious that the recorded retention figure cannot be greater than the calculated maximum unless there are errors in the control work. Nevertheless, cases have been noted where published data have shown retentions 1 or 2 per cent higher than the maximum possible attainable by calculation under the given conditions.

The *SJM* formula with actual apparent purities for juice, sugar, and molasses may also be used for calculating yields for cane purchase payments. (Average molasses purity, or purity attained in a previous run may be used.) An instance was reported some years ago where equitable payments for frozen cane were calculated by this means.[18]

805. Calculated Boiling House Recoveries (Retentions) by *SJM* Formula. The calculation of the *SJM* formula for all practicable juice purities and all possible molasses purities to tenths of a degree would make a very extensive table for which space is not available. On p. 763, Table 38, are given the boiling house recoveries (retentions) for juices of purities from 72° to 92° in whole degrees and for molasses purities from 25° to 40°, also in whole degrees. It will be seen that the differences per degree both of juice purities and molasses purities are rather large and interpolation would not be simple. However, from this table each laboratory may construct a graph on large scale coordinate paper with recoveries (retentions) on the horizontal scale and juice purities on the vertical scale. This will give a series of curves corresponding to each molasses purity. Such a graph may be limited to the

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[18] Meade, *Louisiana Planter,* April 27, 1912.

juice and molasses purities for the particular cane-growing section, and if plotted on a large scale as suggested the curves will give as accurate results as an expanded table.

806. Formula of Winter and Carp. This formula, published by Prinsen-Geerlings[19] is based upon experience in Java but it has found wide use throughout the sugar world. Many modifications have been suggested, but the original formula is still general:

$$x = S\left(1.4 - \frac{40}{P}\right)$$

where x = available sucrose (pol) per cent cane;
S = per cent sucrose (pol) in the juice in terms of the weight of the cane;
P = purity of the juice.

The derivation of the formula is based on Winter's observations of actual results in Java which showed that 1 part of non-sucrose (or non-pol) held 0.4 part of sucrose (pol) in the final molasses.

It will be seen from this relationship of sucrose (pol) in molasses to non-sugars that the formula is based on a molasses purity of 28.57, i.e., $(0.4 \div 1.4) \times 100 = 28.57$. This formula is the equivalent of the *SJM* formula with a molasses purity of 28.57 and sugar of 100 purity, but it is not a modification of Deerr's formula, as is so frequently stated, since it was published prior to Deerr's work.

Whether the molasses purity is apparent, true, or gravity depends on whether it is based on true sucrose or polarization and whether the Brix or dry substance is used with true sucrose figures. In the United States and Cuba the use of apparent purities in this formula is almost universal, i.e., it represents pol in sugar of 100° purity per cent pol in juice.

The theoretical retention by this formula for a juice of P purity becomes

$$100\left(1.4 - \frac{40}{P}\right)$$

and is the basis of the calculation of the boiling house efficiency number defined below.

807. Boiling House Efficiency Number (BHE). Many factories use efficiency numbers to show how nearly they approach a theoretical yield (or retention) of sugar. This number is usually the percentage relation between the actual retention (or boiling house recovery) and the theoretical retention number based upon an available sugar formula. A part of the Winter-Carp-Geerligs formula, $100[1.4 - (40/P)]$, was used by Spencer for the theoretical retention in calculating the efficiency number of the Cuban-American Sugar Company's factories, and the practice is now general in Louisiana and the West Indies. As will be shown later, an error exists in this method as

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[19] *Intern. Sugar J.*, **6**, 439 (1904).

generally used but the error may be easily corrected. The usual method of calculation is best illustrated by this first example.

Example. Let the purity of the raw juice be 86.0 and actual retention (or boiling house recovery) number be 92.32, then $100[1.4 - (40/86)] = 93.49$; $92.32 \div 93.49 \times 100 = 98.7$, the efficiency number. A table is given on p. 761 from which the value of $100[1.4 - (40/\text{Purity})]$ may be ascertained by inspection.

An error occurs in this method because the actual retention is dependent on the purity of the sugar produced, since with the molasses purity remaining fixed the production of a lower-purity sugar will raise the retention automatically. The reason for this is evident: with more impurities going into the sugar there will be less impurities going to form less molasses.

The effect of the sugar purity on the retention may be seen from the following figures:

Example. With a juice of 85° purity, molasses purity of 28.57° and no losses, the retention when producing 96° purity (not polarization) sugar would be 94.52; producing 97° purity sugar under the same conditions would give 94.10 retention; 98° purity sugar, 93.71 retention; 99° purity sugar, 93.32 retention; and 100° purity sugar, 92.94 retention—this last figure being the "theoretical retention" of Winter and Carp. Thus, without having changed the quality of the boiling-house work, and the molasses purity having remained at 28.57, the retention has been increased from 92.94 with 100° purity sugar to 94.52 with raw sugar of 96° purity, merely by leaving more of the molasses on the crystals.

Since the efficiency number by the older method of calculation is the actual retention divided by the theoretical retention, it follows that the efficiency numbers under the above conditions (the factory work being the same) will be:

$$\text{For } 96^\circ \text{ purity sugar } \frac{94.52 \times 100}{92.94} = 101.70\%$$

$$\text{For } 97^\circ \text{ purity sugar } \frac{94.10 \times 100}{92.94} = 101.14\%$$

$$\text{For } 98^\circ \text{ purity sugar } \frac{93.71 \times 100}{92.94} = 100.83\%$$

$$\text{For } 100^\circ \text{ purity sugar } \frac{92.94 \times 100}{92.94} = 100.00\%$$

That is, the BHE (101.70 per cent) when making 96° purity sugar is greater than (100 per cent) when making refined sugar with the same purity of final molasses. Obviously the efficiency should be the same (as far as the pan work is concerned) with the constant purities of the juice and molasses.

In factories producing sugars of uniform polarization, this consideration is of less practical importance, and the actual efficiencies are relatively comparable. But for accuracy in extended comparisons where the sugar purities fluctuate, the retention should be recalculated to a sugar of 100° purity before dividing it by the theoretical retention of Winter and Carp to obtain an equitable efficiency number.

This may be done by adopting the standard sugar of the International Society of Sugar Cane Technologists, termed equivalent standard granulated (ESG), see Sec. 812. The calculation to retention ESG is readily carried out by multiplying the actual retention by the ESG corresponding to the purity of the sugar produced as given in the table on p. 619. If this is done, it will be seen in the examples given above that the retention ESG (equals 92.94) and the BHE (100 per cent) have the same value for all purities of the sugar as they should have with the same molasses purity (at 28.57).

Revising the first example (q.v.) to meet the above requirements in accuracy, one finds:

Purity of raw juice	86.0
Purity of sugar	97.8
Actual retention	92.32
ESG from table p. 619	99.10
Retention ESG = 92.32 × 0.9910	91.49

From Table 37 the theoretical retention = $100(1.4 - 40/86) = 93.49$. Then $91.49 \div 0.9349 = 97.86$, the corrected BHE number.

It will be noted that retention ESG is lower than actual retention and therefore the efficiency number calculated from retention ESG will also be lower for the same class of factory work. Factory operators accustomed to the higher efficiency numbers obtained by the old method will recognize the reason for this reduction when the new method of calculation is employed. The efficiency number is of value in estimating available sugar, as it takes into account the losses in manufacture in the particular factory as compared with a factory operating under the assumed conditions of the Winter-Carp-Geerligs formula.

808. Estimation of Yield. In connection with the efficiency number described above it is possible to estimate the yield of sugar with considerable accuracy from the weight of the cane, the extraction, and the purity of the juice.

The value for $100[1.4 - (40/P)]$ for juices of various purity are given in Table 37 on p. 762. Formerly it was customary to divide the value found in Table 37 by 96 in order to reduce this theoretical retention to sugar of 96° but this practice is incorrect as it does not take into account the impurities in the sugar.

To overcome this objection E. M. Copp has calculated the available 96° sugar corresponding to this theoretical retention (Table 37A). This is the retention for 97° purity sugar (96° pol, 1.03 moisture) by the *SJM* formula (molasses purity 28.57) divided by 0.96.

The following example illustrates the use of this formula and the efficiency number in calculating the available 96° sugar:

Example.

Pol in the cane, per cent	14.1
Extraction, pol per cent pol in the cane	93.49
Purity of the juice	84.1
Corrected boiling house efficiency number	99

Pol extracted per cent cane = 14.1 × 0.9349 = 13.18

Referring to Copp's Table 37A, p. 762, the number corresponding to 84.1° purity is 97.50 and 13.18 × 0.9750 = 12.85, the available 96° sugar per cent cane if the efficiency number were 100. Applying the actual efficiency number, 99, we have 12.85 × 0.99 = 12.72, the estimated available 96° sugar per cent of cane. In practice the method has been found to give good check results with actual yields.

809. ISSCT Definitions. The International Society of Sugar Cane Technologists in their *System of Factory Control* (pp. 23–26) gives several formulas for measuring performance in the boiling house which will be new to many western hemisphere technologists. Unfortunately these definitions specify a sucrose basis and gravity purities and appear to offer no option in the matter.

This point may seem to be covered by the statement regarding purities (p. 22).[20]

> Since the Committee has decided that the control be based on sucrose rather than pol, it logically follows that gravity purities are to be reported wherever sucrose values are required. However, as it may take some time, for practical reasons, until sucrose figures are determined universally, it will be necessary for all factories which use the sucrose basis to make reports also on a pol basis, giving both gravity and apparent purities, sucrose and pol balances, etc.

In the definitions for mill control the Society's Committee gives the option between sucrose and pol (as, for instance, with extraction, Sec. 790), but as will be seen in the definitions given below, the option (sucrose or pol, apparent or gravity purity) is not given, and these criterion figures would seem to be closed by definition to those factories which do not have a sucrose control.

Not only is the option not given but also in several instances the Society's definitions are specifically cited as differing from previous definitions by the use of gravity purities as opposed to apparent purities in the older definitions. This point will be further discussed in the paragraphs describing the ISSCT criterion figures.

Also, it is evident that since gravity purities of molasses are considerably higher than apparent purities a predetermined gravity purity of 28.57 is very much harder to attain than an apparent purity of 28.57. The formulas, if apparent purities are employed (as is general practice in this hemisphere), will show a different range of results than if gravity purities are used. Such

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[20] Page references and quotations in this section are from the *System of Factory Control for Cane Sugar Factories,* New York, 1942, published by the International Society of Sugar Cane Technologists.

ratios as extraction will not vary greatly whether on a sucrose or pol basis but there is no such close relationship between the apparent purity and the gravity purity of a molasses.

810. Basic Boiling House Recovery (ISSCT). The International Society of Sugar Cane Technologists finds that there is confusion in the use of the term available sugar, and (p. 23) "to avoid further confusion," the Committee recommends the term basic boiling house recovery. Quoting (p. 24), "The Committee defines the Basic Boiling House Recovery as the percentage of sucrose in the mixed juice which would be obtained according to the SJM formula, if the molasses had a gravity purity of 28.57." This is the equivalent of the theoretical retention used in Cuba (Sec. 806) but on a sucrose basis with gravity purities instead of on a polarization basis.

The question previously raised presents itself here. What is basic boiling house recovery in a factory working on a polarization basis only? The qualifying "pol" may be added to the term (as is done, for example, with extraction, Sec. 790), but the definition distinctly states that sucrose and gravity purities must be used in contradistinction to Cuban practice where the same figure is calculated on a pol basis. The matter may be clarified by continuing to use the term theoretical retention for this figure where apparent purities and pol are the basis.

811. Standard Sugar. If factories produce sugars of different types and different tests, it is evident that the recoveries (retentions) or yields are not comparable. It is necessary to convert the quantity of sugar into a comparable equivalent. In Cuba and Louisiana the general plan has been to convert to a 96° test basis by the incorrect method of dividing the recovery (retention) by 0.96, but this disregards the purity of the sugar. The conversion to 96° may be made by first obtaining the ESG corresponding to the actual purity of the sugar from the table given on p. 619, then multiplying this by the actual retention to obtain retention ESG, and finally dividing this by 0.9481 (which is ESG for 97° purity sugar multiplied by 0.96). A simpler method for the same operation is to multiply the sucrose (pol) in the sugar of given purity by the factor in Copp's Table 39 (p. 764) for that purity and 28.57 molasses, which gives the equivalent 96° test sugar (97° purity, 96 pol, 1.03 H_2O) for the retention in question.

In Java the standard of comparison is "Crystal," which is calculated by multiplying the weight of the non-sugar in the sugar produced by the factor $M/(100 - M)$, and subtracting the product from the weight of pol in the sugar; M is the apparent purity of the molasses being turned out by the factory. As another example of a standard raw sugar that proposed by Peck may be cited; this contains 96 per cent sucrose and 1 per cent moisture (96.97 purity), and the conversion is to be made by the *SJM* formula, with an assumed gravity purity of 33⅓ for molasses. [Page 24.]

812. Equivalent Standard Granulated (ESG). The International Society of Sugar Cane Technologists has adopted this type of standard sugar which was introduced by Deerr:

> . . . and is defined as the quantity of dry, pure sucrose (100 per cent sucrose, 100 purity), which could theoretically be obtained from a sugar or other raw material, using the *SJM* formula and assuming a gravity purity of 28.57 for final molasses. This molasses purity is identical with the apparent purity assumed in the Winter formula. The ESG differs from the Java "Crystal" in that gravity purities are used in the calculation, and that the purity of the molasses is fixed. [Page 24.]

Again the question might arise as to whether or not ESG can be used with a polarization basis, since by definition it is based on gravity purities, but it would appear that ESG (pol) may be used by factories working on a pol basis, if all purities are understood to be apparent. The only variable in the calculation of ESG is the purity of the sugar. In the table given below the figure is calculated for sugars of purities from 95 to 100. For all practical purposes the purity of the sugar may be taken as sucrose (or pol) plus moisture rather than sucrose (or pol) divided by 100 minus moisture. This type of standard sugar has advantages but the long-continued use of "96 test" as a standard in the western hemisphere will probably operate against its rapid adoption in this section.

EQUIVALENT STANDARD GRANULATED (ESG)

(For converting retention of sucrose (pol) in sugar of a given purity into retention of 100% dry sucrose (ESG). Calculated 1943 by *SJM* formula with 28.57 molasses.)

	0.0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
95	97.89	97.93	97.98	98.02	98.07	98.11	98.16	98.20	98.25	98.29
96	98.33	98.37	98.42	98.46	98.51	98.55	98.59	98.63	98.67	98.70
97	98.76	98.80	98.85	98.89	98.94	98.99	99.02	99.06	99.10	99.14
98	99.18	99.22	99.26	99.30	99.35	99.39	99.44	99.48	99.52	99.56
99	99.60	99.64	99.68	99.72	99.76	99.80	99.84	99.88	99.92	99.96

Copp's Modified ESG. Copp[21] has proposed that ESG be defined as "the available 100 percent sucrose sugar to be obtained from the raw sugar if the molasses were exhausted to the purity of the virtual molasses" instead of to the fixed purity of 28.57. The formula then reads:

$$ESG = \frac{10{,}000(S - M_v)}{S(100 - M_v)}$$

where S = purity of the sugar;
M_v = purity of the virtual molasses (see Sec. 816).

Copp argues that his ESG is more accurate because a factory turning out molasses of higher test than the standard will be credited with doing better

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[21] *Intern. Sugar J.*, July, 1943, p. 183.

work than it actually is doing if ESG is calculated on a molasses purity of 28.57. Deerr, in private correspondence with Copp, recognizes the value of this proposal not only because it is more logical but also because succeeding formulas that depend on ESG, especially boiling house performance, are not mathematically correct otherwise. Under Copp's conception of ESG, the formula for virtual purity (Sec. 816) becomes, with R = actual retention:

$$M_v = \frac{S(100J - JR)}{100S - JR}$$

Copp has calculated a table for his modified ESG with a range of purities of virtual molasses from 20° to 40° including the standard purity of 28.57, but for reasons of space this is not included here. (See Copp's articles and tables *International Sugar Journal,* April and May, 1944.)

813. Boiling House Recovery, ESG. "This is calculated in the same way as the Boiling House Recovery, except that the quantity of Equivalent Standard Granulated corresponding to the sugar produced is substituted for the sucrose in sugar produced. The Committee has decided that this figure be reported." (Page 25.) In a polarization control this is the retention with the pol in sugar produced refigured to ESG (pol), i.e., it is the retention multiplied by the ESG figure in the table on p. 619 corresponding to the purity (apparent) of the sugar.

814. Basic Boiling House Recovery, ESG. "This is the same as the Basic Boiling House Recovery, as defined (Sec. 810) since it is expressed as 100 per cent sucrose." As has been shown previously this corresponds to theoretical retention (Sec. 806) on a polarization basis.

815. Boiling House Performance. "This figure, also introduced by Deerr, is calculated by dividing the Boiling House Recovery, ESG, by the Basic Boiling House Recovery (Sucrose), and multiplying by 100." This figure is similar to the boiling house efficiency number on a pol basis. BHE is equal to it if the retention is figured to retention ESG (pol) before dividing by the theoretical retention. (See p. 616.)

816. Reduced Boiling House Recovery, ESG. The Committee of the International Society of Sugar Cane Technologists does not recommend reporting boiling house performance since they consider reduced boiling house recovery ESG a better comparison figure (p. 25).

This figure is analogous to the Reduced Extraction and defined as the Boiling House Recovery, ESG, that would have been obtained under existing conditions if the factory had worked a mixed juice of a standard gravity purity of 85.

A new concept is introduced here, the "virtual molasses purity" (M_v) which is the gravity purity of the molasses that would have been produced by using the *SJM* formula with the actual boiling house recovery ESG (R') and the gravity purity of the mixed juice (J).

$$M_v = \frac{100J(100 - R')}{J(100 - R') + 100(100 - J)}$$

or according to McAllep more simply,

$$M_v = \frac{100J - JR'}{10{,}000 - JR'} \times 100$$

To obtain the reduced boiling house recovery ESG the value of M_v is then substituted in the formula:

$$R_{85} = 100 \times \frac{100(85 - M_v)}{85(100 - M_v)}$$

> It is a measure of the performance, and may be used directly for international comparisons. It should be emphasized that all purities are gravity purities, and that the recovery is expressed as Equivalent Standard Granulated, as previously defined. The Committee has decided that this figure be reported. [Page 25.]

The wording is such that the use of this figure appears to be precluded except on a sucrose basis. However, as in the case of other definitions of the Society's Committee there would seem to be nothing to prevent a factory having only a polarization control from using the figure with apparent purities, but whether such a figure will serve for other than local comparative purposes is doubtful.

The term reduced boiling house recovery, ESG, might well be called simply reduced recovery, and if used on a pol basis reduced retention.

The figure may be readily obtained without calculation if the graph for the *SJM* formula is made as described in Sec. 805 from Table 38. All that is necessary to obtain the virtual molasses in that case is to select the retention ESG of the factory on the horizontal scale and find the intersection with the actual juice purity on the vertical scale. The virtual molasses purity can then be readily found by interpolation on the graph.

$$\text{Reduced recovery} = 100 \times \frac{100(85 - \text{Virtual molasses purity})}{85(100 - \text{Virtual molasses purity})}$$

This formula has only one variable and the Reduced Recovery (or Reduced Retention) may be found from the table of virtual molasses purities, Table 40, p. 765.

The logic involved in the calculation of virtual molasses may be considered as the reverse of that for maximum retention (Sec. 804). The maximum retention calculates the amount of sucrose (pol) which will go to the sugar, using the *SJM* formula with actual purities of juice, sugar, and molasses; it is, therefore, the calculated retention without losses. The virtual molasses purity as figured by the same formula considers all the sucrose (pol) which did not actually go to sugar as going to molasses, and therefore all losses are included in the sucrose (pol) in the virtual molasses. Just as the maximum retention must of necessity be higher than the actual retention (if the control figures are correct) so the virtual molasses must be of higher purity than the actual, if sirup purity is used as that of the primary material.

When the virtual molasses has been found for the conditions under which the factory is working, this purity is then substituted in the *SJM* formula with 85° purity juice, and the result is considered as that which the factory would have done if working with juice of standard (85°) purity.

817. Overall (Total) Recovery (Sucrose or Pol).

This expression is analogous to the Boiling House Recovery, but referred to sucrose (pol) in cane instead of sucrose (pol) in mixed juice. It appears in the sucrose balance per cent sucrose (pol) in cane, and is therefore to be reported.

This figure, frequently called simply the recovery in Cuba and Louisiana, may be (by definition) on a sucrose or pol basis. It is generally reported in the western hemisphere as sucrose (meaning pol) in sugar per cent sucrose (meaning pol) in cane.

818. Reduced Overall Recovery, ESG.

This figure is simply the Reduced Extraction, $E_{12.5}$, multiplied by the Reduced Boiling House Recovery, ESG, R_{85}, and the product divided by 100. This is easy to calculate, and has been recommended by the Committee for adoption.

This figure may be on a polarization basis if the reduced recovery (Sec. 816) is defensible on that basis.

819. Yield.

This expression is generally taken to mean commercial sugar produced per cent cane, without regard to the composition of the sugar. It is the reciprocal of tons of cane per ton commercial sugar. . . .

Since the overall recovery is referred to sucrose in cane, and the yield to cane, the first can readily be converted into the second by multiplying by per cent sucrose in cane and dividing by 100. The Committee has recommended that the Overall Recovery, ESG, and the Reduced Overall Recovery, ESG, be thus converted into the corresponding Yield of ESG, and Reduced Yield of ESG respectively. [Page 26.]

In the western hemisphere it is customary to calculate the yield to 96° sugar by dividing the pounds of pol in the sugar actually produced by 0.96. This is admittedly incorrect, since it does not take into account the purities of the actual sugar and of the standard 96° test. A table by E. M. Copp by which this may be correctly calculated is given on p. 764 (Table 39). This table gives factors for converting sucrose (pol) in sugar per cent cane to 96° test, the standard sugar (97° purity, 1.03 per cent moisture).

Example.

$$\begin{array}{lll} \text{Sucrose (pol) in sugar \% cane} & = 12.74 & \\ \text{Purity of sugar} & = 97.8 & \left\{\begin{array}{l}\text{Sucrose (pol) } 97.2 \\ H_2O\ 0.62\end{array}\right. \\ \text{Purity of molasses} & = 35.10 & \end{array}$$

From Table 39 the conversion factor for 97.80° purity sugar and 35.1 molasses is 1.0465. Then 12.74 × 1.0465 = 13.33 per cent equivalent yield of 96° sugar. By the incorrect method in common use (dividing 12.74 by 0.96) the yield of 96° test for the above example would have shown 13.27 per cent, a difference of about 0.5 per cent on the sugar in question.

Copp's table is calculated as follows. The *SJM* formula is used to find the factor for sucrose (pol) from the given purity to an equivalent 97° purity sugar, and this is then divided by 0.96 to convert to 96° sugar. In the example given above the calculation is

$$\frac{100 \times 97(97.8 - 35.1)}{97.8(97 - 35.1)} \div 0.96 = 1.0465$$

In using the table the molasses purity may be taken to the nearest whole number and the sugar purity to the nearest tenth.

820. Summary on Available Sugar Formulas. Study of the foregoing paragraphs will convince anyone that the subject of available sugar formulas is far from standardized. The first question at issue is whether the International Society of Sugar Cane Technologists formulas, which invariably specify gravity purities, permit the substitution of apparent purities, but even if sucrose figures are available, it is doubtful whether any formula can have more than local value if the exhaustibility of the molasses is not taken into account.

To compare the work of eastern Cuba, for example, with that of Louisiana by means of any of the formulas given in this section would seem to be of questionable value. Cuba grinds mature cane and the eastern area has a glucose-ash ratio of about 1, whereas Louisiana works with juices of relatively green canes and glucose-ash ratios of 2, 2.5, or 3.

The final molasses from eastern Cuba is rarely lower than 40 true purity, whereas in Louisiana true purities of 30 or under are not unusual, particularly early in the season. McCleery[22] studied the effect of glucose-ash ratios in Hawaiian molasses (see Sec. 822) and found about 10° difference in the expected purity of a molasses having a glucose-ash ratio less than 1 and another molasses with a glucose-ash ratio of 2.5 to 3, so the practical results in Cuba and Louisiana bear out these results. Since a difference of 10° in molasses purity when working a juice of 85° purity will make a difference of over 4 per cent in the retention, it seems hardly worth while to add refinements to formulas for international comparison without giving any consideration whatever to the character or expected minimum purity of the molasses.

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[22] *Proc. Hawaiian Sugar Planters' Assoc.*, October, 1935.

Some question also arises as to whether juice figures can be profitably reduced to a standard purity (as in reduced boiling house recovery, ESG, Sec. 816). Juices below 75° purity such as frequently occur in Louisiana and other subtropical sections require much less reboiling to obtain a low molasses than do high-test tropical juices of 90° purity or above. The results of factories working under such divergent conditions can hardly be calculated to standard comparable results by any such simple formula as the *SJM* relationship.

These considerations would seem to indicate that any simple and universal criterion for boiling house work is open to question because of the number of variables which are involved. It therefore seems likely that the available sugar formulas which have been in use in various localities and to which the local technologists are accustomed will continue to be used even though many of them are largely empirical.

EXHAUSTIBILITY OF MOLASSES

821. Sucrose to Molasses. The greatest loss of sugar in the factory or refinery is that which goes to the residual molasses, and the extent to which this sugar may be recovered economically has been the subject of many studies. Many operating factors, such as the character of the crystals and the density of the final massecuites, the extent of the cooling in the crystallizers and the after-treatment of massecuites, affect the purity of the final molasses, as has been shown in the section by Davies on "Crystallization in Motion" (p. 223). The viscosity is also of great importance in the exhaustion of the molasses (see also p. 218).

822. Effect of Non-Sucrose. It has long been recognized that the composition of the non-sucrose of the molasses has great influence on the degree to which the molasses can be exhausted. A review of the literature on this subject is given by Davies on pp. 223–224, and although there is still no general agreement as to the best criterion of exhaustibility the glucose-ash ratio, i.e., per cent reducing sugars divided by per cent ash, is one which was first suggested by Geerligs and which is still considered of great significance by many technologists. McCleery[28] calculated a table of indicated gravity purities for Hawaiian molasses of different viscosities and different glucose-ash ratios. A graph of part of the results is given in Fig. 201. As will be seen, the difference of 0.55 purity for each 0.10 change in glucose-ash ratio from 0.60 to 1.75 is practically a straight line, but at higher ratios the purity differences decrease rapidly. According to McCleery practical results in factories in Hawaii bear out these expected results fairly closely.

McCleery reports that "refractory" molasses have been found which will not crystallize to the indicated purities and that this may amount sometimes to 3° purity, but it is definitely proved that the factory having a low ratio

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[28] *Proc. Hawaiian Sugar Planters' Assoc.*, October, 1935.

cannot obtain as low molasses purity as one with a high ratio. This relationship between glucose-ash ratio and exhaustibility has been noticed in practical results in other countries.

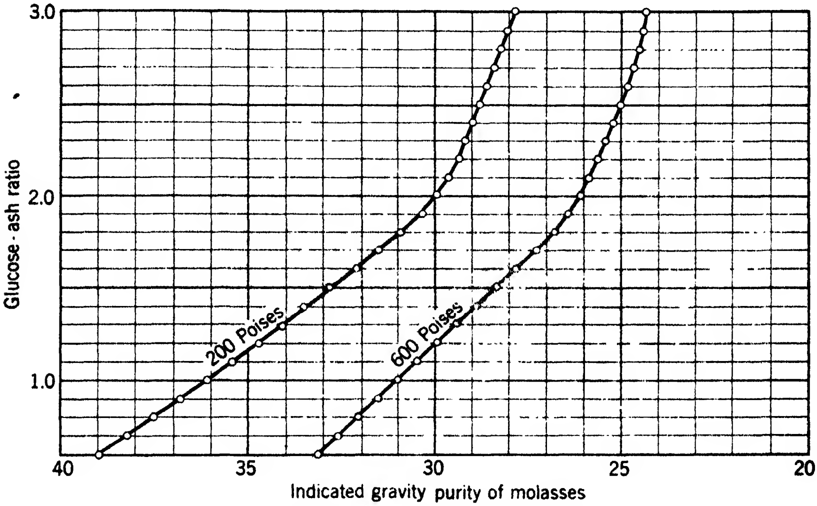

FIG. 201. Molasses Purities and Glucose-Ash Ratios.

LOSSES OF SUGAR IN MANUFACTURE

823. Classification of Losses. The various losses may be classified into two general kinds, determined and undetermined. The only losses which are determined and recorded in regular sugar house control are those in the bagasse and filter press cake and these losses are of the mechanical type, i.e., the sugar is lost as such, in the form of sucrose.

824. Determined Losses. *The Bagasse.* The loss in the bagasse is calculated from the calculated weight of the material (Sec. 772) and its analysis.

If the weight of the imbibition water is known, the bagasse is the weight of the cane plus weight of saturation-water minus weight of the mixed dilute juice.

Filter Press Cake. The loss of sucrose is calculated from the analysis and weight of the press cake.

The weight of the press cake is usually estimated from the actual average weight of the contents of several filter presses or preferably by weighing the entire amount of cake produced. When this is not practicable the cake from a single chamber or frame of the press is weighed from time to time to obtain an average weight, and this number is multiplied by the number of cakes in the press, to obtain the total weight. The cake weighs approximately 60 to 62 lb. per cu. ft.

There is a loss of sucrose in the juice absorbed by the filter cloth. This is usually included with the unknown losses. This quantity varies with the filter press methods from almost nothing with double filter-pressing to 0.5 lb. or more per filter cloth.

825. Undetermined Losses. (*a*) *Apparent Losses.* These are not real losses, as the name implies, but they may play a large part in the reported losses of a factory. They are due to some error in cane, juice, or sugar weights; or to errors of analysis or to incorrect stock estimates. These last will correct themselves in to-date figures. Apparent losses are reducible to a minimum by careful supervision of weights, tests, and methods. It is equally evident that the same errors which cause apparent losses may, if operating the opposite way, cloak true losses and tend to make the factory figures appear much better than they really are. As an illustration, any error in the sampling, weighing, or analysis of the mixed juice which would tend to show less juice, or less sugar in juice, than is really present would improve the loss figures in the boiling house to the extent of such error. Practices which tend to give favorable figures should be scrupulously avoided as such practices are a form of self-deception that may cover up large monetary losses which might be prevented.

(*b*) *Mechanical Losses.* These include all waste of sugar in the form of sucrose. Besides the known losses in bagasse and press cake enumerated above, losses of sugar solutions to the sewer may result through leakage or spillage, though in a well-regulated factory these losses should be negligible. Entrainment is a mechanical loss, the sugar being carried over from the vacuum apparatus to the condenser waters in the vapor in the form of minute bubbles (vesicular transference). Modern apparatus practically eliminates this source of loss but a constant check should be kept on condenser waters (see p. 576).

The loss by entrainment may be estimated by Norris' table, p. 712, from the analysis of the water flowing from the condenser and its weight as calculated from the temperature changes and the quantity of water evaporated. Detailed instructions are printed with the table.

(*c*) *Inversion and Decomposition Losses.* The chemical nature of inversion has already been described (Sec. 433). It may be well to repeat that sucrose, when acted upon with acids or acid salts, changes into a mixture of dextrose and levulose called invert sugar (frequently called glucose together with all other reducing sugars). The extent of the inversion depends upon the nature of the acid, mineral acids having a much greater inverting power than organic acids; the quantity of the acid present in relation to the amount of sucrose: the temperature of the acid-sucrose mixture (increase of temperature speeds up the inversion greatly) and finally the time that the acid and sucrose are in contact (see Fig. 108). The hydrogen-ion concentration (pH) is a function of the first two factors (kind and amount of acid present) so the importance of pH control in the prevention of inversion losses is therefore evident.

The estimation of inversion in a sugar factory is a complicated matter because of the simultaneous decomposition of sucrose and levulose from the action of heat and alkalies. Glucose ratios (relationship between sucrose and reducing sugars, Sec. 754) on the successive products are of value in indicating excessive inversion, but the assumption in the use of such ratios is that no reducing sugars have been decomposed.

Both inversion and decomposition losses can be reduced by a strict pH control on the clarification. Walten, McCalip, and Hornberger[24] showed that the initial pH of the juice should be such that it will reach the sirup stage with a pH between 6.7 and 7.0. If the sulfitation process is employed, a lower pH may be safely carried. (See p. 109.) Under these conditions inversion during clarification and subsequent evaporation and boiling can be reduced to a minimum, and the decomposition of glucose due to excessive alkalinity will be avoided, so far as possible.

Levulose is usually destroyed to some extent in the manufacture, therefore it is advisable to figure a glucose balance as well as a sucrose balance to obtain light on the losses.

826. The Detection and Prevention of Losses. The detection and prevention of sugar losses in the factory and in the refinery present problems of great complexity. When excessive losses occur in either type of sugar plant it is natural and proper to look for stealage, drips to sewer, or other mechanical losses, but in the writer's experience this type of loss rarely amounts to an appreciable percentage in a plant with a careful control. On the other hand, unaccountable losses which appear to be of a chemical nature, i.e., either inversion or decomposition or both, may occur in factories or refineries with elaborate control methods.

Lyle[25] gives a detailed account of the sources, extent, determination, and prevention of the various sugar losses which may occur in the refinery. He reports the curious fact that, in spite of the most elaborate and conscientious control, at least two-thirds of the sugar lost cannot be accounted for by known means. This is also the experience of most refiners in the United States, and it is probably equally true for raw sugar factories. Another noteworthy fact regarding raw factory losses is that two closely related factories having the same type of management and supervision will differ quite widely and consistently in the extent of the losses year after year. It follows naturally that if losses which occur regularly and systematically cannot be traced, similar losses of greater extent may occur sporadically or for particular seasons or parts of seasons. Excessive losses which would seem to be easy to detect and control are frequently just as elusive as the regular losses.

Methods for tracing unknown losses by means of changes in the saline coefficient have been suggested from time to time. Zimmerman[26] based his calculations on the soluble sulfates in the sulfated ash. More recently Davies and Yearwood[27] made an extended investigation based on the chloride input

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[24] *J. Ind. Eng. Chem.*, **17**, 51 (1925).

[25] *Technology for Sugar Refinery Workers*, London, 1942, pp. 294–309.

[26] *Intern. Sugar J.*, **16**, 383 (1914).

[27] *Sugar*, December, 1942, p. 29.

and output of the evaporators and pans, the chloride content of the sirups and massecuites being determined potentiometrically. Their findings indicate large losses of sucrose and reducing sugars during evaporation and pan boiling which the standard control figures (purities and glucose ratios) would not show; in fact, these figures appear quite normal. If these figures of Davies and his colleagues are corroborated in further work it would appear that the principal sources of the unknown losses in a factory are the evaporator and pan stations. Also, similar investigations may indicate losses in the pans in refinery work.

LABORATORY AND FACTORY RECORDS

827. Test Books and Records. It is difficult to plan a set of books and forms without knowing something of the needs of the management, the force of chemists available for control, and whether this control is to be partial or fairly complete.

The usual reports include: (1) A slip for the manager, superintendent, and engineer should give preliminary data of the mill work and control analyses of the juices, the output of sugar, and the fuel consumption; this should be supplemented by frequent reports to the engineer on the analysis of the bagasse. (2) The preliminary report should be followed by what may be termed an operating report, which should include data covering the entire line of chemical control and the manufacture, together with mill and manufacturing statistics. (3) Run reports at stated intervals, giving a résumé of all data collected both for the run and to date, including a sucrose (or pol) balance and a statement of yield and losses. The data should be full enough to indicate the methods of manufacture and should supply the management with a permanent record of methods. Working and lost time should also be reported to indicate what portion of the factory's capacity is being utilized. (4) Laboratory records: (*a*) used in the analytical work; (*b*) extraction figures, etc.; (*c*) records of pan work; (*d*) unit book, used in recording the quantities of materials, products, and by-products and in calculating weighted averages.

Printed forms should be supplied for the entries of the routine laboratory work. A large space should be provided in these forms for the figuring with a view to tracing errors. The use of printed forms also promotes systematic work. A looseleaf binder is convenient for these forms, and a sheet should be used each day. All figuring should be on the sheets or in special books and never on scraps of paper. The use of calculating machines has greatly facilitated this type of work and has eliminated errors in calculations.

It is advisable to have printed forms for calculating and recording mill data, operating and lost time, and fuel consumption. A special blank should be posted at the mills for reporting the delays and their causes. These figures should be tabulated from time to time for the use of the general manager and the chief engineer.

The daily laboratory reports, for a fairly complete control, should include: (1) analyses of the mixed, absolute (calculated) and residual juices; (2) fiber and sucrose (pol) in the cane; (3) analysis of the sirup—the Brix for the control of the evaporation and the purity for that of the defecation; (4) analyses of the massecuites and molasses to control the pan boiling and the work of the crystallizers; (5) moisture and polarization of the sugars and occasionally ash tests. The moisture has a bearing on the storage qualities of the sugar; the polarization must meet market requirements; the ash is an additional check upon the purification of the juice. (6) Analysis of the final molasses to meet market conditions and to control the pans, crystallizers, and centrifugals; (7) Analysis of the filter press cake to control the loss of sugar; (8) analyses of the bagasse at frequent intervals, including moisture and polarization for mill control; (9) Frequent examination of the feed water for sugar, for the protection of the boilers; (10) Entrainment tests in the condenser water, to protect against carelessness in the evaporation and in the pan boiling.

The manufacturing data that should be included in this report are records of the cane ground per hour and per day; operating and lost time; saturation and dilution; mill extraction; available sugar, sugar in process, and sugar produced; fuel consumption. These figures should be for the day and to date.

A convenient arrangement of a run report is to place the manufacturing data on the left-hand side of the sheet and the analytical data and figures derived from them on the right.

CHEMICAL CONTROL WHEN MAKING HIGH-TEST MOLASSES

828. Outline of Production. The manufacture of high-test molasses (actually an inverted cane sirup, see Sec. 333) has become a very large industry in Cuba because of quota restrictions, war conditions, and other crop-limiting circumstances. The process consists of extracting, clarifying, and evaporating the juice as for sugar manufacture, except that the pH is carried at a minimum (6.0 to 6.4), since inversion at this point makes no difference. The meladura is run into the inverters (generally the crystallizers in practice) and inverted by acid or yeast invertase. The inverted sirup is then concentrated in the vacuum pans, and the result is a mixture of about one-third sucrose and two-thirds reducing sugars, totaling about 75 per cent and having a Brix of about 85.

829. Basis of Control. The control of the mill work and clarification station may be carried out on the same general lines as those already outlined, but it is evident that the factory balance must be on the basis of total sugars as invert sugar, and the technical control must be based on sucrose (not polarization), with the results calculated to an invert sugar basis. As will be shown below the routine running control is on a polarization basis. Since 95 lb. of sucrose when inverted makes 100 lb. of invert sugar the total sugars as invert sugar entering in the mixed juice will be

$$\frac{\text{Sucrose in juice}}{0.95} + \text{Reducing sugars (as invert sugar) in juice}$$

In the same way the total sugars as invert sugar in the molasses will be

$$\frac{\text{Sucrose in molasses}}{0.95} + \text{Reducing sugars (as invert sugar) in molasses}$$

These points are emphasized as some of the earlier publications on the subject have referred to total sugars in the control as sucrose plus reducing sugars. Obviously, this figure is meaningless for any purpose except as a commercial standard, and even then the total sugars on an invert basis is a more correct figure (see Sec. 307).

For practical purposes the total sugars as invert in the press cake may be taken as pol in press cake/0.95, or if desired, the reducing sugars in the press cake may be estimated from the glucose ratio of the juice (p. 585), i.e., it may be assumed that the relationship of reducing sugars to pol in press cake is the same as that in the juice. The estimated reducing sugars (as invert) may be added to pol/0.95 to obtain total sugars in press cake as invert.

The running control of the process is on a polarization basis as in the manufacture of sugars. The inversion may be so regulated as to give the desired per cent of inverted sucrose, but the more usual practice is to invert a part of the meladura completely, say 60 per cent, and then to concentrate this with the remainder of the uninverted meladura to give the desired result. Either method may be controlled by the direct polarization (or apparent purity if preferred), but it should be remembered that the algebraic sign of the polarization (or purity) will be minus in complete inversion. With this in mind the Cobenze diagram (Fig. 202) may be used to calculate the mixture or the chart (Fig. 203) on p. 640 may also be used.

Example. An inverted sirup polarizes −6, the uninverted sirup +44. What percentage of each will give a high-test molasses of +12? Then from the Cobenze diagram: $-6 - (+12) = 18$ parts of uninverted sirup to be mixed with $44 - 12 = 32$ parts of inverted sirups or $32/50 = 64$ per cent of inverted sirup must be mixed with 36 per cent of uninverted.

830. Factory Balance. With the weights of mixed juice (determined as in the ordinary control, Sec. 768) and the weights of press cake and molasses, together with the analyses referred to above, all converted to invert sugar, a factory balance may be readily found. The results from a large factory using yeast for inversion are as follows:

Total sugars as invert in sirup (molasses)	89.31%
Total sugars as invert in press cake	0.52
Total sugar undetermined or inversion losses	1.17
Total sugar as invert entered in mill juice	**100.00%**
Total sugars as invert lost in manufacture % cane	**0.24%**

Many articles on the chemical control of high-test molasses have been published, most of which have appeared in the *Proceedings of the Association of Cane Sugar Technologists of Cuba.* Some of the earlier publications did not stress the need for conversion of all sugars to an invert basis, and several have advocated rather complicated systems. The control based on weights and analyses of mixed juice and of the product offers no new problems except the Clerget determination in those factories which do not ordinarily maintain a true sucrose control.

831. Cooling Molasses Before Storage. Losses of total sugars in storage have been recognized as due to failure to cool the molasses after it leaves the concentration pans. F. A. Lopez-Ferrer [28] who has written extensively on the subject of high-test molasses, shows that if the high-test molasses is placed in the storage tanks much above 40° C., the losses will be considerable. A molasses of 78.12 total sugars stored at 60° C. dropped to 74.34 total sugars in twenty-three days, and the sugars then decreased very slowly until they reached 73.82 six weeks later. On the other hand, a molasses stored at 38° C. showed 79.65 total sugars with no change during three months. Lopez-Ferrer's laboratory experiments indicated that 43° C. was the maximum temperature at which the high-test molasses should be put into the storage tanks.

Continuous cooling of the molasses described by Osuna [29] is effected by adding one or two steam jet vacuum boosters to a pan so that a vacuum of 29.6 in. is attainable. The sirup from the vacuum pans after concentration enters the cooling pan at 145°–150° F., and leaves at 85° F. at the rate of 130 gpm., the entire operation being continuous.

Intermittent cooling has been satisfactorily carried out in one large factory by using four calandria pans under ordinary vacuum from the central condenser system (25 in.). A small air leak in the bottom of the pan aids circulation and cooling. About 200,000 gal. per day are readily cooled in this way from 150° to 100° F. or below. This cooling capacity is slightly more than that reported by Osuna for the single high-vacuum pan described above.

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[28] *15th Annual Proc. Cane Sugar Tech. Cuba* (1941), p. 203.

[29] *14th Annual Proc. Cane Sugar Tech. Cuba* (1940), p. 283.

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CHAPTER 36

SUGAR HOUSE CALCULATIONS

832. Introductory. All materials to be dealt with by the chemist are composed of sucrose and non-sucrose, the latter including water, dextrose, levulose, organic non-sugars, and inorganic matter (ash). Certain of these substances persist throughout the manufacture, others through but one or two stages of it. A knowledge of the proportions of these substances in the original material, products and by-products, is the basis for the construction of algebraic equations, with which yields, quantities, capacities, etc., may be calculated.

For the purposes of the usual calculations, in addition to the proportions in which the various constituents are present, certain relations between the constituents themselves are often required, such as purity and saline coefficient. The problems are very simple when a constituent of the original material passes practically unchanged through the processes, e.g., the fiber in dry milling.

The following formula illustrates the principles involved in many of the sugar house calculations. The water used in saturating the bagasse in milling cane dilutes the extracted juice; the percentage of this dilution is ascertained by means of an equation based upon the fact that the solids (Brix) of the extracted absolute juice are present in the mixed juice:

Let 100 = the weight of mixed juice;
b = degree Brix of mixed juice;
B = degree Brix of absolute juice;
x = weight of dilution-water in mixed juice;
$100 - x$ = the weight of absolute juice.

Then,

$$100b = B(100 - x) \quad \text{and} \quad x = \frac{100B - 100b}{B} = \frac{100(B - b)}{B}$$

This is the usual dilution formula and is used because it is the diluted juice that is weighed or measured. The value of x multiplied by the percentage of dilute juice extracted from the cane gives the dilution in terms of the weight of the cane.

In a similar way, calculations may be based upon the fiber of the cane, the dry matter of the press cake, the ash, or a constituent of it, etc.

Since most of the numbers ascertained in sugar analysis are not absolute, the results of sugar house calculations based upon them are approximations, but are usually sufficiently accurate for the purposes of the manufacturing control.

The full work of deducing the formula is uusally given in the following paragraphs, with a view to assisting the beginner in the construction of formulas that are not given or that are necessitated by special conditions.

833. Dry Milling Formula. The fiber is the constant:

Let 100 = the weight of the cane;
B = the weight of the bagasse from 100 cane;
F = the percentage of the fiber in the cane;
F' = percentage of fiber in the bagasse;
x = percentage of juice extracted.

Then,

$$x = 100 - B \tag{1}$$

$$F'B = 100F \tag{2}$$

whence

$$B = \frac{100F}{F'}$$

Substituting the value of B in (1)

$$x = 100 - 100F/F' = 100(F' - F)/F'$$

The similarity of this formula and that for calculating dilution (Sec. 832) is noticeable. This should be expected since in the one the constant (Brix) is diluted and in the other the constant (fiber) is concentrated, i.e., its percentage relation to the bagasse as compared with cane increases.

834. Dilution Formulas. The formula for the dilution of the mill juice in terms of the diluted or mixed juices has been given in Sec. 832. To reduce this number and the following dilution number to percentage terms of the weight of the cane, it is necessary to multiply the values of x by the percentages of mixed and absolute juice, respectively. The dilution per cent absolute or undiluted juice is calculated as follows.

Let 100 = the weight of absolute juice;
B = the Brix of the absolute juice;
b = the Brix of the mixed juice;
x = the percentage of dilution in terms of the absolute juice.

Then, $100 + x$ = the weight of the mixed juice, and since all the solids (Brix) of the absolute juice are found in the mixed juice, $b(100 + x) = 100B$, and $x = 100B/(b - 100) = 100(B - b)/b$. This number has sometimes been employed to indicate the quantity of imbibition water that has been used, though in fact it indicates only the part of the water that has passed into the juice.

835. Concentration and Evaporation Formulas. These formulas are similar to those for dilution and are derived in the same way.

Let 100 = the weight of the juice, etc.;
b = the Brix of the juice;
B = the Brix of the concentrate;
x = the percentage, by weight, of water evaporated.

Then,

$$100b = (100 - x)B$$

whence

$$x = \frac{100(B - b)}{B} \quad \text{(See Table 23, p. 707.)}$$

The percentage of evaporation by volume is derived as follows.

Let 100 = the volume of the juice, etc.;
b = the Brix of the juice of g specific gravity;
B = the Brix of the concentrate of G specific gravity;
x = the percentage, by volume, of water evaporated.

Then

$$GB(100 - x) = 100gb$$

and

$$x = 100\left(1 - \frac{gb}{GB}\right) \quad \text{(See Table 24, p. 708.)}$$

Having two solutions of known degrees Brix, B and B', determine the degree Brix of a mixture composed of the volumes, V and V', of these solutions having specific gravities of G and G'. Then,

$$x = \text{degree Brix required} = \frac{VBG + V'B'G'}{VG + V'G'}$$

If there is no great divergence in the specific gravities, the formula may be used for practical purposes as follows.

$$x = \frac{VB + V'B'}{V + V'}$$

Formula for the calculation of the water required, per cent by weight, to reduce a sugar solution of a given density to any required density.

x = per cent of water required;
B = initial degree Brix;
b = Brix after dilution;
$\frac{B - b}{B} = E$;
$\frac{100E}{1 - E} = x$, the per cent required.

By substitution for E,

$$x = 100\frac{(B - b)}{b}$$

To determine the volume V of a sugar solution before concentration.

b = degree Brix;
s = the specific gravity of the solution before concentration;
B = degree Brix;
S = specific gravity after concentration to a volume of 100.

$$V = \frac{100SB}{sb}$$

836. Commercial Sugar Formulas. (*a*) This formula has a wide application in the sugar house control and in the estimation of the capacities required in crystallizers, etc. It employs the polarization of the primary material and gives the yield in terms of this material.

Let x = the percentage yield of commercial sugar of p polarization and S per cent dry matter;
100 = the weight of the primary material (massecuite, molasses, etc.) of P polarization and B per cent dry matter (Brix);
M = purity of the residual molasses.

Then $P - px/100$ = the weight of sucrose (pol) in the molasses;
$B - Sx/100$ = the weight of dry matter (Brix) in the molasses.

Since the purity of a sugar material is the percentage of sucrose (pol) in its dry matter (Brix) we have

$$M = 100\,\frac{P - px/100}{B - Sx/100}$$

By clearing of fractions, transposing, and reducing,

$$x = \frac{100P - BM}{p - SM/100} \qquad (a)$$

the yield of commercial sugar. If the product is refined sugar of 100° polarization, as is customarily assumed in refinery work, the formula reduces to

$$x = \frac{100P - BM}{100 - M}$$

(*b*) This formula uses the purity instead of the polarization as above and is applicable in the calculation of the yield of sugar from massecuites, molasses, etc. It also gives percentages in terms of the primary material.

Let x = the percentage of commercial sugar of C' purity coefficient and S per cent of dry matter;
100 = the weight of primary material, C its purity and B its Brix;
M = the coefficient of purity of the residual molasses.

Then $BC/100$ = weight of sucrose (pol) in the material;
$Sx/100$ = the weight of dry matter in the sugar;
$\frac{Sx}{100} \times \frac{C'}{100} = \frac{C'Sx}{10{,}000}$, the weight of sucrose (pol) in the sugar;
$B - Sx/100$ = the weight of solids (Brix) in the molasses;
$\frac{BC}{100} - \frac{C'Sx}{10{,}000}$ = the weight of sucrose (pol) in the molasses.

As in the preceding sugar formula, an equation based upon the purity of the residual molasses is formed:

$$M = 100 \frac{\left(\frac{BC}{100} - \frac{C'Sx}{10{,}000}\right)}{\left(B - \frac{Sx}{100}\right)}$$

By clearing of fractions, transposing, and reducing, we have

$$x = 100 \frac{C - M}{C' - M} \times \frac{B}{S} \qquad (b)^1$$

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[1] The previous formula (a) will be seen to be identical with the one above except that the first employs the polarization and the Brix, and the second the purity and Brix, of the primary material and of the commercial sugar. The polarization—Brix—purity relationship gives

$$\frac{100P}{B} = C \quad \text{and} \quad \frac{100p}{S} = C'$$

$$P = \frac{BC}{100} \quad \text{and} \quad p = \frac{SC'}{100}$$

Substituting these values in the formula (a)

$$x = \frac{100P - BM}{p - SM/100}$$

we have

$$x = \frac{\frac{100BC}{100} - BM}{\frac{SC'}{100} - \frac{SM}{100}}$$

Clearing of fractions, we have

$$x = 100 \frac{(C - M)}{(C' - M)} \times \frac{B}{S},$$

which is the second formula (b).

(*c*) This formula has the same applications as the preceding. It is derived the same way, but is based upon moisture-free materials.

Let x = the percentage yield of anhydrous sugar in terms of the dry matter (Brix) in the primary material, and let the other letters have the same meaning as in the previous formula (*b*).

Then $100 - x$ = the dry matter (Brix) of the residual molasses;

$C'x + M(100 - x) = 100C;$

whence

$$x = 100\frac{C - M}{C' - M} \tag{c}$$

This formula is used for calculating available sugar in stock in process in raw sugar work. (See p. 610.) When the product has a polarization of 100 (sucrose) the formula then becomes $x = 100\frac{C - M}{100 - M}$ which is the formula used in refineries to calculate the yield of refined sugar from the purity of a massecuite and its resulting sirup (molasses).

(*d*) Another widely used available sugar formula deals with purities only and gives the yield of sucrose (pol) in terms of the sucrose (pol) present in the primary material.

Let x = the yield of sucrose (pol) present in the commercial product, per cent of the anhydrous sucrose (pol) (C) present in the original material, and let all other letters have the same significance as before.

It is evident that this resolves itself into the previous formula multiplied by the sucrose (pol) per cent dry matter (Brix), i.e., the purity, of the sugar (C') divided by the sucrose (pol), per cent dry matter (Brix) (purity) of the primary material (C) or

$$x = 100\frac{C'(C - M)}{C(C' - M)}$$

This formula was developed by Noël Deerr who used the letters s, j, and m for the symbols, giving $x = 100\frac{s(j - m)}{j(s - m)}$, and it is widely known as the *sjm* formula of Deerr. (*Cane Sugar*, Manchester, 1911, p. 509.) See Sec. 804.

When C' becomes 100, i.e., the product is sucrose, the formula then is

$$x = \frac{100(C - M)}{C(100 - M)} \times 100 \tag{d}$$

This formula is sometimes called the drop in purities formula.

The above formulas may be applied in the calculation of the yield of sugar from the cane or juice. In fact it has been shown on p. 614 that the last formula given corresponds to the theoretical retention figure of the Winter-Carp-Geerligs formula, $100[1.4 - 40/C)]$ if the purity 28.57 is substituted for *M*. These formulas find their chief uses in calculating the yield of sugar in process in massecuites, etc.

837. Crystallizer Capacity. The commercial sugar formulas may be used in estimating the capacity required in certain machinery, notably pans, crystallizers and molasses tanks.

Let it be required to estimate the quantity of crystallizer massecuite of 94° Brix and 60° purity that would be produced from clarified juice of 20° Brix, 18 per cent sucrose (pol), and 90° purity, the sugar that has been extracted having an analysis of 96° polarization and 99 per cent dry matter: Using formula (*a*), Sec. 836, we have

$$x = \frac{(100 \times 18) - (20 \times 60)}{96 - (99 \times 60/100)} = 16.39$$

sugar per cent of the weight of the juice. Let the juice be 80 per cent of the weight of the cane, then 16.39 × 0.80 = 13.11 sugar per cent cane. The sugar contained 99 per cent dry matter, therefore 13.11 × 0.99 = 12.98 per cent dry sugar. The juice contains 20 × 0.80 = 16 per cent dry matter (Brix solids) on cane, and 16 – 12.98 = 3.02, the percentage of dry matter (Brix) on cane going into the crystallizer massecuite; 3.02 ÷ 0.94 = 3.21, the massecuite per cent cane. Massecuite of 94° Brix weighs 94.4 lb. per cu. ft., therefore 3.21 ÷ 94.4 = 0.034 cu. ft. massecuite per 100 lb. cane, or 0.68 cu. ft. per ton of cane. Massecuite swells considerably, owing to the crystallization of its sugar and the decomposition of certain salts (see p. 230), and it may also be diluted from time to time (see p. 220). An increase of volume of 25 per cent is safe allowance for alteration, or 0.68 + 25 per cent = 0.85 cu. ft. Further, the massecuite should remain in the crystallizer about four days, the time depending upon the size and type of the crystallizer, so we then have 0.85 × 4 = 3.4 cu. ft. per ton of cane daily milling capacity. If the milling capacity is 1850 tons of cane the crystallizer capacity should be 3.4 × 1850 = 6290 cu. ft. The size of the crystallizers will depend upon the size of the vacuum pans, and under the usual conditions with pans of 12 ft. in diameter striking 1000 cu. ft. would be approximately 1000 + 25 per cent = 1250 cu. ft.; 6290 ÷ 1250 = 5+, the required number of crystallizers to actually hold the massecuite. Allowance must also be made for one empty crystallizer to receive massecuite and one to be discharging to the centrifugals, or in all seven crystallizers of about 1250 cu. ft. gross capacity each.

This estimate is based upon juice of exceptionally high purity. In actual practice the estimates should be upon the juice of the lowest purity that is likely to prevail over an extended period at any time of the manufacturing season. The lower the initial purity, the larger will be the quantity of crystallizer massecuite. (Cuban factory practice, for example, would be to have about double this number of crystallizers for a grinding capacity of 1850 tons of cane per day.)

838. Mixed Massecuites. Proportion of Molasses and Sirup. These massecuites should be boiled to a definite purity, depending upon that desired in the molasses to be obtained from them. This formula assumes that the densities of sirup and molasses are the same. This is sufficiently accurate for practical purposes.

(*a*) Let 100 = total weight of massecuite in the strike;
P = purity of the sirup;
p = purity of the molasses to be boiled-in;
M = purity of the required massecuite;
x = percentage by weight of the strike to be formed of molasses;
$100 - x$ = percentage by weight of the strike to be derived from sirup.

Then,

$$x = \frac{100(P - M)}{P - p}$$

This formula may be applied with less accuracy when P is the purity of a footing or nucleus upon which a strike is to be completed with molasses.

(b) This calculation may be made with greater facility by Cobenze's[2] method for mixtures, illustrated in the diagram and example, Fig. 202. Let 85 be the coefficient of purity of a sirup and 55 that of a molasses, and let it be required to make a massecuite of these having a purity of 65°. Arrange the numbers as in the diagram. Subtract the purity number for the massecuite from that of the sirup and the number for the molasses from that of the massecuite and arrange the remainders as shown in the diagram; the remainder, 10, is the number of parts of sirup required and 20 that of the molasses. If the percentage of each constituent is required, divide the number of parts of each by the total number of parts and multiply by 100. As in the previous method, using purities only in calculating the mixture, it must be assumed that the densities of the solutions are the same.

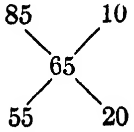

FIG. 202. Cobenze's Diagram for Calculating Mixtures.

This method may be used for all mixtures and facilitates the solution of many otherwise complicated problems. If any three numbers used in the diagram are given the other two are readily ascertained.

839. Chart for Rapid Calculation of Mixtures. A chart which may be used in place of Cobenze's diagram is described by J. S. Baker[3] (Fig. 203). It is based on the relation

$$x = \frac{C - B}{A - B}$$

where A = concentration of stronger constituent;
B = concentration of weaker constituent;
C = intermediate desired concentration;
x = fraction of final mixture made up of stronger constituent.

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[2] A. Cobenze, *Compendium der prakt. Photografic,* ninth edition, p. 379; Van Nostrand's *Chemical Annual,* 1913, p. 563.

[3] *Ind. Eng. Chem.,* September, 1934, p. 910.

To construct this chart, draw perpendicular identical scales at the ends of a base line having 100 equal divisions (regular coordinate paper will serve) with *B* and *C* on the left and *A* on the right, and let the base line scale, as measured from the left end, equal the percentage of the final mixture consisting of the stronger constituent.

To use the chart, draw a horizontal straight line at the elevation above the base line, representing the desired concentration *C* (in the example 65).

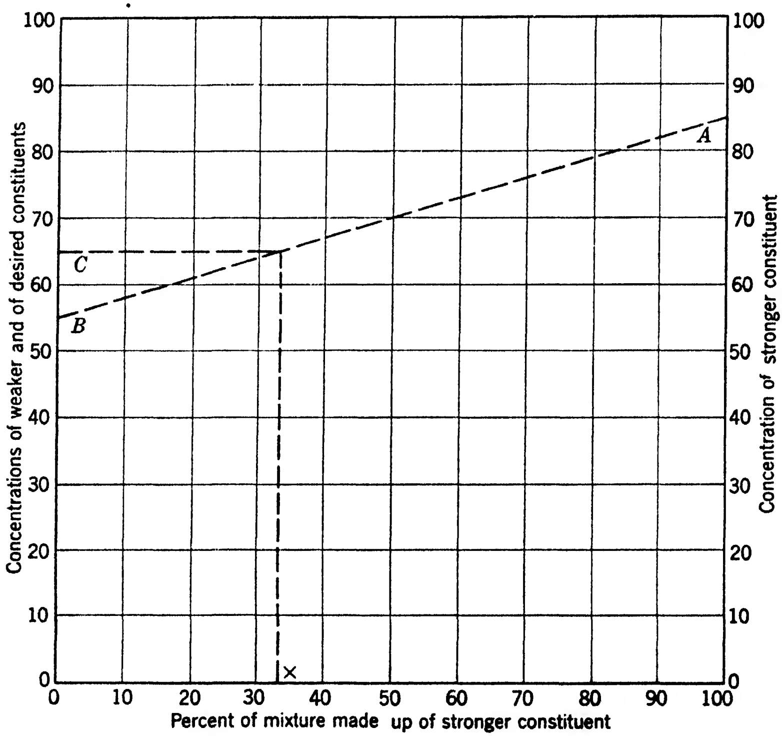

FIG. 203. Diagram for Calculating Mixtures.

Connect another straight line from the point representing the weaker concentration *B* (55) on the left scale with the point representing the stronger concentration *A* (85) on the right scale. The horizontal distance as measured on the base line, from the left perpendicular to the point of intersection of these two lines gives the percentage (100x) of the final mixture made up of the stronger constituent (66⅔ per cent).

A similar chart may be made for a mixture of different gravities, the units of the base line in this case representing the volume per cent. Such a chart as that described by Baker might readily be painted or drawn on a blackboard with a straightedge for use on the pan floor.

The same method as given by Baker is described by Auguste Esnouf[4] in connection with a series of interesting sugar house nomograms for dilution and other similar problems.

840. Calculation of Added Water in Double Filter Pressing of Scums (Cachaza). This is a problem in dilution and may readily be solved by the formula in Sec. 834 for dilution in terms of the normal juice. Let B of the formula represent the solids in the first press cake, b, the solids in the reduced cake; and x, the percentage of added water. Then $x = 100(B - b)/b$. The following example will illustrate the calculation.

Example. A press cake contained 35.06 per cent dry matter and after reduction with water to a cream this contained 14.24 per cent dry matter:

$$x = \frac{100(B - b)}{b} = \frac{100(35.06 - 14.24)}{14.24} = 146.2 \text{ per cent}$$

841. Weight Per Unit Volume at Other than Standard Temperatures. The weight per unit volume (gallon or cubic foot) of a sugar solution of known Brix at any standard temperature (17½° C., 20° C., 27½° C.) is calculated by multiplying the weight of the unit volume of water at the standard temperature by the specific gravity of the sugar solution corresponding to the Brix. This is the method by which the tables for 20° C. (p. 731) and that for 27½° C. (p. 736) were calculated. The calculation at other than standard temperatures for which specific gravities are not available involves other procedures.

A sugar solution does not expand on heating at the same rate as water. The expansion also varies with the sugar content of the solution. Tables showing the rate of expansion on heating of sugar solutions to various temperatures have been prepared, and the figures given in Table 11, p. 698, compare the volume at these temperatures with the volume of the same solution at 20° C.

The weight at any desired temperature of a unit volume of a sugar solution of known Brix may be calculated from this table in the following example.

Example. What is the weight of a cubic foot of 50° Brix sugar solution (standard temperature 20°) at 27½° C.? (The temperature of 27½° is selected for the example as the result is already figured by other means.)

1 cu. ft. of 50° Brix sugar solution at 27½° as compared to 20° will occupy (table 11, Bureau Standards, p. 698) 1.0028 cu. ft.

1 cu. ft. of 50° Brix solution at 20° C. weighs 76.69 (table 30, p. 731) then the same solution at 27½° C. will weigh

$$\frac{76.69}{1.0028} = 76.476$$

This compares with 76.478 given in Table 31 on p. 736 for a 50° Brix solution at the standard temperature of 27½° C., as calculated from the weight of water and the specific gravity.

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[4] *Intern. Sugar J.*, August, 1934, p. 310.

A much simpler method which is sufficiently accurate for all practical purposes is as follows.

In the example given above:

The Brix at 20° = 50.00
The hydrometer correction to 27½° (table 29, p. 730) = 0.59

The observed Brix at 27½° on a hydrometer standardized at 20° C. = 49.41

The weight per cubic foot of a solution of 49.41° Brix at 20° C. (table 30, p. 731) is 76.49.

This method is well within the limits of accuracy of tank measurements and may be used in taking stock in the factory or refinery (see p. 610), i.e., to the Brix at the standard temperature (20° C.) is applied the correction for the required temperature, and from this *observed* Brix the weight per cubic foot is obtained from the table; or the Brix reading may be taken at the tank temperature without correction to 20° C., and the weight per cubic foot corresponding to the observed Brix at tank temperature taken from the table for 20° C.

842. Calculations Based upon the Relation between the Ash and Sucrose, etc. These methods are used in ascertaining whether sugar or other matters are destroyed or decomposed and removed in a boiling process, for example. The mineral constituents of the materials are assumed to remain unchanged during the process, therefore a comparison of the saline coefficients should show whether sucrose has been destroyed, and similarly whether other constituents have been decomposed. A method of this class must be used with great caution, since very slight inaccuracy of analysis or loss of mineral matter may lead to an erroneous conclusion. The true or Clerget number should be used in sucrose comparisons.

Example. (All sucrose numbers are by the Clerget method.) A clarified juice containing 15 per cent sucrose, 0.3 per cent ash, and 50 saline coefficient was evaporated to sirup containing 50.7 per cent sucrose, 1.02 per cent ash, and a saline coefficient of 49.71. The reduction of the saline coefficient by 50 − 49.71 = 0.29 indicates decomposition of sucrose. This loss in percentage terms of the sucrose in the juice is calculated by finding what per cent 0.29, the decrease in the saline coefficient is of the original coefficient 50, in this case 0.58.

This method has long been used in beet and cane sugar manufacture in estimating losses, but its application is somewhat limited by the fact of the deposition of a part of the ash constituents on the evaporating surface and in storage tanks. C. H. A. Zimmermann[5] proposes to utilize only the soluble sulfates of the sulfated ash in this method, since the salts from which they are formed persist through the manufacture. The material is ashed as usual with the addition of sulfuric acid. The ash is transferred to an ashless filter or an alundum filtering crucible, and the soluble matter is removed by wash-

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[5] *Intern. Sugar J.*, **16**, 338 (1914).

ing with hot water. The residue is dried and weighed. The difference between the original weight of ash and this residue is the weight of the soluble sulfates. The calculations are made as before.

The similar work of Davies, Yearwood, and colleagues[6] has been referred to (Sec. 826). They used the chloride content of juices, sirups, and massecuites to determine losses during evaporation and pan boiling. The chloride was determined potentiometrically and losses of considerable extent were found, involving simultaneous destruction of solids (Brix) and sucrose, and the simultaneous destruction of sucrose and reducing sugars, none of which would be indicated by the ordinary methods of control (purities, glucose ratios, etc.). The chloride method is less open to question than previous methods based on entire ash or soluble ash since the chloride will not change during evaporation or boiling. The percentages of chloride are so small as to require determinations of great accuracy, but Davies reports that results based on chloride ratios are reliable.

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[6] *Sugar*, December, 1942, p. 30.

CHAPTER 37

METHODS OF CANE PURCHASE

843. Systems of Cane Payment. The problem of how much the grower shall receive for his cane is one that has been studied extensively in all sugar-producing countries. The subject resolves itself into three steps: (1) What general system of payment shall be chosen? (2) How shall the cane be evaluated? (3) What proportion of the total value of the cane shall be allotted to the grower?

844. Selection of a System. *Flat Price.* The simplest of all systems is the so-called flat price, by which the cane is paid for at a price based on the price of raw sugar. In Cuba, for example, the flat price is a predetermined percentage of 96° sugar on the weight of cane: from 4 to 7 per cent of the sugar (or the value of that amount of sugar) goes to the grower, depending on the competition for cane. Louisiana formerly paid about one dollar for each cent of the value per pound of 96° test sugar, i.e., if raw sugar was worth 3.50¢ per lb., cane was worth $3.50 per ton. (This method of basic price calculation is still in use for standard cane.) By this flat price system, the quality of the cane as regards sucrose content and purity of juice is not considered. The disadvantages of such a system are obvious. "The same price is paid for wood and water as for sucrose."[1]

845. Purchase on Analysis. Systems of payment which in one way or another take into account the amount of sucrose in the cane are in use in most of the large cane-producing countries of the world except Cuba.

The advantages of a system of payment on test are several; in fact, the disadvantages are only those involved in selecting an equitable arrangement for purchase which is agreeable to both the grower and the manufacturer. There is some expense and trouble entailed in the sampling, analyzing, and calculating which any such system requires, but the returns to the factory and to the grower are greatly increased. Even in Cuba where the cane is purchased on a flat price, it has been found valuable in many large factories to have a special laboratory in which the routine checking and comparing of the juice of canes from different fields and railway switches is carried out.

The grower knows that he will be paid on the basis of the quality of the cane, as well as its weight, and he therefore employs agricultural methods which will give him increased sucrose yield as well as increased cane yield. The delivery of cane to the mill as soon as possible after cutting is also of

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[1] Moberly, *Proc. 5th Congr. Intern. Soc. Sugar Cane Tech.* (Brisbane, 1935), p. 134.

prime importance to the grower with such a system, and the delivery of fresher cane greatly reduces costs at the factory. The losses due to cane lying in the field are discussed in Sec. 19.

The choice of any system involving purchase on a sucrose (or polarization) basis immediately brings us to the second step in the problem, viz., the method of evaluation. It is obvious that whatever system may be selected for cane purchase on the basis of its analysis, the primary requisite is a representative sample, either of the juice from the cane or of the cane itself. Therefore, before considering the systems of evaluation, the methods of sampling will be discussed.

SAMPLING FOR PURCHASE SETTLEMENT

846. Difficulties of Sampling. The first practical difficulty in any cane purchase system which may be devised lies in the sampling. The best method, where practicable, is to sample the juice at the mills in the factory, but this can be done only where the cane arrives in fairly large lots, e.g., in car loads. Where the cane is delivered in carts or trucks, or where the cane is dumped on a pile, as frequently done in Louisiana, sampling of cane stalks must be resorted to and the juice extracted in a laboratory mill. This is admittedly less satisfactory, but results in practice in Louisiana have shown that fairly close correlation may be maintained between average sucrose (pol) in the factory and average sucrose (pol) for the cane purchased.[2]

In commenting on Littell's report cited above, Moberly[3] says that "experience with cane sampling in South Africa has never been very favorable," experiments having shown that "samples of less than 250 lbs. of cane stalks were not representative of 25 ton loads. This weight of sample is too large to handle in our laboratory mill if many samples are to be tested."

847. Methods of Sampling. *Cane in Large Lots Direct to Mill.* The number of the cart or car of cane is noted, and the chemist is given a ticket bearing this number and is advised to prepare for sampling. If cart cane is to be sampled, it is advisable to group a number of loads under one test. The chemist notes the position of the cane on the conductor or elevator, using markers if need be, and after the cane reaches the crusher he allows a definite interval for the expressed juice to wash the rolls and then preferably samples continuously and automatically or at brief intervals manually during the passage of the cane. The sample is sent to the laboratory with the ticket corresponding to it. Sample follows sample in this way.

This method, in use in one of the large factories in Louisiana, has shown very close agreement between growers sucrose (pol) and factory sucrose (pol).[4]

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[2] R. H. Littell, *Sugar J.*, January, 1941.

[3] *Intern. Sugar J.*, February, 1942, p. 45 (abs.).

[4] Walter Godchaux, Jr., private communication.

The use of revolving cane knives may render this method of taking samples impractical, and a method is used in Queensland[5] by means of which a signal system suitably geared to the carrier makes contact with a colored electric bulb when the cane to be tested enters the crusher and lights a second bulb of another color when the cane has passed through the crusher. The arrangement is homemade and may be modified and extended to change samples automatically, to sound warning bells in the testing laboratory, etc.

848. Cane in Carts or Trucks. The practice in Louisiana is to make an average test of the deliveries from each individual grower each day. The method used at Raceland Factory is as follows:[6]

> The wagons, trucks, etc., are weighed and the kind of cane noted. Two stalks of cane of each variety are taken from each bundle in the load. In the case of cars, narrow and standard gauge, five stalks per ton are taken from the cars. The stalks are then put into a rack divided into as many sections as there are growers, composited in this way all during the hauling day, picked up at the end of each hauling day, sub-sampled according to different varieties, and analyzed. Sub-sampling is usually done at the end of the day when the various varieties can be assorted, and a large enough number taken from these bundles to have at least fifty stalks for grinding. The juice from these fifty stalks is caught and sent to the laboratory for analysis.

This method of sampling and compositing is similar to that described by Littell[7] at Billeaud Plantation, Louisiana, and both these factories report that the agreement between yearly average factory sucrose (pol) and growers sucrose (pol) is so close as to be within the limits of error of analysis. As stated above Moberly[8] says this is contrary to experience in South Africa where cane sampling has never been satisfactory except with samples too large to grind in a laboratory mill.

849. Analysis. *Grinding Cane Samples.* Because of the large volume of work entailed in handling the cane and juice samples, it is customary to have a separate laboratory devoted to this work. A power-driven laboratory three-roller mill is used to grind the cane stalks, and the rolls can be adjusted to give any desired extraction. It is important to maintain the adjustment so that the extraction obtained is in a constant relationship with the factory mills. "The sucrose factor used is calculated each day by the ratio of the average sucrose analysis of the test mill to the factory mill."[9] (Sucrose in these quotations means pol.)

Brixing and Polarizing Juice. On receipt of the samples by the laboratory, the samples and tickets are arranged in regular order. A numbered cylinder is filled to overflowing with the strained juice, and the remainder of the sample

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[5] Moberly, *Facts About Sugar,* October, 1937, p. 401 (with diagram) (abs.).

[6] Walter Godchaux, Jr., *loc. cit.*

[7] *Loc. cit.*

[8] *Loc. cit.*

[9] Littell, *loc. cit.*

is held in reserve pending the completion of the test. The cylinders should be arranged in convenient groups of ten, for example, and after the latest to arrive has been standing ten minutes the hydrometers are inserted. After a further interval of five minutes the degree Brix and temperatures are to be noted. The observed Brix, temperatures, and corrected Brix should be entered on a duplicate of the load ticket. These operations should be conducted very systematically, allowing a definite period for each.

The polarization test should be made by Horne's dry lead method, p. 515. The laboratory equipment should include a considerable number of 200 ml. (approximate) cylinders like *C* of Fig. 130. Each of these should have a mark etched on it to indicate approximately the 100-ml. point. A spoon having a conical bowl should be provided for measuring the dry lead. It should hold about 1 gram of lead, "struck" measurement.

Arrange a group of these cylinders, corresponding to those used for the density determination, and fill each to the mark with juice. Add a measure of lead to the juice, cover the cylinder with the palm of the hand, and mix its contents by vigorous shaking. Filter, polarize, and calculate the polarization by Schmitz's table, p. 755. Enter this test on the ticket.

SYSTEM OF EVALUATION

850. Selection of the System. By the methods already described, or similar ones which may be prescribed to meet local conditions, we now have the Brix, polarization, and purity (by calculation) of a representative sample of the juice of the cane delivered by the grower. The next step is the estimation of its value to the manufacturer, or the selection of some equitable criterion figure which will be acceptable to the grower and manufacturer.

The method of evaluation, based on the analysis of the juice, may fall into one of several classes: (1) density of the juice, (2) sucrose (polarization) in the juice, (3) sucrose (polarization) in the cane, (4) purity and sucrose (pol) of the juice and other factors such as fiber and variety, (5) available sugar in the juice (or the cane).

851. Density Basis. Formerly in some countries purchases were based on Brix (or Baumé) of the juice, but the disadvantages of the system are obvious, since green cane and ripe cane may command an equal price. This method of purchase on density of juice is now obsolete.

852. Polarization Basis. A method adopted generally in Louisiana which has the value of simplicity depends solely on the polarization (called sucrose) of the so-called normal (undiluted) juice as calculated by factors from the factory crusher juice or from laboratory mill juice. By this system, a standard sugar cane is one giving a normal juice polarizing 11.50 to 12.00 per cent. (In some sections the polarization standard is 11.50 to 12.50 per cent.) Two per cent reduction is made for each 1/10 per cent reduction in polarization from 11.49 to 9.50 per cent; and a premium of $\frac{5}{10}$ per cent is paid for each $\frac{1}{10}$ per cent increase from 12.00 to 12.50 per cent; with 1 per cent premium for each $\frac{1}{10}$ per cent increase above 12.50 per cent.

The simplicity of this system may offset some of the evident disadvantages. It needs no extended argument to show that the purity of the juice, as well as the sucrose content, is of great importance to the manufacturer, both in the return of sugar which he gets and in the cost of manufacture. It is probable that in the long run the premiums and penalties will take care of these inequalities but individual variations may remain.

853. Sucrose (Polarization) in the Cane. This is the method in use in South Africa.[10] The sucrose (pol) in the cane is calculated from the sucrose (pol) in the crusher juice and the Java ratio (see p. 582) for the particular mill. Allowances are made by means of factors for Java and Coimbatore canes as compared with Uba cane, which is the basis. These factors were arrived at through the use of average values of juice purity, fiber content and overall recovery for the different varieties[11] so to some extent the purity and fiber are taken into consideration.

The system described is more scientific than the one in use in Louisiana but it is still an arbitrary basis of evaluation.

854. Purity and Sucrose (Pol), Fiber, etc. The system in use in South Africa from 1927 to 1937[12] involved other factors besides sucrose (pol) in cane. The sucrose (pol) content of the cane was used to calculate the value but bonuses were paid on purities of crusher juice above 89° and penalties were assessed on purities below 83°. Juice below 78° purity was rejected irrespective of sucrose content, and cane with sucrose less than 9 per cent was also rejected. Adjustments for fiber content were also allowed for but never put into effect because of the difficulties of analysis.

This system was nearer a scientific basis than those already described, but apparently the added complications were not considered of sufficient value to retain, since the method has been discarded for the simpler one involving sucrose (pol) in cane only.

855. Available Sugar in the Cane (or Juice). This method is the most scientific of those so far described, and it is widely used throughout the world. Available sugar formulas are discussed in detail on pp. 611–624 and may be divided into two classes. (1) The available sugar may be taken as that recoverable under certain ideal or average conditions. (2) The available sugar may be taken as that recoverable under the actual conditions in which the factory is working.

The first of these methods is in use in Australia and involves the so-called commercial cane sugar (CCS) formula which is worked out as follows:

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[10] Hinchy, *Intern. Sugar J.*, February, 1942.

[11] Coates and Hinchy, *Intern. Sugar J.*, January, 1941, p. 11.

[12] Hinchy, *loc. cit.*

$$\text{Brix in first expressed juice} \times \frac{100 - (\text{Fiber} + 3)}{100} = \text{Brix in cane}$$

$$\text{Pol in first expressed juice} \times \frac{100 - (\text{Fiber} + 5)}{100} = \text{Pol in cane}$$

$$\text{Brix in cane} - \text{Pol in cane} = \text{Impurities in cane}$$

$$\text{Pol in cane} - \frac{\text{Impurities in cane}}{2} = \text{Commercial cane sugar}$$

The resulting factor is applied to the whole weight of the cane.

Another formula which has been used is the widely known *SJM* formula of Noël Deerr (see Sec. 804) which is a yield formula based on the purity of the sugar, purity of the juice, and purity of the final molasses. A specialized form of this formula is that of Winter and Carp (see Sec. 806) which was published several years previous to the *SJM* formula, but which is the equivalent of it with a sugar of 100 per cent purity and a final molasses of 28.57. The formula of Winter and Carp is as follows:

$$x = S\left(1.4 - \frac{40}{C}\right)$$

where x = available sucrose (pol) per cent cane;
S = per cent sucrose (pol) in terms of weight of cane;
C = purity of juice.

The Winter-Carp formula falls into the class of theoretical recoveries, since it employs a fixed purity of final molasses and not the actual purity. It is used in Java with factors which have been computed from the results of the previous campaign, depending on the variety of cane. Puerto Rico also uses a modification of this formula,[13] but takes into account the fiber content of the cane (according to varieties) by modifying the percentage paid to the growers. Varieties with higher fiber, e.g., Uba, are paid for at a lower rate.

The use of this available sugar formula as a basis for cane payments was suggested by Spencer in earlier editions of this handbook and the method he outlined follows.

The chemist should first determine two dry milling factors. (See Sec. 783.) While operating the milling plant for a short period without saturation water, sample and separately analyze the juice from the crusher and the mixed juice from the entire milling plant. Calculate the factors: (1) Brix of the undiluted juice divided by Brix of the crusher juice, and (2) polarization of undiluted juice divided by polarization of the crusher juice.

It is obvious that where laboratory mills are used for extracting juice from cane samples, similar factors must be developed between small mill juice and factory mill juice. As has already been emphasized the setting of the small mill must be maintained to give the same extraction at all times.

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[13] del Valle, *Proc. 5th Congr. Intern. Soc. Sugar Cane Tech.* (Brisbane, 1935), p. 145.

Multiply the Brix of any given juice sample (crusher or small mill) by the Brix factor and the polarization by its factor, and the purity by dividing pol by Brix. The available sugar for this purity may be obtained from the table on p. 766, and this in turn to a basis of the factory by applying the boiling house efficiency (Sec. 807) and normal undiluted juice extraction numbers.

Example.

Factor for the Brix	0.978
Factor for the polarization	0.965
Brix of the crusher juice	18.0
Polarization of crusher juice	15.7
Assumed mill extraction, undiluted juice	78.0
Average efficiency number (BHE) of the factory	97.0
Brix of calculated undiluted juice (18.0 × 0.978)	17.60
Pol calculated undiluted juice (15.7 × 0.965)	15.15
Purity calculated undiluted juice (15.5 ÷ 0.1760)	86.08
Pol extracted by the mills in undiluted juice (15.15 × 0.78)	11.82% cane

Referring to Copp's Table 37*a* on p. 762 we find 98.66, the theoretical retention of 96° sugar corresponding to 86.1 purity. This number multiplied by the pol extracted gives the yield of sugar per cent cane on a basis of 100 factory efficiency, 11.82 × 98.66 = 11.66, and since the efficiency number is 97, then 11.66 × 0.97 = 11.31, the percentage yield of 96° sugar that may be expected from the cane for the factory in question.

A method used in Jamaica[14] based on the Winter-Carp formula and using no milling factors is as follows. The factory figure for tons of cane per ton of commercial sugar is first multiplied by the relationship between the polarizations of the crusher juice for the factory and the polarization of the crusher juice for the particular purchased cane, and this in turn is multiplied by the relationship between the theoretical retentions (Table 37) for the two juice purities, factory and purchased cane. This gives the tons of cane per ton of commercial sugar for the purchased cane.

Example. Factory crusher juice pol 12.23, purity 77.80°, factory tons cane per ton commercial sugar = 12.18. Purchased cane, crusher juice pol 14.55, purity 83.38. Then,

$$12.18 \times \frac{12.23}{14.55} \times \frac{88.59}{92.04} = 9.85 \text{ tons of purchased cane per ton of commercial sugar}$$

It will be seen that the above simple method is based on no assumption except that the growers' cane will bear the same relationship to the whole factory cane as do the crusher juices of the two canes.

Further Refinements of Available Sugar Formulas. An extended study of the fundamental principles of cane payments by G. S. Moberly[15] outlines methods of determining recoverable sugar including factory losses, which are

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[14] Personal communication from Mr. J. A. Tannock.

[15] *Proc. 5th Congr. Intern. Soc. Sugar Cane Tech.* (Brisbane, 1935), p. 134.

not taken account of in the yield formulas given above. However, it is doubtful whether such refinements are necessary in actual practice. Variables, e.g., varieties of cane, for which arbitrary or average allowances must be made are so numerous that the exactness of the formula employed probably has little bearing. As will be shown in a later section, the percentage paid to the grower is also a matter of arbitrary or average determination which casts further doubt on the need for an exact formula.

856. Rejections. Most systems of cane payment allow for the rejection of frozen canes, burned canes, etc., either on an arbitrary basis or on analysis of the juice (acidity, etc.) and such rejections may be made even after the cane has been ground. Burned cane in some countries may be rejected if not brought to the mill within a certain period after burning, e.g., five days.

Frozen cane in Louisiana is generally judged on the basis of acidity. Acidity greater than 4.50 ml. (0.1 *N* akali on 10 ml. of juice) is cause for rejection and acidities above 2.25 ml. are penalized at the rate of 3.775 per cent of the payment for each additional 0.25 ml. of acidity. Fort and Lauritzen[16] showed that total acidity is not a satisfactory basis for estimation of deterioration, and they recommended that excess acidity as measured by the drop in pH or by distillation should be the means of evaluation.

857. Percentage or Amount Paid to the Grower. In the systems already outlined and in any of the numerous other systems which have been devised, the question still remains, "What portion of the total value of the cane (no matter how determined) shall go to the grower?" Here again we find that the methods range from arbitrary percentages to formulas arrived at by more or less scientific methods.

In Puerto Rico, the mills pay the growers from 62.5 to 65 per cent of the yield of 96° sugar, the percentage varying according to the average fiber content of the variety cane purchased.[17] In Louisiana the price paid is on a system of premiums and allowances. It has already been shown (Sec. 852) that one set of premiums and allowances is based on the polarization of the juice. The price for standard sugar cane is $3.50 per ton when 96° sugar is 3.50 cents per lb. ($1.00 per ton for each cent of price per pound of sugar) with 3 per cent increase for each ¼ cent above 3.50 cents up to 3.75 cents and 3 per cent reduction for each ¼ cent below 3.50 cents down to 2.75 cents, the lower limit.

As against these arbitrary methods of payment, the elaborate calculations of Coates and Hinchy[18] for South Africa are of interest. These take into account differences in average analyses of different varieties, overall recoveries, manufacturing costs, fixed costs, etc., to arrive at the total additional value of other varieties than Uba cane, which is taken as a base. The basic price in South Africa is also calculated from formulas which include the average cost of production of a ton of cane, average sucrose (pol) content

[16] *Ind. Eng. Chem., Anal. Ed.,* May, 1938, p. 251.

[17] del Valle, *loc. cit.*

[18] *Loc. cit.*

of cane, and cost of manufacture of a ton of sugar. Allowances are made to small growers for greater costs of production.[19]

Australia has an equally elaborate method of distribution based on the CCS formula and a work coefficient, but according to O'Brien[20] the calculations have led to an arbitrary set of figures, so that the grower now gets between 70 and 71 per cent.

Evidently there is no ideal or perfect system of cane purchase since the methods used in different countries vary so widely and are so frequently changed.

Admittedly any system based on analysis is superior to a system which pays a flat price regardless of cane quality, but it is doubtful whether complicated methods of calculation result in a more equitable distribution than do the simple systems which have been worked out through economic custom. No matter what system is chosen, it generally happens that approximate figures, estimates, allowances, and averages enter into the calculations somewhere so that, to some extent at least, the final figures are more or less arbitrary.

[19] Hinchy, *loc. cit.*

[20] *Proc. 5th Congr. Intern. Soc. Sugar Cane Tech.* (Brisbane, 1935), p. 146.

CHAPTER 38

FERMENTATION AND MICROORGANISMS IN SUGAR MANUFACTURE AND REFINING

by

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858. Introduction. In cane sugar manufacture fermentation is an ever-present problem from the time that the juice is first extracted until the sugar reaches its final state of purity and is distributed as the refined granulated product. Although the susceptibility of the intermediate products of raw sugar manufacture to deterioration decreases from the raw juice to the finished sugar, this fact does not constitute a criterion of the relative importance of the problem at the different stages of the manufacturing process, since the finished products are subjected to these changes over much longer periods than any of the products of the preceding stages. Cane juice as freshly extracted[1] contains a very large number of microorganisms, which gain access to it from the rind of the cane and from the trash and dirt adhering thereto, and also from other more extraneous sources. This number may vary from less than one hundred thousand to several million per cubic centimeter, depending upon the cleanliness of the cane and the sanitary condition of the mill in which the extraction is effected. Where the rind of the cane is uninjured and where special methods of extraction are used so as to eliminate all chances of outside infection, cane juices may be obtained in a practically sterile condition. Freshly extracted cane juices possess a considerable degree of germicidal action,[2] and for several hours after their extraction the number of microorganisms decreases as a result of enzymic action. In frozen or burned cane this protective action is lost, and as a result the deterioration of the juice of such cane progresses with undiminished rapidity from the time that it is extracted until the action is checked either by outside intervention or by the exhaustion of the food supply for the microorganisms.

859. Bacteriology in the Factory and Refinery. The operation of a modern raw cane sugar factory or refinery requires bacteriological as well as chemical knowledge. The raw sugar operator is concerned with the problem of mill sanitation as well as with the necessity of producing a sugar of a microbial content within limits which do not render it liable to deterioration

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[1] W. L. Owen, *La. Expt. Station, Bull. 125.*

[2] C. A. Browne, *J. Am. Chem. Soc.*, **28** (1906).

in storage. The superintendent of a raw sugar factory recognizes the importance of applying preventive measures against the rapid development of microorganisms in any and all stages of manufacture from the mill to the centrifugals. He is equally aware of the necessity of having his sugars conform to the factor of safety (see Sec. 342) and of maintaining warehousing conditions that will not be conducive to moisture absorption.

The refiner is becoming increasingly concerned with the problems connected with the production of sugars as free as possible from microbial contamination in general and specifically free from excessive number of thermophilic bacteria. The industrial users of refined sugar are becoming more and more discriminative as regards the bacteriological purity of the sugars that they use, and for beverage purposes, where the product is unpasteurized, their desire is to obtain a sugar having the lowest possible number of viable microorganisms.

Bacteriology is playing an increasingly important role in the sugar industry in other more recent developments such as in the increasing use of yeast invertase in the production of invert or high-test molasses in Cuba and Puerto Rico, and in the possibilities of the manufacture of yeast for cattle feed from surplus cane.

860. Classification of Microorganisms. The microorganisms occurring in cane juices may be classified according to their economic importance in sugar manufacture, as follows:

Bacteria. 1. Species which destroy sucrose without the formation of gum and which are of importance only in the juice.

2. Gum-forming species which include the dextran forming *Leuconostoc mesenteroides,* and the levan-forming mesentericus group of bacteria. The former is of importance only in the juices and the dilute liquors, whereas the other group persists throughout the entire manufacturing process and may cause a slow inversion of sucrose in sirups and molasses.

3. Species known as thermophiles, which develop at temperatures between 46°–73° C., are sometimes responsible for the deterioration of hot clarified juices, and often develop in the filter presses and juice settlers. Among the best known of these species is the *Microspira Northii.*[3]

Mold Fungi. 1. Very strongly inverting species which are active in juices as well as in sirups and constitute the most active agency in the deterioration of raw sugars, example, *Aspergillus repens,*[4] and *Aspergillus niger.*

2. Species which induce only a feeble inversion of sucrose at the density corresponding to that of the molasses films surrounding sugar crystals, but which may induce considerable losses of sucrose in cane juices—example, the Citromyces group, and *Monilia fusca* (Browne).

3. Species which are very prevalent in sugar products, but are of little economic significance, owing to their weak inverting power—example, *Monilia nigra* (Browne).[5]

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[3] C. A. Carpenter and H. F. Bomonti, *Rep. Hawaiian Sugar Technologists,* 1921.

[4] W. L. Owen, *Facts About Sugar,* 1925.

[5] C. A. Browne, *Ind. Eng. Chem.,* **10** (1918).

Yeast. 1. Those species which are found in raw juices, and which induce an active inversion of sucrose at that density.

2. Species of non-sporulating yeast known as Torulae, containing no invertase, and which induce an active destruction of reducing sugar, and often a selective action upon levulose, in cane sirups and molasses, and in the molasses films surrounding raw sugar crystals.

861. Factors Affecting the Number of Microorganisms in Cane Juices. The number and kind of microorganisms occurring in cane juices depend to a very large extent upon the amount and nature of the substances adhering to the rind of the cane from which the extracted juice becomes infected during milling. The mere mechanical filtration of freshly extracted cane juices has been found to eliminate on an average approximately 75 per cent of the microorganisms originally present, whereas the use of vegetable carbons and Filter-Cel[6] in the proportions in which they are ordinarily used in sugar manufacture removes approximately 99 per cent of the microorganisms originally contained in the juice. It is evident from these results that it is of very great importance to use the most efficient type of juice strainers in order to eliminate at the very beginning as many as possible of the microorganisms with which the freshly extracted juice is contaminated.

862. Mill Sanitation. From the fact that freshly extracted cane juices are immediately infected with a very large number of microorganisms, many of which are capable of rapidly inverting sucrose, it is highly important to avoid exposing them to the deteriorative action of the latter for more than the briefest possible interval. It is also essential to keep the mill as free as possible from deteriorated material such as fermented portions of bagasse or particles of trash which have absorbed fermented juice. Frequent washing and cleaning of mills, and the use of steam at frequent intervals, has been known to reduce greatly the drop in purity between crusher and mill juices.[7] It still is a debatable question whether the use of antiseptics at the mills is to be recommended as a regular procedure, owing to the large quantities that would be required and the expense that would be involved. However, the use of suitable germicides from time to time in connection with the washing down and the cleaning of the mills, or after any undue delay caused by breakdowns or other causes is to be recommended. Formaldehyde in the proportion of 1:200[8] or chloride of lime in the proportion of 1:50 will prove a very efficient method of maintaining the sanitary conditions of mills. This is, of course, a much higher concentration than would ever be required for the preservation of juices, for which purpose formalin in the proportion of from 1:5000 to 1:10,000 is usually recommended (see Chapter 10). For the elimination of infection from fermented juices, especially of the viscous type, chloride of lime will be found to be the more efficient of the two germicides, owing to its greater penetrating power.

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[6] W. L. Owen, *Intern. Sugar J.*, **26,** 1924.

[7] W. L. McCleery, *Rept. Hawaiian Sugar Technologists,* 1925.

[8] W. L. Owen, *La. Expt. Station, Bull. 153.*

863. The Influence of Temperature upon the Nature of the Fermentation of Cane Juices. Although the nature of the infection of the juice from sound cane at the time of its extraction is subject to comparatively little variation, the type of the predominant fermentation varies considerably at different times. One of the most prominent factors determining the course of the spontaneous fermentation of cane juice is the temperature to which it happens to be exposed. Experiments have shown [9] that at temperatures below 20° C. the viscous type of fermentation seems to predominate more frequently than at temperatures between 20° and 30° C. This would tend to explain the susceptibility of juices from frozen cane to dextran fermentation.

The temperature limits of two of the most important groups of gum-forming bacteria found in cane juices are as follows:

GROUP	MIN. TEMP.	OP. TEMP.	MAX. TEMP.
Leuconostoc mesenteroides	11–14	30–35	40–43
Bacillus mesentericus (Levan forming bacteria)	15–22	37	45

Other factors which tend to determine the type of fermentation which cane juices undergo are (1) pH of the juices and (2) the amount and kind of contaminating material that gains access to it.

864. Number of Microorganisms at the Various Stages of Raw Sugar Manufacture. In the process of sugar manufacture the microorganisms occurring in the raw juice tend to be successively eliminated at all of the subsequent stages of the manufacturing process, by the methods of clarification and by the temperatures employed in connection therewith.

Recent investigations by Millstein, Tobin, and McCleskey of the Department of Bacteriology of the Louisiana State University [10] using an improved tryptone medium have shown the following values for the microbial content of various sugar house products.

SAMPLE	LOW	HIGH	
Raw juice (*a*)	8,000,000	16,000,000	(*a*) Early in the season.
Raw juice (*b*)	570,000,000	750,000,000	(*b*) Late in the season.
Clarifier effluent	0	11	
Press juice	0	51,000	
Evaporator	200	3,300	
Storage tank	1,300	7,100	
Crystallizer	2,000	44,000	
Massecuite	1,200	10,600	
Raw sugar	340	5,100	
Molasses	3,000	310,000	

It is evident from the above data that good clarification of the juices in raw sugar manufacture will be reflected in a lower microbial content of the various products at the successive stages of manufacture.

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[9] W. L. Owen, *Intern. Sugar J.*, **17** (1921).

[10] *Sugar J.*, February, 1941, p. 13.

865. Microorganisms in Frozen Cane. In recent years much light has been thrown on the role played by microorganisms in the deterioration of juice from frozen cane, and progress has been made toward the solution of the baffling problems of how the juice from such cane can be efficiently clarified in the sugar factory. Walton and Fort[11] and their co-workers have studied the mannitic fermentation of juices from frozen cane with particular reference to the occurrence of dextran and mannite in jellied molasses made from this material.

The appraisal of the sucrose recovery value of juice from cane injured by freezes of varying intensities has been studied by Fort and Lauritzen[12] who recommend the use of "excess acidity" values for this purpose as determined by the decreases in the pH of such juice or by the titration of its distillate. Investigations have established the fact that "excess acidity" rarely occurs as an isolated problem, but it is almost invariably associated with gum formation as a concomitant factor. McCalip and Hall's[13] investigations on the formation of dextran in sucrose solutions and the precise effects of varying amounts of this material upon the viscosity of these solutions and upon their clarification have shown that in the normal course of the above fermentation the viscosity increase of the solution proceeds to a limited degree only after which it decreases with the increasing acidity of the solution.

In spite of the extensive study which this subject has received in Louisiana during recent years much more must be learned to explain why juices from frozen cane should invariably undergo fermentations which are predominantly of the gum-forming type. If we attribute this phenomenon to the mere exposure by splitting freezes of the interior of the stalk to the unrestrained development of microorganisms, we are confronted by the enigma that juice extracted from the cane and allowed to ferment usually develops a predominantly alcoholic and acetous fermentation. Owen has shown that low temperatures are conspicuously conducive to the viscous fermentation and also that the juice which has been frozen appears to be more susceptible to this type of fermentation. Regardless, however, of how the juice may be handled in the laboratory in order to promote its fermentation by dextran-forming species of microorganisms the investigator will rarely be as successful in inducing it as is the combination of circumstances where cane is subjected to splitting freeze and allowed to stand over in this condition for short periods of time. It might well be that the *Leuconostoc mesenteroides* is one of the predominant species in the epiphytic flora of cane and hence the rupture of the tissue by freezing results in a predominantly high inoculation of the juice with this organism. If, as a sequel to this condition, low temperatures happen to prevail we would have as a result an ideal setting for the formation of highly viscous cane juices.

[11] C. F. Walton and C. A. Fort, *Ind. Eng. Chem.*, **23**, 1295 (1931).

[12] C. A. Fort and J. I. Lauritzen, *Ind. Eng. Chem., Anal. Ed.*, **10**, 251 (1938).

[13] M. A. McCalip and H. H. Hall, *Proc. 6th Congr. Intern. Sugar Tech.* (Baton Rouge, 1938).

In confirmation of the above observation McCalip and Hall[14] have noted that when warm weather quickly follows freezing temperatures sugar factories are usually confronted with the problem of rapidly increasing acidities in juices with diminishing trouble from "gumminess."

Of all the fermentations that occur spontaneously in juice from frozen or damaged cane, the alcoholic is probably responsible for the greatest destruction of sucrose. However, as Fort and Lauritzen[15] have pointed out, the net loss in recoverable sucrose from this fermentation is usually less than where gum fermentation takes place even where the actual destruction in sucrose in the latter is lower. A typical alcoholic fermentation while introducing no clarification difficulties is responsible for considerable error in the analysis of the juice which frequently leads to false assumptions regarding the efficiency of sucrose recoveries. As an example the above authors show that an alcoholic concentration of 0.64 per cent by volume in a juice depresses the Brix to an extent which results in an apparent increase in purity of 1.2 points.

866. Thermophilic Bacteria in Refined Sugars. Until approximately a decade ago interest in the bacteriology of sugars was largely confined to the groups of microorganisms concerned with its deterioration in storage. In 1928 Cameron and Williams[16] published the results of their investigations of the occurrence of thermophilic bacteria in refined sugars, which constituted the first demonstrated proof of a definite thermophilic flora in this commodity. Subsequent investigations by Cameron and his co-workers[17] of the National Canners Association Laboratories and by James[18] of the United States Bureau of Chemistry traced the incidence of spoilage in canned corn to the number of viable spores of thermophilic bacteria in the sugars used in canning. All these investigations led to the elaboration by the National Canners Association in 1931 of the following standards for canners sugar. These standards are as follows:

1. *Flat sour spores.* In the five samples examined there shall be a maximum of not more than seventy-five and an average of not more than fifty spores per 10 grams.

2. *Thermophilic anaerobic spores.* These shall be present in not more than 60 per cent of the five samples and in any one sample to the extent of not more than four of the six tubes.

3. *Sulfide Spoilage Bacteria.* These shall be present in not more than two (40 per cent) of the five samples and in any one sample to the extent of not more than five spores per 10 grams. This would be equivalent to two colonies in the six inoculated tubes.

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[14] *Loc. cit.*

[15] C. A. Fort and J. I. Lauritzen, *Sugar Bull.*, April 15, 1939.

[16] E. J. Cameron and C. C. Williams, *Centr. Bakt.*, II, **76**, 28–37, 1928.

[17] E. J. Cameron and W. D. Bigelow, *Ind. Eng. Chem.*, **23**, 1330 (December, 1931).

[18] L. H. James, *J. Bact.*, **13**, 409 (1927).

The above standards were supplemented in 1935 to include all thermophilic bacteria irrespective of their proved connection with spoilage in the canning industry.

867. Source of Thermophilic Bacterial Flora of Sugars. Investigations by Cameron and Bigelow,[19] Owen and Mobley,[20] and Calton,[21] have shown that spores of thermophilic bacteria are brought into the refinery with the raw sugars and frequently increase in number in certain parts of the refinery. It sometimes happens that refined sugar contains a larger number of spores of these bacteria than the raw sugar from which they are made. Consistent seasonal and diurnal variations in the concentration of these spores in the products of Louisiana refineries were studied by Owen and Mobley[22] who also found that starch used in confectioners' sugar is a prolific source of thermophilic bacteria, an observation which was also confirmed by Cameron.[23] More recent work of Hall and Keane[24] on the effect of irradiation upon the viable thermophilic spores in beet sugars will be referred to later.

Recent investigations of the occurrence of thermophilic bacteria in the products from the various stages of raw sugar manufacture by Millstein, Tobin, and McClesky[25] show the following values:

SAMPLE	LOW	HIGH
Raw juice	14	170
Seip effluent (clarifier)	0	4
Dorr effluent (")	0	8
Press juice	3,700	205,000
Evaporator	200	15,500
Storage tank	16,100	38,500
Crystallizer	350	15,000
Massecuite	1,700	17,100
Raw sugar	100	2,000
Molasses	1,200	16,500

The present status of the problem of manufacturing cane sugars conforming to the standards adopted by the canning industry is that these can be attained either by: (1) segregating that portion of the refined sugar shown by actual analysis to be acceptable for canning, or (2) having the entire granulated sugar output conform to the standard by exercising the necessary vigilance in refinery operation.

If the second method is to be followed in attaining the standard, it is necessary to maintain scrupulous cleanliness throughout the refinery, to attain the highest efficiency in all phases of processing, especially in the filtration of

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[19] E. J. Cameron and W. D. Bigelow, *loc. cit.*

[20] W. L. Owen and R. L. Mobley, *Ind. Eng. Chem.*, **24**, 1042 (1932).

[21] F. R. Calton, *Ind. Eng. Chem.*, **28**, 1235 (1936).

[22] W. L. Owen and R. L. Mobley, *loc. cit.*

[23] E. J. Cameron, *loc. cit.*

[24] H. H. Hall and J. C. Keane, *Ind. Eng. Chem.*, **31**, 1168 (1939).

[25] *Sugar J.*, **3**, No. 9, 13–14 (February, 1941).

liquors, and to avoid holding over sirups in which thermophilic development may take place.

868. Bacteriologically Pure Sugars for Special Industrial Usages. Just as the requirements of canners for sugars containing a minimum number of thermophilic bacteria have been fully fulfilled by sugar refiners, so other special bacteriological requirements in other industries are already in the process of formulation into definite standards. Owen and Mobley[26] have proposed the following standards governing the limit of microorganisms other than thermophiles which should be tolerated in sugars used in the beverage industry:

1. That the percentage of mold infections of any lot of refined sugars should not exceed 20.
2. That the number of mold spores per gram should never exceed 10.
3. That not more than 20 per cent should contain viable yeast cells.
4. That the number of yeast present per gram should not be more than 50.
5. That the average number of bacteria other than thermophilic (standards for which are already in use) should not be over 50, and the maximum tolerated should be 100.

Sugars of sufficiently high purity to satisfy the most discriminating users both as to their freedom from mesophilic (organisms growing at medium temperatures) as well as from thermophilic bacteria can be produced by the refineries. For the control of the mesophilic microbial content of sugars scrupulous attention must be directed to the protection of the sugar from exposure to dust around the centrifugals and as it leaves the granulators. Attention must also be directed to the maintenance of high efficiencies in the clarification of the liquors and especially in their filtration.

869. Sterilization of Refined Sugars by Irradiation. Killing thermophilic spores in dry white sugar by the use of radiant energy rays has been demonstrated to be practical in beet sugar work by Hall and Keane.[27] In this work the lamps giving off the rays were installed in the granulator so that the sugar spilled over the entire length of the lamps. It was found that turbulence in the sugar during irradiation was an advantage. By this treatment about 50 per cent of the spores were killed.

A more recent application of germicidal ultraviolet rays on a commercial scale is described by M. R. Lederer.[28] He shows that experiments carried on in several refineries in the east indicate that germicidal ultraviolet rays may be used on a plant scale to sterilize dry refined sugars. The development of such irradiation methods will probably assure thermophilic free sugars at all times, but it should be pointed out that the production of such sugars may

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[26] W. L. Owen and R. L. Mobley, *Facts About Sugar*, **30**, No. 12 (December, 1935).

[27] *Ind. Eng. Chem.*, September, 1939, p. 1168.

[28] Before Sugar Industry Engineers, December, 1942.

be attained without such aids by the scrupulous attention to manufacturing details already referred to in this chapter.

870. Presence of Growth-Promoting Substances. In addition to the special requirements connected with the manufacture of sugars of approximate sterility there is also an increasing interest in the production of sugars having a minimum content of those impurities constituting what is known as growth-promoting substances. These may be merely elements of nutrition requisite for the growth of microorganisms or they may be of the nature of bioses or vitamins, which accelerate growth where the minimum elements of nutrition are present. Hall, James, and Stuart[29] in their study of growth-promoting factors in refined sugars from various sources have found that the presence of this material as evidenced by the ability of the sugars to support yeast growth varies from season to season. The stimulating substances in sugars were found to be soluble in alcohol as a result of which sugars crystallized from alcoholic solution are practically free from these growth-promoting factors.

871. Other Types of Fermentation in Sugar Cane Products. The viscous fermentation of cane juices is associated with a very pronounced reducing action, resulting in the marked bleached appearance of the fermented juice. Various deoxidation products result from this fermentation, among which Browne[30] found mannite to be the most commonly occurring. The amount of mannite occurring in fermenting juices, however, is subject to wide variations, owing to the readiness with which it may be utilized as a source of energy for other groups of microorganisms.

In addition to the viscous fermentation of cane juices and the ordinary alcoholic fermentation, there are other less commonly occurring, and less well-understood fermentative phenomenon, of which Browne has made a very thorough study. Among these may be mentioned the cellulosic fermentation, in which sucrose is assimilated by certain forms of bacteria such as Bacterium xylinium, with the formation of cellulose. This organism forms clumps of gelatinous sheaths in cane juices, and Browne found that the product of this fermentation consisted of cellulose, and represented approximately 7 per cent of the total sugars fermented.

Among the interesting products of fermentation in sugar cane products may be mentioned mannan, which Browne identified in the deposits and scums formed in fermenting juices, sirups, and molasses, and also chitine, which he isolated from the scums which form on the surface of molasses left over in the hot room. These scums consist of the growth of the fungus Citromyces, and Browne's investigations have shown that the former are characterized by an exceptionally high fat content (27.5 per cent) which in composition very strikingly resembles butter fat.

872. Sirups with Distinctive Flavors by Controlled Fermentation Processes. Another bacteriological development which is attracting more

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[29] H. H. Hall, L. H. James, and L. S. Stuart, *Ind. Eng. Chem.*, **25**, 1052 (1933).

[30] C. A. Browne, *J. Am. Chem. Soc.*, **28** (1906).

and more attention on the part of sirup and molasses producers is that of modifying and improving the flavor of these products by means of an induced and controlled fermentation by species of aroma-producing yeast and bacteria. Hall[31] has found that the flavor and aroma that characterizes Barbados molasses is due to the presence of end fermentation products formed by a species of bacteria known as *Clos. saccharolyticum* in the presence of yeast cells, the latter only serving as a "flavorgenetic" raw material from which the flavor is produced by the bacteria. By inoculating sirups and molasses from other sources with these cultures obtained from Barbados molasses the flavor of the latter may be imparted to the former.

Investigations by the writer[32] have resulted in a process for imparting more desirable flavors to sirups and molasses by inducing a controlled fermentation thereof with certain species of torulae, a form of pseudo-yeast, which rarely contain invertase and even where they exhibit inverting action in dilute substrates practically never do so in densities corresponding to that of sirups and molasses. However, they induce a vigorous fermentation of reducing sugars at the normal density of cane sirups, and it has been observed that some of the esters formed in this fermentation impart particularly desirable flavors to sirups and molasses. An increased smoothness is also imparted to the fermented product which gives it, apart from its improved aroma, a quality resembling the effect of added commercial glucose to a typical cane sirup.

[31] H. H. Hall, *J. Bact.*, **33**, 577 (1935).

[32] W. L. Owen, "Bacteriology in Relation to the Cane Sugar Industry," *Reference Book of the Sugar Cane Industry of the World*, La. Planter, 1928.

CHAPTER 39

ANALYSIS OF LIMESTONE, LIME, SULFUR, AND SULFUROUS ACID

ANALYSIS OF LIMESTONE[1]

873. Introductory. For analysis by carloads the rapid method described later will be sufficient, but composite samples representing a week's deliveries should be analyzed by the longer method giving the following constituents: (1) insoluble, (2) iron and aluminum oxides, (3) calcium carbonate, (4) magnesium carbonate, (5) calcium sulfate, if present.

874. Preparation of the Sample. Fragments should be chipped from at least seventy-five pieces of the stone and reduced to ¼-inch size, then mixed and subsampled by quartering. The small sample should be reduced to a fine powder (60 mesh) in an iron mortar or on a grinding plate. Particles of metallic iron, from the mortar or plate, should be removed by stirring the powder with a magnet. Sift the powder through an 80-mesh sieve and mix it thoroughly by sifting or otherwise.

875. Determination of Insoluble Matter. Treat 1 gram of the powdered limestone in a beaker, with 50 ml. of water and 20 ml. of concentrated HCl, being cautious in adding the acid to prevent the projection of particles of the material from the beaker. Cover the beaker with a watch glass and heat the liquid ten to fifteen minutes. Collect the residue on a quantitative filter paper, wash it thoroughly with hot water, and reserve the filtrate, *A*, for further treatment. Place the filter and residue in a tared platinum crucible and incinerate. The weight of this residue, *A*, multiplied by 100 is the percentage of insoluble matter.

876. Determination of Iron and Alumina. Add a *slight* excess of pure ammonia to the filtrate, *A*, while it is still hot, boil it until only a slight odor of ammonia can be detected, collect the precipitate on a small filter, filtering rapidly while the solution is hot. Wash with hot water and incinerate. Keep the filtrate, *B*.

The residue consists of the mixed oxides of iron and alumina (Fe_2O_3, Al_2O_3). Multiply the weight of the residue by 100 to obtain the percentage.

877. Determination of Calcium. To the filtrate from the iron and alumina determination, *B*, corresponding to 1 gram of the stone, add sufficient hydrochloric acid to render it slightly acid. Concentrate this solution to about 250 ml., neutralize it with ammonia, heat to boiling, and add an

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[1] Adapted to conform to *Methods of Analysis*, Great Western Sugar Company (Denver, 1920) through courtesy of the author S. J. Osborn, Chief Chemist.

excess of boiling-hot ammonium oxalate solution. Digest on the water bath for about one hour (or set aside for twelve hours) until the precipitate has completely settled out, leaving a clear supernatant liquid. Decant through a filter paper, washing the precipitate two or three times by decantation with hot water containing a little ammonium oxalate and hydroxide. Then collect the precipitate of calcium oxalate on a quantitative filter, wash with cold water (filtrate *C*), dry and incinerate the filter in a tared platinum crucible, then ignite the residue strongly. The residue consists of almost pure calcium oxide (CaO), and may be weighed as such.

If the limestone contains a considerable amount of magnesium, the precipitate should be redissolved on the filter by warm dilute hydrochloric acid and the calcium reprecipitated by neutralization with ammonia and the addition of 1 or 2 ml. of ammonium oxalate solution. Filter, wash with cold water (add filtrate and washings to *C*), dry and incinerate the filter as before and ignite the residue strongly. The weight of CaO multiplied by 100 gives the per cent of calcium oxide in the stone. This value multiplied by 1.7847 gives the per cent of calcium carbonate.

The ignited calcium oxide in this determination combines with water and carbon dioxide very readily, hence cooling and weighing should be done as rapidly as possible.

878. Determination of Magnesium. To the filtrate, *C*, from the calcium determination, after concentration to approximately 100 ml., add a slight excess of ammonium hydroxide, chill the solution, then add sodium phosphate solution in excess, drop by drop, with vigorous stirring. After fifteen minutes add a decided excess of ammonia. Set aside during several hours, preferably overnight, to insure a complete precipitation. Collect the precipitate in a Gooch crucible, wash it with dilute ammonium hydroxide, containing 1 part NH_4OH of 0.96 specific gravity to 3 parts water. The washing should be continued until free from chlorides. The precipitate is ammonium-magnesium phosphate; dry it, first at a gentle heat, then increase the temperature to expel the ammonia, and finally ignite it a few minutes in the flame of a blast lamp to convert the residue into magnesium pyrophosphate. Cool the residue in a desiccator and weigh it. The weight of the magnesium pyrophosphate ($Mg_2P_2O_7$) multiplied by 0.36208 gives the corresponding weight of magnesium oxide. The magnesium is present in limestone as carbonate. Multiply the weight of the pyrophosphate by 0.7573 and the product by 100 to ascertain the percentage of magnesium carbonate in the stone.

879. Determination of Carbon Dioxide. It is not usually necessary to determine the carbon dioxide, as it may be calculated from the quantity required to combine with the lime and magnesia, except when sulfates are present. The loss on ignition (Sec. 883) may be taken as representing the carbon dioxide.

If the determination of carbon dioxide is made, Schroetter's or similar apparatus, may be used as follows. The description refers to Fig. 204. Fill the tube on the left, to above the upper bulb, with concentrated sulfuric acid, and that on the right with dilute hydrochloric acid. Weigh the flask and contents, then introduce approximately 1.5 to 2 grams of the powdered limestone, by the opening in the flask, and weigh again. Lift the stopper on the hydrochloric acid tube, and open the stop cock and admit a little acid. In the decomposition of the stone, the carbon dioxide is set free and bubbles through the sulfuric acid, which retains any water vapor that would otherwise pass off with the gas. Repeat this operation from time to time until no more carbon dioxide is disengaged. Heat gently to expel the carbon dioxide from the solution, cool, and weigh. After cooling and wiping the apparatus, it should be placed inside the balance case a few minutes before weighing. The loss in weight is that of the carbon dioxide set free. Divide this weight by that of the limestone used and multiply the quotient by 100 to obtain the percentage of carbon dioxide.

The carbon dioxide in the limestone used in sugar manufacture is almost entirely combined with calcium; a small portion is sometimes in combination with magnesium. Occasionally the stone contains a vein of dolomite, a carbonate of calcium and magnesium.

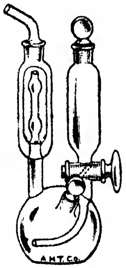

Fig. 204. Schroetter's Alkalimeter

In the absence of gypsum, calcium sulfate, if either the percentages of calcium or magnesium and carbon dioxide are given, the percentages of the two carbonates may be calculated: The percentage of calcium oxide (CaO) × 1.7847 = percentage of calcium carbonate ($CaCO_3$); the percentage of carbon dioxide in the magnesium carbonate ($MgCO_3$) multiplied by 1.9164 = the percentage of magnesium carbonate.

Example. A sample of limestone contains 54.8 per cent calcium oxide and 43.4 per cent carbon dioxide; required, the percentages of calcium and magnesium carbonates.

Calculation.

54.8 × 1.7875 = 97.96, per cent calcium carbonate
97.96 − 54.8 = 43.16, carbon dioxide in the calcium carbonate
43.4 − 43.16 = 0.24, carbon dioxide in the magnesium carbonate
0.24 × 1.916 = 0.46, the per cent magnesium carbonate.

Many sugar house chemists calculate the carbonates in this way, in order to economize time. This method often will supply all the information necessary relative to the purity of the stone, but it is not usually advisable to depend entirely upon it. A serious objection to this process is the fact that there may be slight errors in the determinations of the calcium and carbon dioxide which would lead to false deductions. It is advisable, as a rule, to determine both the bases and the acids.

880. Determination of Sulfate. The limestone may contain small quantities of calcium sulfate. Digest 5 grams or more of the powdered limestone with hydrochloric acid, using heat. Dilute the solution, filter it, and wash the residue thoroughly with hot water. Concentrate the filtrate in a beaker to a volume of about 50 ml., heat it to boiling and add a solution of barium chloride, a few drops at a time, maintaining the boiling temperature. Remove the beaker from the heat, after each addition of the chloride, to permit the barium sulfate to settle, and test the supernatant liquid for sulfate. Continue the boiling of the solution and the additions of the reagent so long as a precipitate forms; set aside overnight. Collect the precipitated barium sulfate in a tared Gooch crucible, wash it with hot water, heat it to redness, cool and weigh it. The weight of barium sulfate × 0.343 ÷ weight of limestone used × 100 = percentage of sulfuric anhydride (SO_3); the weight of barium sulfate × 0.5832 ÷ weight of limestone used × 100 = percentage of calcium sulfate.

RAPID METHOD

881. Insoluble and Iron and Aluminum Oxides. Dissolve 0.4–0.5 gram in 50 ml. of water and 20 ml. of concentrated hydrochloric acid in a beaker covered with a watch glass. When effervescence has ceased, heat to boiling and digest on a hot plate for ten to fifteen minutes. Add a few drops of concentrated nitric acid, and boil. Then cool somewhat, make slightly alkaline with ammonium hydroxide, and boil for a few moments. Filter, wash well with hot water, ignite, and weigh as "insoluble" + Fe_2O_3 + Al_2O_3. (Filtrate *A*.)

882. Calcium. *0.2 N Potassium Permanganate:* Dissolve 6.32 grams of the pure crystals in water, filter through asbestos, and make up to 1 liter. One milliliter of this solution will be found to be equivalent to about 0.01 gram of calcium carbonate. Keep in a dark bottle well protected from the light. The solution should be allowed to stand several days, if possible, before it is standardized, and its value will vary but little after this time. As a matter of precaution, however, it is well to restandardize it once a month.

Standardization. Weigh out 1.34 grams of sodium oxalate of the highest purity obtainable, dissolve in water, add 50 ml. of dilute sulfuric acid (1 to 10), dilute to a volume of about 150 ml., heat to 70° C., and titrate to a permanent pink with the permanganate solution. From several such titrations determine the calcium carbonate equivalent of 1 ml. of the permanganate. Sodium oxalate of the Bureau of Standards may be used for a primary standard. Traces of moisture can be removed from sodium oxalate by heating at 120° C. for two hours.

Determination. Dilute the filtrate *A* to a volume of at least 150 ml., heat to boiling, and to the boiling solution add drop by drop from a pipet, buret, or capillary tube, 20 ml. of hot ammonium oxalate (4 per cent solution). By adding the reagent in this manner the calcium oxalate will be precipitated in a granular form which will permit of rapid filtration, and it will not pass through the filter paper. Filter after standing for at least one-half hour and wash thoroughly, but not excessively, with hot water.

Wash the precipitate into a beaker with a jet of hot water, allowing the open paper to cling to the side of the beaker above the liquid. Add 50 ml. of dilute sulfuric acid (1 to 10), pouring it over the surface of the paper. Dilute to a volume of about 150 ml., heat to 70° C., and titrate with the permanganate solution. The reaction requires a short time after the addition of the permanganate, but, after it once begins, the permanganate may be added quite rapidly until the end point is nearly reached. Continue the titration drop by drop until a faint pink color persists, then introduce the filter paper and add a few drops more of the permanganate, if necessary, until the permanent end point is reached.

$$\frac{\text{Milliliters of 0.2 } N \text{ permanganate} \times \text{CaCO}_3 \text{ equivalent per ml.}}{\text{Weight of sample taken}} \times 100 = \%\ \text{CaCO}_3$$

883. Carbon Dioxide. Ignite 0.5 gram in a platinum crucible to constant weight over a good burner, followed by a blast, or in an electric furnace. The loss on ignition is considered to represent the amount of carbon dioxide. Check the efficiency of the furnace or burner occasionally by the ignition of pure calcium carbonate.

If moisture is present in the original sample, it must first be dried for one hour at 100° to 105° C. and then weighed. This precaution is, however, rarely necessary.

Multiply the percentage of CO_2 (loss on ignition) by 2.2742 to obtain the percentage of total carbonates as calcium carbonate. Subtract the percentage of calcium carbonate, as determined above, and multiply the difference by 0.8426 to obtain the percentage of magnesium carbonate.

ANALYSIS OF LIME

884. Sampling.[2]

If the lime is in the form of large lumps, it is difficult to obtain a representative sample. About 1 lb. per ton should be taken to form the bulk sample, which is then reduced to a sub-sample of about ¼ in. diam. in a large iron mortar. Lastly a final sub-sample of about ¼ lb. is taken, ground quickly to pass a 0.5 mm. sieve, and placed in a screw-topped bottle. This serves as the sample for analysis, and if these operations have been carried out with despatch only a negligible amount of CaO will have been converted to $CaCO_3$ during the time taken.

885. Determination of the Available Calcium Oxide in Lime. Add sufficient water (about 30 ml.) to 10 grams of lime, in a mortar, to form a thick milk. Add 150 ml. of a solution of refined sugar of 35° to 40° Brix and mix it intimately with the lime, which forms a soluble saccharate. Transfer the solution and residue to a 200-ml. flask, using a sugar solution of the above composition to wash the last portions from the mortar; make to mark,

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[2] Von Stieglitz, *Intern. Sugar J.*, August, 1941, p. 248.

mix, and filter; titrate 20 ml. of the filtrate with normal hydrochloric acid (Sec. 909), using phenolphthalein as an indicator. The buret reading × 0.028 = the weight of calcium oxide (CaO) in 1 gram of the lime, and × 100 = percentage of available calcium oxide.

886. Determination of the Proportion of Unburned and Slaked Lime. Slake 1 gram of lime with water, add an excess of normal sulfuric acid (Sec. 911) and heat to expel carbon dioxide present; add a few drops of methyl orange solution or other suitable indicator, and ascertain the excess of sulfuric acid used, by titration with normal sodium hydrate (Sec. 913). Calculation: (milliliters of normal sulfuric acid − milliliters of normal soda solution) × 0.028 = the total weight of calcium oxide in 1 gram of the lime, and × 100 = the percentage of total calcium as calcium oxide. This number minus the percentage of available calcium oxide equals percentage of unburned and slaked lime as calcium oxide.

887. Loss of Ignition. Weigh out approximately 1 gram on an analytical balance, in a covered platinum crucible. Ignite to constant weight in an electric furnace or over a blast lamp, cool in a desiccator, and weigh.

$$\frac{\text{Loss in weight}}{\text{Weight of sample taken}} \times 100 = \text{Per cent loss on ignition}$$

ANALYSIS OF SULFUR AND SULFUROUS ACID

888. Sulfur. Estimation of the Impurities. Commercial roll-sulfur is usually very pure. Its quality can generally be satisfactorily determined from its color and relative freedom from dust and small fragments. If an analysis is desired, transfer 0.5 gram of the powdered sulfur to a flask provided with a well-fitted glass stopper. Add at one time an excess of saturated bromine water and shake thoroughly. Water dissolves 2 to 3.25 per cent of bromine at ordinary temperatures, and, as at least 15 parts bromine are required for 1 part of sulfur, it is advisable to use from 275 to 400 ml. of the bromine water to insure sufficient reagent for the oxidation of the sulfur to sulfuric acid. Boil the solution to expel the excess of bromine, collect the residue and wash with hot water; dry and weigh. A Gooch crucible is convenient for collecting the residue. The weight of the residue × 200 = percentage of impurities. The percentage of sulfur may be calculated directly from that of the sulfuric acid in the filtrate (Sec. 880), or with sufficient accuracy for practical purposes, by subtracting the percentage of impurities from 100.

889. Estimation of Sulfuric Acid in Sulfur Dioxide. The following method is recommended by Herzfeld.[3] Pass the sulfur dioxide gas into a 40 to 50 per cent sucrose solution for ten to twenty minutes. Test a portion of the solution for sulfuric acid by the addition of barium chloride and hydro-

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[3] *Zeitschrift,* 1911, 917; *Intern. Sugar J.,* **14,** 113 (1912).

chloric acid. An insoluble precipitate of barium sulfate forms in the presence of sulfuric acid. If a quantitative test is desired, a measured volume of the gas must be used and the precipitated sulfate must be collected in a Gooch filter and be washed with hot water, dried, and ignited to redness. The weight of the barium sulfate × 0.343 = the weight of sulfur trioxide (SO_3) in the volume of gas used.

CHAPTER 40

ANALYSIS OF FLUE GASES

890. Purpose of Analysis. The analysis of flue gases is made as a part of the boiler house control for the purpose of ascertaining whether the full efficiency of the fuel is being obtained. Occasionally factories using the carbonation process use the flue gases as a source of carbon dioxide.

For these purposes the ordinary volumetric analysis is usually employed, and the constituents are reported in volume per cents. The constituents determined are carbon dioxide (CO_2), carbon monoxide (CO), oxygen (O), and nitrogen (N).

891. Preparation of the Apparatus and Reagents. The most convenient apparatus for this analysis is that of Orsat, Fig. 205. A form of this apparatus having a four-way stopcock instead of those on the pipets *B*, *C*, *D*, and *G* is often used, but that illustrated is easier to manipulate and is preferable.

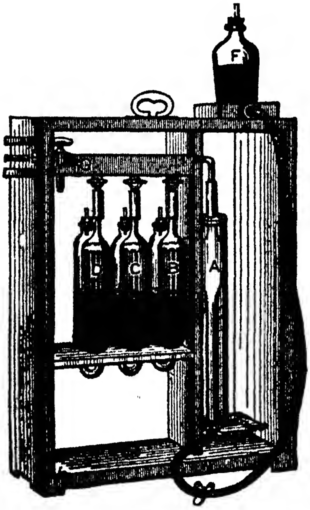

Fig. 205. Orsat Gas Apparatus

The apparatus consists of a water-jacketed buret *A*, for the measurement of the gases and three absorption U-tubes *B*, *C*, *D*, with suitable connections and stopcocks. The absorption branches of the U-tubes are filled with pieces of small-bore glass tubing to increase the surface exposed to the gases.

The absorption tubes are connected with the buret by barometer tubing of very small bore. The branches of the U-tubes at the rear are usually connected with a soft rubber bulb (not shown in the figure) to prevent exposing the solutions to the air and thus weakening them. The water jacket on the buret is for maintaining a fairly constant temperature during the test.

The following solutions are used in the absorption tubes.

For tube B: Use a concentrated solution of caustic potash (KOH) of about 60° Brix.

For tube C: Dissolve 5 parts of pyrogallic acid in 50 parts of hot water and add 100 parts of caustic potash solution of approximately 50° Brix.

For tube D: This tube is to be filled with a solution of cuprous chloride prepared as follows. Place 35 grams of cupric chloride in a bottle, add 200 ml. of concentrated hydrochloric acid and a quantity of copper turnings or copper foil. Stopper the bottle and set it aside for two days, shaking it occasionally, then add 120 ml. water.

Ready prepared cuprous chloride may be obtained of the dealers instead of making it in the laboratory. Twenty-six grams of cuprous chloride should be dissolved in 200 ml. concentrated HCl as above, 120 ml. of water added, and copper trimmings or foil placed in the storage bottle. Each of the tubes in *D* should have a piece of copper wire in it.

The U-tube should be filled half full of the solutions, and the rubber bulb should then be connected with the branches, or in lieu of the bulb, oil may be poured on the surfaces of the liquids. The bulb is preferable, however.

892. Sampling and Analysis of Flue Gases. A piece of ½-in. iron pipe should be inserted into each flue leading to the chimney, reaching about halfway to the center of the flue. A double-acting rubber-bulb pump, with suitable valves, is used in drawing the gas from the flue and discharging it into a soft rubber bulb used as a receiver. The bulb should be filled with the gases and emptied several times, to expel all the air it or the pump may contain, and then be filled with the sample and its rubber tube closed with a pinchcock. It is convenient to have a number of these bulbs so that duplicate samples may be drawn from each flue. A pair of aspirator bottles may also be used for collecting the sample of gas.

In analyzing the samples proceed as follows. Fill the bottle *F*, Fig. 205, with distilled water (which has previously been saturated with the gases to be analyzed), close the cocks on *B, C,* and *D,* and open the three-way cock *G* to the air; lift *F* until the water fills the buret to the upper mark, then close the three-way cock and pinchcock on the rubber tube connecting *F* with the buret, and place *F* on the table.

A U-tube containing a little water and a light plug of cotton in each branch is connected at *E*.

Open the pinchcock and then cautiously open the cock on the U-tube *B* and let the caustic-potash solution rise to the mark on the upper part of the tube, lowering *F* still more if the pressure is not sufficient. Fill the tubes *C* and *D* in the same way, filling the buret with water each time by altering the position of the bottle *F*. With the absorption branch of each U-tube filled with its solution and the buret with water, the apparatus is ready for the tests. Connect the sample bulb with the small U-tube on *E* and permit a little of the gas to escape into the air through the side branch of the three-way cock *G* to expel the air or previous sample from the connections.

Open the cock to *G* to connect with the apparatus and the pinchcock on the water tube, and let the gas displace the water in the buret. Hold the bottle, *F*, so that the level of the water in it will be level with the zero of the buret, disconnect the sample bulb and manipulate the cock *G* so that the levels of the water in the bottle and buret will be the same. Lift the bottle *F* and cautiously open the cock on *B*, and let the gas displace the caustic potash, filling and emptying the U-tube with gas by manipulating the bottle, but at no time letting the water rise above the 100 mark on the buret. The caustic potash solution will absorb the carbon dioxide. As soon as absorption ceases, hold the bottle with the surface of the water in it at the level of that in the buret and note the buret reading, which is the percentage of carbon dioxide (CO_2). Next repeat these manipulations with the residue of the gas using U-tube *C*. Note the total reading of the buret and subtract the first reading from it to obtain the percentage of oxygen (O). Again proceed with the last residue of gas as before, using U-tube *D*. The second buret reading subtracted from the third gives the percentage of carbon monoxide (CO). The final residue usually consists almost entirely of nitrogen (N) and the percentage is obtained by subtracting the third buret reading from 100. The gases may contain very small quantities of hydrogen sulfide and sulfur dioxide; these introduce a slight error in the determinations.

Hydrogen sulfide is tested for with filter paper moistened with lead acetate or subacetate, which turns black in the presence of the gas.

Sulfur dioxide may be detected by shaking a little of the gas in a test tube with iodized starch solution. If this acid is present the blue color is discharged.

After each analysis the residual gas should be expelled from the apparatus and the buret left filled with water. The cocks should be well greased with petrolatum or some preparation for this purpose.

893. Automatic CO_2 Devices. Many modern boiler houses are equipped with automatic devices for analyzing the flue gases. These generally indicate and record at short intervals the percentage of CO_2 present. They are of two general types, direct and indirect, the direct actually analyzing the gas sample by absorbing the CO_2 in caustic as in the Orsat, whereas the indirect depend upon the difference in density of carbon dioxide and air.

All these devices require close attention to keep them in accurate working order. Descriptions and detailed directions can be obtained from the makers.

894. Interpretation of Flue Gas Analyses. The burning of any fuel under boiler conditions requires the use of an excess of air, i.e., more air than the theoretical amount required to supply oxygen to all the combustible. Any attempt to limit the air to within a close approximation of the theoretical will result in smoke, loss of unburned carbon in the form of soot, and incomplete combustion as shown by the presence of carbon monoxide (CO) in the flue gases. The loss under these conditions may be very great as only about one-third the heat value is obtained when carbon is burned to the monoxide as when it is completely burned to carbon dioxide. There should be no carbon monoxide whatever in the flue gases.

On the other hand, too great an excess of air also means an avoidable loss. The amount of air is indicated by the percentage of CO_2 in the flue gases, the relationship varying with the fuel as shown in Fig. 206. It is the heat required to raise this excess air from outside temperatures to that of the stack gases that causes the loss.

Reference to Fig. 26 in Chapter 4 will show that, given a fixed stack temperature, e.g., 500° F., and 200 per cent excess air (indicated by about 7 per cent CO_2 when burning bagasse), the heat lost in the stack gases is about 43 per cent of the total heat in the bagasse fired, whereas 100 per cent excess air (10 per cent CO_2) shows 36 per cent loss of heat, and 50 per cent excess air (13 per cent CO_2) shows 33 per cent loss. With modern boilers and furnaces proper combustion with 50 per cent excess is easily attainable. These figures indicate the large heat losses that may result from improper regulation of the air supply or from leaky boiler settings.

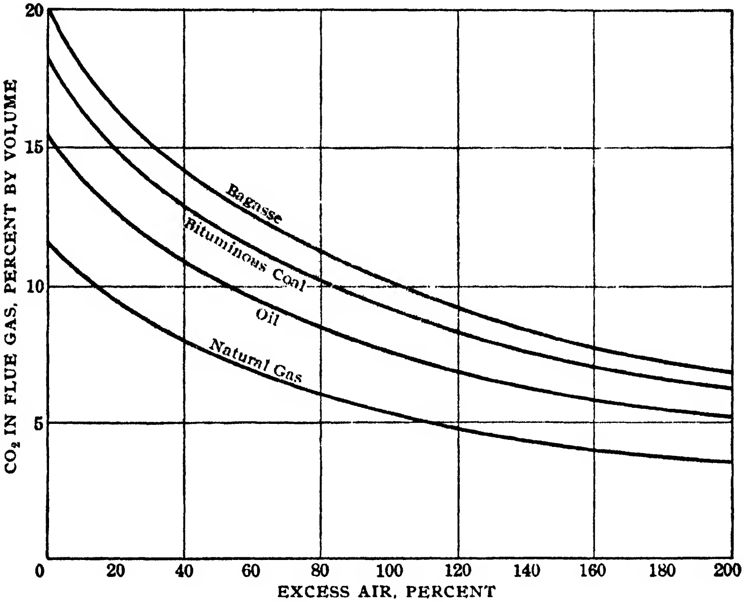

FIG. 206. Excess Air and CO_2 for Various Fuels.

CHAPTER 41

SPECIAL REAGENTS

895. Litmus Solution. Litmus solution and litmus papers no longer play an important part in factory control since pH (see Chapter 23) is now universally used instead of litmus paper to determine the reactions. This solution may be prepared either (*a*) directly from pure azolitmin which may be purchased from the dealers, or (*b, c*) by separating this coloring matter from crude litmus. The crude litmus should not be used without purification of the azolitmin in the preparation of either the solution or the test paper, since the other coloring matters present impair the sensitivity.

a. Dissolve 1 gram of pure azolitmin in 100 ml. of dilute alcohol containing about 20 ml. of 85 per cent alcohol, by weight. Filter the solution and preserve it as indicated in (*b*).

b. Boil 100 grams of commercial litmus with 600 ml. of distilled water and set it aside to cool and deposit the residue. Decant the clear solution and evaporate it on the water bath to about 200 ml. Filter the concentrate and dilute the filtrate to 300 ml. with distilled water and add 100 ml. of diluted sulfuric acid containing 16.2 grams of the pure concentrated acid. Heat the mixture four hours on the water bath with frequent stirring. Collect the precipitate which forms and wash it with cold water, until the washings assume a peculiar fiery red color and on addition of caustic alkali a deep blue and not a violet color. Reject the washings and dissolve out the purified coloring matter with 100 ml. lukewarm 90 per cent alcohol to which a few drops of ammonia have been added. Distil off the alcohol, after filtration, and evaporate the residue to dryness on the water bath. Dissolve the dried residue in 600 ml. of distilled water and neutralize the solution with sodium hydrate. The neutral solution should have a pure violet tint.

Litmus solution decomposes when stored in a stoppered bottle. It should be kept in a salt-mouth bottle, which should be only half filled with the solution, and a loose plug of cotton should be placed in the mouth of the bottle to keep out dust and admit air.

c. Exhaust powdered litmus with successive portions of hot alcohol to remove the coloring matter that accompanies the azolitmin. Digest the residue in cold water acidulated with sulfuric or hydrochloric and reject the solution. Treat the residue with boiling water to extract the azolitmin and then filter the solution. The filtrate should be neutralized and preserved as in (*b*).

896. Litmus Papers. These papers are prepared by soaking filter paper in acid (red), neutral and alkaline (blue) azolitmin solution. The paper should be of the best quality and be thoroughly washed. Well-washed quantitative filter paper is suitable.

The neutral paper is prepared by soaking the paper in the azolitmin solution obtained by one of the methods of the previous paragraph. The sheet of paper should be drawn over a glass rod to remove the surplus solution, and then hung to dry in a room free of laboratory fumes. The sheet should be occasionally turned when first hung to prevent the solution from accumulating at the edges by drainage.

The red paper is prepared as above, but from an azolitmin solution that has been faintly acidulated with sulfuric acid. The acidulation is conveniently and efficiently accomplished as follows. Divide the azolitmin solution into two equal parts and faintly acidulate one and then mix the two. Repeat this proceeding until the mixture is very faintly red. Test the sensitiveness of the solution by soaking a strip of filter paper in it. The paper should be slightly red when dried. Proceed as above in preparing an alkaline solution for the blue paper, using a dilute sodium hydrate solution.

897. **Phenolphthalein Solution.** Dissolve 1 gram of phenolphthalein in 100 ml. of 50 per cent alcohol and neutralize it with acid or alkali as may be necessary.

898. **Phenolphthalein or Dupont Paper.** This paper is made by soaking the very finest quality of filter or glazed paper in an alcoholic solution of phenolphthalein. Dupont regulated the sensitiveness of the paper by adding dilute sulfuric acid to the alcoholic solution and made the sensitiveness correspond to different proportions of lime or other alkalinity of juices. This paper may be used in the control of sulfitation processes.

899. **Methyl Orange.** Dissolve 1 gram of the pure material in distilled water and make to 1 liter.

900. **Iodate Paper.** Dissolve 2 grams of starch in 100 ml. of water with heating and add 0.2 gram of potassium iodate dissolved in 5 ml. of water. Soak filter paper in this solution and dry it.

This paper is used in testing for sulfurous acid, which, even in slight traces, frees the iodine and colors the paper blue through the reaction with the starch.

901. **Lead Subacetate.** Heat to boiling, for about half an hour, 860 grams of neutral lead acetate, 260 grams of litharge, and 2 liters of water. Cool, settle, and decant the clear solution.

This solution may be prepared without heat, provided the mixture is set aside several hours, with frequent shaking. The solution should be diluted with cold, recently boiled, distilled water to 54.3° Brix. (1.25 sp. gr.)

The International Commission for Uniform Methods of Sugar Analysis (1909) prescribes boiling 3 parts normal lead acetate, 1 part lead oxide, and 10 parts of distilled water. The U.S. Bureau of Standards finds that laboratory-made lead subacetate solutions, even though prepared by official methods, vary widely in composition. They therefore recommend[1] that the

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[1] *U.S. Bur. Standards Circ. C 440*, p. 120 (1942).

solution be made by dissolving the reagent known as Horne's dry lead (Sec. 475) which, as made by Horne's formula, has been found to be of quite uniform composition. The solution should be made with recently boiled distilled water to a density of about 1.25 (30° Baumé or 54.3° Brix). Lead subacetate solution stored in stock bottles for daily use should be protected from the carbon dioxide in the air by soda-lime tubes. The burets used for dispensing for routine analyses should be similarly protected.

902. Neutral Lead Acetate. Dissolve neutral lead acetate in distilled water to make a saturated solution, then add sufficient acetic acid to bring the solution to neutrality or faint acidity to litmus paper, after which it should be diluted to 30° Baumé.

903. Aluminum Hydroxide. Alumina Cream. To a saturated solution of common alum in water add ammonia little by little until in slight excess, then enough additional alum to render the solution slightly acid to litmus paper. The sulfates may remain in solution to precipitate the excess of lead, but it is generally preferable to wash the precipitate by decantation with water until it contains only traces of sulfates.

904. Preparation of Pure Sugar. The following method of purifying sugar, for use in testing polariscopes, was adopted by the Fourth International Congress of Applied Chemistry, Paris, 1900, on the recommendation of the committee appointed with a view to unifying the methods of sugar analysis used in various countries. Prepare a hot saturated solution of the purest commercial sugar obtainable, and precipitate the sugar with absolute ethyl alcohol. Spin the precipitated sugar in the laboratory centrifugal and wash it with alcohol. Redissolve and reprecipitate the sugar as before, washing it in the centrifugal with alcohol. The sugar so obtained should be dried between pieces of blotting paper and preserved in a stoppered jar. The moisture in the sugar should be determined and proper allowance made for it when weighing the sample for analysis.

The preparation of pure sucrose is a difficult operation as will be found by reference to the work of Bates and Jackson, pp. 392–396 of *Bureau of Standards Circular C 440.* For most sugar-laboratory work any dry high-grade granulated sugar (preferably cube sugar or a large grain granulated) will be as close to pure sucrose as is obtainable by any ordinary method and this may generally be employed wherever pure sucrose is specified for use.

905. Preparation of Pure Invert Sugar. Dissolve 4.75 grams of pure sucrose in 75 ml. of water, add 10 ml. of dilute hydrochloric acid (24.85° Brix at 20° C. as is used in Clerget methods) and set aside for twenty-four hours at a temperature not lower than 20° C. Neutralize the acid carefully with sodium hydroxide, shaking constantly during the addition of the alkali. Make to 1 liter. Ten milliliters of this solution contains 0.050 gram of invert sugar.

Eynon and Lane recommend that the inverted solution be kept acid without neutralization as the neutralized solutions deteriorate rapidly. The aliquot portions may be neutralized as needed.

906. Heavy Invert Sugar Sirup. For laboratory or other experimental purposes a heavy invert sugar sirup may be desired. The following formula will give a near-white sirup (80° Brix) if temperatures are carefully regulated:

1000 grams best (large grain) granulated
282 ml. distilled water
2.2 ml. dilute HCl made by diluting 4.3 ml. of concentrated HCl to 100 ml.

Mix well and heat to 85° C. for ten minutes. Then raise temperature to 105° C. and hold at that temperature (without boiling) for fifteen minutes. Cool rapidly to prevent discoloration. Do not neutralize acid, which is present in about 1 part HCl to 25,000 parts of sirup. The pH is about 2.8.

907. Fehling's Solution, Soxhlet's Modification. Two solutions are employed which are mixed in equal portions immediately before use. Most of the reducing sugar methods described in this book use this reagent.

a. Copper Sulfate Solution. Dissolve 34.639 grams of copper sulfate ($CuSO_4 \cdot 5H_2O$) in water, dilute to 500 ml. and filter through prepared asbestos.

b. Alkaline Tartrate Solution. Dissolve 173 grams of Rochelle salt (sodium potassium tartrate) and 50 grams of sodium hydroxide in water, dilute to 500 ml., allow to stand for two days, and filter through prepared asbestos. The filtration of both solutions may be omitted if pure chemicals are used.

908. Normal Acid and Alkali Solutions. According to Sutton,[2] "Normal solutions, as a rule, are so prepared that 1 liter shall contain the hydrogen equivalent of the active reagent weighed in grams ($H = 1.008$)." Thus normal sulfuric acid contains 49.040 grams H_2SO_4 per liter; normal hydrochloric acid, 36.465 grams HCl per liter, etc. Half-normal, one-fifth normal, and one-tenth normal (decinormal) solutions are frequently used and may be prepared by diluting the normal solutions. Normal, half-normal, one-fifth normal solutions, etc., are usually indicated as follows: N, $0.5\,N$, $0.2\,N$, $0.1\,N$, etc. These solutions are prepared and checked as described in the following sections.

909. Standard Hydrochloric Acid. The reagent acid usually has a specific gravity of 1.20, approximately. Acid of this specific gravity contains about 40 per cent of hydrochloric acid, hence a little less than 50 grams of it is required to contain the 18.233 grams necessary to form a half-normal solution which is a convenient strength. It is advisable to dilute a somewhat larger quantity of the acid, e.g., 40 ml. to 1000 ml., with distilled water, rather than to attempt to approximate the correct quantity.

To standardize this acid, first prepare pure sodium carbonate as follows. Wash several grams of any good brand of ordinary baking soda several times with cold water by decantation to remove possible soluble chlorides and sulfates; dry in a hot air oven at 100° C. and then heat to dull redness in a

[2] *Volumetric Analysis.*

platinum dish for half an hour (or to constant weight). The heating changes the sodium bicarbonate, $NaHCO_3$ to sodium carbonate, Na_2CO_3. Store the pure Na_2CO_3 in a desiccator in a glass-stoppered bottle. To standardize the acid, weigh accurately about 1.1 grams of the carbonate, dissolve in 100 ml. of distilled water, add two to three drops methyl orange, and titrate to the point where the color changes from yellow to pinkish-orange. One gram of pure $Na_2CO_3 = 37.736$ ml. of $0.5\,N$ acid. Weight of $Na_2CO_3 \times 37.736 =$ milliliters of acid that should have been used if acid is exactly $0.5\,N$. This figure divided into milliliters of acid actually used × 1000 = number of milliliters of the acid that must be made up with water to 1 liter to yield exactly $0.5\,N$ acid. The preliminary titration should show the acid too strong in order that it may be diluted to the correct strength.

Example. Suppose 1.11 grams sodium carbonate takes 39.70 ml. of acid to neutralize. Then,

$$1.11 \times 37.736 = 41.89$$

$$\frac{39.70 \times 1000}{41.89} = 947.7$$

Then 947.7 ml. of the acid must be made to 1 liter to obtain exactly $0.5\,N$ acid.

Instead of using solutions of exact normality many chemists prefer to approximate the desired strength and then use a factor in the calculations. In the above example the factor before dilution would be

$$\frac{1000}{947.7} = 1.0551$$

After the dilution the acid should again be titrated against sodium carbonate in the same manner as above to check the correctness of the dilution. The acid so checked will serve as a convenient basis for the preparation of other standard acids and alkalies.

$$\begin{aligned}\text{1 ml. normal hydrochloric acid} &= 0.036465 \text{ gram HCl}\\ &= 0.035457 \text{ gram Cl}\\ &= 0.028035 \text{ gram CaO}\end{aligned}$$

910. Standard Oxalic Acid. This is the simplest of the normal solutions to prepare, and when strictly pure oxalic acid can be obtained it may be used in checking the preparation of all the standard alkali and acid solutions. Oxalic acid of tested purity as obtained from the dealers will serve the purpose.

Reject all crystals that show indications of efflorescence. Dissolve 63.035 grams of this acid in distilled water and dilute to 1000 ml. to prepare the normal solution, or, preferably, dry the powdered acid at 100° C. to constant weight and use 45.018 grams in preparing the normal solution. It is advisable to employ weaker solutions than the normal, usually the one-tenth normal acid. This should be prepared from the normal solution as required, since the latter keeps well, provided it is not exposed to direct sunlight.

1 ml. normal oxalic acid = 0.06304 gram $H_2C_2O_4 \cdot 2H_2O$

911. Standard Sulfuric Acid. Add approximately 28 ml. of concentrated sulfuric acid to distilled water, cool the solution, and dilute to 1000 ml. Standardize by titration with normal alkali or gravimetrically by precipitation with barium chloride. Many chemists use the gravimetric method as the starting point in the preparation of standard acids and alkalies.

1 ml. normal sulfuric acid = 0.049040 gram H_2SO_4
= 0.028035 gram CaO
= 0.051815 gram SrO
= 0.076685 gram BaO

912. Standard Sulfuric Acid for the Control of the Carbonation. Add approximately 21 ml. of concentrated sulfuric acid to distilled water, cool the solution, and dilute to 1000 ml. Titrate this solution with a normal soda or potash solution, using phenolphthalein as an indicator. Dilute the acid so that 14 ml. of it will be required to neutralize 10 ml. of the normal alkali.

1 ml. this standard acid = 0.035 gram H_2SO_4
= 0.02 gram CaO

It is usual to add the phenolphthalein to this solution before dilution to 1000 ml.

913. Standard Alkali Solutions. Ammonium hydroxide (NH_4OH), caustic soda (NaOH), and caustic potash (KOH) are used in preparing the alkali solutions. The normal soda or potash solutions are used, but the ammonia should be weaker, preferably decinormal.

Dissolve 42 grams of chemically pure caustic soda in water, in preparing the normal reagent, cool the solution, dilute to 1000 ml. and standardize it by titration against a normal acid. In preparing the potash solution, use 58 grams of chemically pure caustic potash. Standard ammonia is prepared by diluting the reagent to approximately the required strength, and standardizing it by titration with decinormal or half-normal acid, as may be required, using cochineal as an indicator.

1 ml. normal caustic soda solution = 0.04001 gram NaOH
= 0.03100 gram Na_2O
1 ml. normal caustic potash solution = 0.05610 gram KOH
= 0.04710 gram K_2O
1 ml. half-normal ammonia solution = 0.00852 gram NH_3
= 0.01752 gram NH_4OH
1 ml. decinormal ammonia solution = 0.00170 gram NH_3
= 0.00351 gram NH_4OH

Phenolphthalein cannot be used as an indicator with ammonia.

914. Decinormal Potassium Permanganate. Dissolve 3.16 grams of chemically pure, dry potassium permanganate ($KMnO_4$) in distilled water, and dilute to 1000 ml. This solution is conveniently checked by titration with decinormal oxalic acid. To 10 ml. of decinormal oxalic acid add several volumes of water and a few cubic centimeters of dilute sulfuric acid. Warm the solution to approximately 60° C. and add the permanganate solution little by little. Discontinue the addition of the permanganate as soon as the solution acquires a faint pink or rose color. The temperature of the solution must be maintained at approximately 60° C., and a little time must be allowed for the reaction.

Potassium permanganate solution should be preserved in a tightly stoppered bottle, and should be checked from time to time. The formation of a precipitate indicates a change in the solution. It is simpler to determine a factor from time to time, rather than attempt to maintain the solution strictly decinormal.

$$\text{1 ml. decinormal potassium permanganate}\begin{cases} = 0.00316 \text{ gram } KMnO_4 \\ = 0.00636 \text{ gram Cu} \end{cases}$$

REFERENCE TABLES

TABLE 1

INTERNATIONAL ATOMIC WEIGHTS

1944

Reprinted from the *Journal of the American Chemical Society*

	Symbol	*Atomic Number*	*Atomic Weight*		*Symbol*	*Atomic Number*	*Atomic Weight*
Aluminum...	Al	13	26.97	Molybdenum..	Mo	42	95.95
Antimony....	Sb	51	121.76	Neodymium..	Nd	60	144.27
Argon.......	A	18	39.944	Neon........	Ne	10	20.183
Arsenic......	As	33	74.91	Nickel.......	Ni	28	58.69
Barium......	Ba	56	137.36	Nitrogen....	N	7	14.008
Beryllium....	Be	4	9.02	Osmium......	Os	76	190.2
Bismuth.....	Bi	83	209.00	Oxygen......	O	8	16.0000
Boron.......	B	5	10.82	Palladium....	Pd	46	106.7
Bromine.....	Br	35	79.916	Phosphorus...	P	15	30.98
Cadmium....	Cd	48	112.41	Platinum.....	Pt	78	195.23
Calcium.....	Ca	20	40.08	Potassium....	K	19	39.096
Carbon......	C	6	12.010	Praseodymium	Pr	59	140.92
Cerium......	Ce	58	140.13	Protactinium..	Pa	91	231
Cesium......	Cs	55	132.91	Radium......	Ra	88	226.05
Chlorine.....	Cl	17	35.457	Radon.......	Rn	86	222
Chromium...	Cr	24	52.01	Rhenium.....	Re	75	186.31
Cobalt......	Co	27	58.94	Rhodium.....	Rh	45	102.91
Columbium..	Cb	41	92.91	Rubidium....	Rb	37	85.48
Copper......	Cu	29	63.57	Ruthenium...	Ru	44	101.7
Dysprosium..	Dy	66	162.46	Samarium....	Sm	62	150.43
Erbium......	Er	68	167.2	Scandium....	Sc	21	45.10
Europium....	Eu	63	152.0	Selenium.....	Se	34	78.96
Fluorine.....	F	9	19.00	Silicon.......	Si	14	28.06
Gadolinium..	Gd	64	156.9	Silver........	Ag	47	107.880
Gallium.....	Ga	31	69.72	Sodium......	Na	11	22.997
Germanium..	Ge	32	72.60	Strontium....	Sr	38	87.63
Gold........	Au	79	197.2	Sulfur........	S	16	32.06
Hafnium.....	Hf	72	178.6	Tantalum....	Ta	73	180.88
Helium......	He	2	4.003	Tellurium....	Te	52	127.61
Holmium....	Ho	67	164.94	Terbium.....	Tb	65	159.2
Hydrogen....	H	1	1.0080	Thallium.....	Tl	81	204.39
Indium......	In	49	114.76	Thorium.....	Th	90	232.12
Iodine.......	I	53	126.92	Thulium.....	Tm	69	169.4
Iridium......	Ir	77	193.1	Tin..........	Sn	50	118.70
Iron.........	Fe	26	55.85	Titanium.....	Ti	22	47.90
Krypton.....	Kr	36	83.7	Tungsten.....	W	74	183.92
Lanthanum..	La	57	138.92	Uranium.....	U	92	238.07
Lead........	Pb	82	207.21	Vanadium....	V	23	50.95
Lithium.....	Li	3	6.940	Xenon.......	Xe	54	131.3
Lutecium....	Lu	71	174.99	Ytterbium....	Yb	70	173.04
Magnesium..	Mg	12	24.32	Yttrium......	Y	39	88.92
Manganese...	Mn	25	54.93	Zinc.........	Zn	30	65.38
Mercury.....	Hg	80	200.61	Zirconium....	Zr	40	91.22

TABLE 2

EQUIVALENTS OF THE UNITED STATES CUSTOMARY AND METRIC WEIGHTS AND MEASURES *

Miscellaneous Equivalents

1 Avoirdupois pound = 453.5924277 grams
1 Meter = 39.37 inches (U. S. law of 1866)
1 U. S. mile = 1.60935 kilometers
1 Kilometer = 0.62137 U. S. mile
1 Acre = 0.4047 hectare
1 Hectare = 2.471 acres
1 U. S. liquid ounce = 29.574 cubic centimeters
1 U. S. apothecaries' dram = 3.6967 cubic centimeters
1 U. S. dry quart = 1.1012 liters
1 U. S. bushel = 0.35239 hectoliter
1 Long ton (2240 av. lbs.) = 1016.05 kilograms
1 Short ton (2000 av. lbs.) = 907.18 kilograms
1 Metric ton = 2204.62 avoirdupois pounds

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* From "Tables of Equivalents," 4th Ed., U. S. Bureau of Standards.

TABLE 2 (Continued)

TABLES OF EQUIVALENTS

LENGTHS						AREAS			
Inches	Millimeters	Inches	Centimeters	Feet	Meters	Square Inches	Square Millimeters	Square Feet	Square Meters
0.03937	1	0 3937	1	1	0.304801	0.00155	1	1	0.09290
0.07874	2	0.7874	2	2	0 609601	0 00310	2	2	0.18581
0.11811	3	1	2.54001	3	0.914402	0 00465	3	3	0.27871
0.15748	4	1.1811	3	3 28083	1	0 00620	4	4	0.37161
0.19685	5	1.5748	4	4	1 219202	0.00775	5	5	0.46452
0.23622	6	1.9685	5	5	1.524003	0 00930	6	6	0 55742
0.27559	7	2	5.08001	6	1.828804	0 01085	7	7	0 65032
0.31496	8	2.3622	6	6.56167	2	0.01240	8	8	0.74323
0.35433	9	2.7559	7	7	2 133604	0.01395	9	9	0.83613
1	25.4001	3	7.62002	8	2 438405	1	645 16	10.764	1
2	50.8001	3.1496	8	9	2.743205	2	1,290.33	21.528	2
3	76.2002	3.5433	9	9.84250	3	3	1,935 49	32 292	3
4	101.6002	4	10.16002	13.12333	4	4	2,580.65	43.055	4
5	127.0003	5	12.70003	16.40417	5	5	3,225.81	53.819	5
6	152.4003	6	15 24003	19.68500	6	6	3,870.98	64.583	6
7	177.8004	7	17.78004	22 96583	7	7	4,516.14	75.347	7
8	203.2004	8	20.32004	26.24667	8	8	5,161.30	86.111	8
9	228.6005	9	22.86005	29.52750	9	9	5,806.46	96.875	9

TABLE 2 (*Continued*)

TABLES OF EQUIVALENTS

Volumes				Capacities		Masses			
Cubic Inches	Cubic Cent.	Cubic Feet	Cubic Meters	U. S. Liquid Gallons	Liters	Grains	Grams	Avoirdupois Pounds	Kilograms
0.0610 =	1	1	= 0.02832	0.26417 =	1	1	= 0.06480	1	= 0.45359
0.1220 =	2	2	= 0.05663	0.52834 =	2	2	= 0.12960	2	= 0.90718
0.1831 =	3	3	= 0.08495	0.79251 =	3	3	= 0.19440	2.20462 =	1
0.2441 =	4	4	= 0.11327	1	= 3.78543	4	= 0.25920	3	= 1.36078
0.3051 =	5	5	= 0.14159	1.05668 =	4	5	= 0.32399	4	= 1.81437
0.3661 =	6	6	= 0.16990	1.32085 =	5	6	= 0.38879	4.40924 =	2
0.4272 =	7	7	= 0.19822	1.58502 =	6	7	= 0.45359	5	= 2.26796
0.4882 =	8	8	= 0.22654	1.84919 =	7	8	= 0.51839	6	= 2.72155
0.5492 =	9	9	= 0.25485	2	= 7.57087	9	= 0.58319	6.61387 =	3
1	= 16.3872	35.314 =	1	2.11336 =	8	15.4324 =	1	7	= 3.17515
2	= 32.7743	70.629 =	2	2.37753 =	9	30.8647 =	2	8	= 3.62874
3	= 49.1615	105.943 =	3	3	= 11.35630	46.2971 =	3	8.81849 =	4
4	= 65.5486	141.258 =	4	4	= 15.14174	61.7294 =	4	9	= 4.08233
5	= 81.9358	176.572 =	5	5	= 18.92717	77.1618 =	5	11.02311 =	5
6	= 98.3230	211.887 =	6	6	= 22.71261	92.5941 =	6	13.22773 =	6
7	= 114.7101	247.201 =	7	7	= 26.49804	108.0265 =	7	15.43236 =	7
8	= 131.0973	282.516 =	8	8	= 30.28348	123 4589 =	8	17.63698 =	8
9	= 147.4845	317.830 =	9	9	= 34.06891	138.8912 =	9	19 84160 =	9

TABLE 3

Mensuration

Parallelogram. Area of any parallelogram = Base × Altitude; Area of rhombus = Product of two adjacent sides × Sine of angle included between them.

Trapezium. Area = (Diagonal × Sum of perpendicular let fall on it from opposite angles) ÷ 2 = Area of the two triangles into which it may be divided.

Trapezoid. Area = Half the sum of the two parallel sides × Perpendicular distance between them.

Any Quadrilateral. Divide the quadrilateral into two triangles and find the sum of the areas of these, or Area = Half the product of the two diagonals × Sine of the angle at their intersection.

Triangle. Area = Base × Half the altitude = Half the product of two sides × Sine of the included angle = Half the sum of the three sides minus each side severally; multiply this half sum and the three remainders together and extract the square root of the product. Area of an equilateral triangle = One-fourth the square of one of its sides × 0.433013.

Hypothenuse and one side of a right-angle triangle being given to find the other side: Required side = $\sqrt{\text{Hypothenuse}^2 - \text{Given side}^2}$; if the two sides are equal, Side = Hypothenuse × 0.7071.

Area given to find base: Base = 2 × Area ÷ Perpendicular height.

Area given to find height: Height = 2 × Area ÷ Base.

Two sides and base given to find perpendicular height, both angles at base are acute: Base/Sum of the sides = Difference of the sides/Difference of the divisions of the base made by drawing the perpendicular. Half this difference added to or subtracted from half the base will give the divisions of it.

Polygon. Area of irregular polygon: draw diagonals dividing the polygon into triangles and find the sum of the areas of these.

Area of regular polygon: Area = (Length of a side × Perpendicular distance of side to center × Number of sides) ÷ 2 = Half the perimeter × Perpendicular distance of side to center.

Perpendicular to center = Half of one side × Cotangent of the angle subtended by half the side.

Irregular Figure (Laboratory method). Draw the figure on paper of uniform thickness and cut it out; compare the weight of this piece with that of a sheet of the paper of known area.

Circle. Ratio of circumference to diameter = 3.1415929 (usually taken as 3.1416). This number is usually represented by the Greek letter *pi*, π.

Circumference = Diameter × π.

Length of an arc: 360/Number of degrees of the arc = Circumference of the circle/Length of the arc, or Diameter of the circle × Number of degrees in the arc × 0.0087266 = Length of the arc.

Area of a circle = Square of the radius × π = Square of diameter × 0.7854.

Areas of circles are to each other as the squares of their diameters.

TABLE 3 (*Continued*)

MENSURATION

Ellipse. Area = Product of the semi-axes × 3.1416 = Product of the axes × 0.7854.

Prism. Area = (Perimeter of base × Altitude) + Areas of the two ends.

Volume = Area of base × Altitude.

Pyramid. Convex surface of a regular pyramid = Perimeter of base × Half the slant height.

Volume = Area of base × One-third of altitude.

Area of a frustum of a regular pyramid = Half the slant height × Sum of the perimeters of the two bases (= Convex surface) + Area of the two bases.

Volume of a frustum of a regular pyramid = Sum of areas of the two bases and a mean proportional between them × One-third of the altitude. (Mean proportional between two numbers = Square root of their product.)

Rectangular Prismoid (a solid bounded by six planes, of which two bases are rectangles, having corresponding sides parallel, and the four upright sides are trapezoids). Volume = (Sum of the areas of the bases + Four times the area of a parallel section equidistant from the bases) × One-sixth of the altitude.

Cylinder. Area of convex surface = Circumference of base × Altitude. Convex surface + Two end areas = Total area.

Volume of a cylinder = Area of base × Altitude.

Cone. Total area = Circumference of base × Half slant height (= Convex surface) + Area of the base.

Volume of a cone = Area of base × One-third of altitude.

Frustum of a cone: Area = (Half the side × Sum of the circumference of the two bases) + Area of the two bases.

Volume of frustum of a cone = (Area of two bases + Mean proportional between them × One-third of the altitude). (See Volume of a frustum of a pyramid relative to a mean proportional.)

Parabola. Area = Base × ⅔ altitude.

Sphere. Surface = Diameter × Circumference of its great circle = Square of diameter × 3.1416 = Convex surface of its circumscribing cylinder.

Surfaces of spheres are to each other as the squares of their diameters.

Volume of a sphere = Surface × One-third of the radius = Cube of the diameter × 0.5236.

Volumes of spheres are to each other as the cubes of their diameters.

Cask. Volume of a cask in U.S. gallons = (39 × Square of bung diameter + 25 × Square of head diameter + 26 × Product of the diameters) × Length ÷ 26,470.

Molasses Tanks, Crystallizers. See Table 6.

TABLE 4

Circumferences and Areas of Circles

Diameter, Inches	Circumference, Inches	Area, Square Inches
⅛	.39270	.012272
¼	.78540	.049087
⅜	1.1781	.110447
½	1.5708	.19635
⅝	1.9635	.306796
¾	2.3562	.441787
⅞	2.7489	.601322
1	3.1416	.7854
⅛	3.5343	.99402
¼	3.9270	1.2272
⅜	4.3197	1.4849
½	4 7124	1.7671
⅝	5.1051	2.0739
¾	5.4978	2.4053
⅞	5.8905	2.7612
2	6.2832	3.1416
⅛	6.6759	3.5466
¼	7.0686	3.9761
⅜	7.4613	4.4301
½	7.8540	4.9087
⅝	8.2467	5 4119
¾	8.6394	5.9396
⅞	9.0321	6.4918
3	9.4248	7.0686
⅛	9.8175	7.6699
¼	10.210	8.2958
⅜	10.603	8.9462
½	10.996	9.6211
⅝	11.388	10.3206
¾	11.781	11.0447
⅞	12.174	11.7933
4	12.566	12.5664
⅛	12.959	13.3641
¼	13.352	14.1863
⅜	13.744	15.033
½	14.137	15.9043
⅝	14.530	16.8002
¾	14.923	17.7206
⅞	15.315	18.6655
5	15.708	19.635
⅛	16.101	20.629
¼	16.493	21.6476
⅜	16.886	22.6907
½	17.279	23.7583
⅝	17.671	24.8505
¾	18.064	25.9673
⅞	18.457	27.1086
6	18.850	28.2744
⅛	19.242	29.4648
¼	19.635	30.6797
⅜	20.028	31.9191
½	20.420	33.1831
⅝	20.813	34.4717
¾	21.206	35.7848
⅞	21.598	37.1224
7	21.991	38.4846
⅛	22.384	39.8713
¼	22.776	41.2826
⅜	23.169	42.7184
½	23.562	44.1787
⅝	23.955	45.6636
¾	24.347	47.1731
⅞	24.740	48.7071
8	25.133	50.2656
⅛	25.525	51.8487
¼	25 918	53.4563
⅜	26.311	55.0884
½	26.704	56.7451
⅝	27 096	58.4264
¾	27.489	60.1322
⅞	27.882	61.8625
9	28.274	63.6174
⅛	28.667	65.3968
¼	29.060	67.2008
⅜	29.452	69.0293
½	29.845	70.8823
⅝	30.238	72.7599
¾	30.631	74.6621
⅞	31.023	76.5888
10	31.416	78.54
⅛	31.809	80.5158
¼	32.201	82.5161
⅜	32.594	84.5409
½	32.987	86.5903
⅝	33.379	88.6643
¾	33.772	90.7628
⅞	34.165	92.8858
11	34.558	95.0334
⅛	34.950	97.2055
¼	35.343	99.4022
⅜	35.736	101.6234
½	36.128	103.8691
⅝	36.521	106.1394
¾	36.914	108.4343
⅞	37.306	110.7537
12	37.699	113.098
⅛	38.092	115.466
¼	38.485	117.859
⅜	38.777	120.277
½	39.270	122.719
⅝	39.663	125.185
¾	40.055	127.677
⅞	40.448	130.192
13	40.841	132.733
⅛	41.233	135.297
¼	41.626	137.887
⅜	42.019	140.501
½	42.412	143.139
⅝	42.804	145.802
¾	43.197	148.49
⅞	43.590	151.202
14	43.982	153.938
⅛	44.375	156.7
¼	44.768	159.485
⅜	45.160	162.296
½	45.553	165.13
⅝	45.946	167.99
¾	46.338	170.874
⅞	46.731	173.782
15	47.124	176.715
⅛	47.517	179.673
¼	47.909	182.655
⅜	48.302	185.661
½	48.695	188.692
⅝	49.087	191.748
¾	49.480	194.828
⅞	49.873	197.933
16	50.265	201.062
⅛	50.658	204.216
¼	51.051	207.395
⅜	51.444	210.598
½	51.836	213.825
⅝	52.229	217.077
¾	52.622	220.354
⅞	53.014	223.655
17	53.407	226.981
⅛	53.800	230.331
¼	54.192	233.706
⅜	54.585	237.105
½	54.978	240.529
⅝	55.371	243.977
¾	55.763	247.45
⅞	56.156	250.948
18	56.549	254.47
⅛	56.941	258.016
¼	57.334	261.587
⅜	57.727	265.183
½	58.119	268.803
⅝	58.512	272.448
¾	58.905	276.117
⅞	59.298	279.811
19	59.690	283.529
¼	60.476	291.04
½	61.261	298.65

TABLE 4 (*Continued*)

CIRCUMFERENCES AND AREAS OF CIRCLES

Diameter, Inches	Circumference, Inches	Area, Square Inches	Diameter, Inches	Circumference, Inches	Area, Square Inches	Diameter, Inches	Circumference, Inches	Area, Square Inches
20	62.832	314.16	**40**	125.664	1256.6	**60**	188.496	2827.4
½	64.403	330.06	½	127.235	1288.2	**61**	191.637	2922.5
21	65.973	346.36	**41**	128.805	1320.3	**62**	194.779	3019.1
½	67.544	363.05	½	130.376	1352.7	**63**	197.920	3117.2
22	69.115	380.13	**42**	131.947	1385.4	**64**	201.062	3217.0
½	70.686	397.61	½	133.518	1418.6	**65**	204.204	3318.3
23	72.257	415.48	**43**	135.088	1452.2	**66**	207.345	3421.2
½	73.827	433.74	½	136.659	1486.2	**67**	210.487	3525.7
24	75.398	452.39	**44**	138.230	1520.5	**68**	213.628	3631.7
½	76.969	471.44	½	139.801	1555.3	**69**	216.770	3739.3
25	78.540	490.87	**45**	141.372	1590.4	**70**	219.911	3848.5
½	80.111	510.71	½	142.942	1626.0	**71**	223.053	3959.2
26	81.681	530.93	**46**	144.513	1661.9	**72**	226.195	4071.5
½	83.252	551.55	½	146.084	1698.2	**73**	229.336	4185.4
27	84.823	572.56	**47**	147.655	1734.9	**74**	232.478	4300.8
½	86.394	593.96	½	149.226	1772.1	**75**	235.619	4417.9
28	87.965	615.75	**48**	150.796	1809.6	**76**	238.761	4536.5
½	89.535	637.94	½	152.367	1847.5	**77**	241.903	4656.6
29	91.106	660.52	**49**	153.938	1885.7	**78**	245.044	4778.4
½	92.677	683.49	½	155.509	1924.4	**79**	248.186	4901.7
30	94.248	706.86	**50**	157.080	1963.5	**80**	251.327	5026.5
½	95.819	730.62	½	158.650	2003.0	**81**	254.469	5153.0
31	97.389	754.77	**51**	160.221	2042.8	**82**	257.611	5281.0
½	98.960	779.31	½	161.792	2083.1	**83**	260.752	5410.6
32	100.531	804.25	**52**	163.363	2123.7	**84**	263.894	5541.8
½	102.102	829.58	½	164.934	2164.8	**85**	267.035	5674.5
33	103.673	855.30	**53**	166.504	2206.2	**86**	270.177	5808.8
½	105.243	881.41	½	168.075	2248.0	**87**	273.319	5944.7
34	106.814	907.92	**54**	169.646	2290.2	**88**	276.460	6082.1
½	108.385	934.82	½	171.217	2332.8	**89**	279.602	6221.1
35	109.956	962.11	**55**	172.788	2375.8	**90**	282.743	6361.7
½	111.527	989.80	½	174.358	2419.2	**91**	285.885	6503.9
36	113.097	1017.9	**56**	175.929	2463.0	**92**	289.027	6647.6
½	114.668	1046.3	½	177.500	2507.2	**93**	292.168	6792.9
37	116.239	1075.2	**57**	179.071	2551.8	**94**	295.310	6939.8
½	117.810	1104.5	½	180.642	2596.7	**95**	298.451	7088.2
38	119.381	1134.1	**58**	182.212	2642.1	**96**	301.593	7238.2
½	120.951	1164.2	½	183.783	2687.8	**97**	304.734	7389.8
39	122.522	1194.6	**59**	185.354	2734.0	**98**	307.876	7543.0
½	124.093	1225.4	½	186.925	2780.5	**99**	311.018	7697.7
						100	314.159	7854.0

TABLE 5

TEMPERATURE CONVERSION TABLES (ALBERT SAUVEUR)

(Courtesy of *Chem. Met. Eng.*)

0 to 100						**100 to 1000**					
C.		F.	C.		F.	C.		F.	C.		F.
−17.8	**0**	32	10.0	**50**	122.0	38	**100**	212	260	**500**	932
−17.2	**1**	33.8	10.6	**51**	123.8	43	**110**	230	266	**510**	950
−16.7	**2**	35.6	11.1	**52**	125.6	49	**120**	248	271	**520**	968
−16.1	**3**	37.4	11.7	**53**	127.4	54	**130**	266	277	**530**	986
−15.6	**4**	39.2	12.2	**54**	129.2	60	**140**	284	282	**540**	1004
−15.0	**5**	41.0	12.8	**55**	131.0	66	**150**	302	288	**550**	1022
−14.4	**6**	42.8	13.3	**56**	132.8	71	**160**	320	293	**560**	1040
−13.9	**7**	44.6	13.9	**57**	134.6	77	**170**	338	299	**570**	1058
−13.3	**8**	46.4	14.4	**58**	136.4	82	**180**	356	304	**580**	1076
−12.8	**9**	48.2	15.0	**59**	138.2	88	**190**	374	310	**590**	1094
−12.2	**10**	50.0	15.6	**60**	140.0	93	**200**	392	316	**600**	1112
−11.7	**11**	51.8	16.1	**61**	141.8	99	**210**	410	321	**610**	1130
−11.1	**12**	53.6	16.7	**62**	143.6	100	**212**	413	327	**620**	1148
−10.6	**13**	55.4	17.2	**63**	145.4	104	**220**	428	332	**630**	1166
−10.0	**14**	57.2	17.8	**64**	147.2	110	**230**	446	338	**640**	1184
− 9.44	**15**	59.0	18.3	**65**	149.0	116	**240**	464	343	**650**	1202
− 8.89	**16**	60.8	18.9	**66**	150.8	121	**250**	482	349	**660**	1220
− 8.33	**17**	62.6	19.4	**67**	152.6	127	**260**	500	354	**670**	1238
− 7.78	**18**	64.4	20.0	**68**	154.4	132	**270**	518	360	**680**	1256
− 7.22	**19**	66.2	20.6	**69**	156.2	138	**280**	536	366	**690**	1274
− 6.67	**20**	68.0	21.1	**70**	158.0	143	**290**	554	371	**700**	1292
− 6.11	**21**	69.8	21.7	**71**	159.8	149	**300**	572	377	**710**	1310
− 5.56	**22**	71.6	22.2	**72**	161.6	154	**310**	590	382	**720**	1328
− 5.00	**23**	73.4	22.8	**73**	163.4	160	**320**	608	388	**730**	1346
− 4.44	**24**	75.2	23.3	**74**	165.2	166	**330**	626	393	**740**	1364
− 3.89	**25**	77.0	23.9	**75**	167.0	171	**340**	644	399	**750**	1382
− 3.33	**26**	78.8	24.4	**76**	168.8	177	**350**	662	404	**760**	1400
− 2.78	**27**	80.6	25.0	**77**	170.6	182	**360**	680	410	**770**	1418
− 2.22	**28**	82.4	25.6	**78**	172.4	188	**370**	698	416	**780**	1436
− 1.67	**29**	84.2	26.1	**79**	174.2	193	**380**	716	421	**790**	1454
− 1.11	**30**	86.0	26.7	**80**	176.0	199	**390**	734	427	**800**	1472
− 0.56	**31**	87.8	27.2	**81**	177.8	204	**400**	752	432	**810**	1490
0	**32**	89.6	27.8	**82**	179.6	210	**410**	770	438	**820**	1508
0.56	**33**	91.4	28.3	**83**	181.4	216	**420**	788	443	**830**	1526
1.11	**34**	93.2	28.9	**84**	183.2	221	**430**	806	449	**840**	1544
1.67	**35**	95.0	29.4	**85**	185.0	227	**440**	824	454	**850**	1562
2.22	**36**	96.8	30.0	**86**	186.8	232	**450**	842	460	**860**	1580
2.78	**37**	98.6	30.6	**87**	188.6	238	**460**	860	466	**870**	1598
3.33	**38**	100.4	31.1	**88**	190.4	243	**470**	878	471	**880**	1616
3.89	**39**	102.2	31.7	**89**	192.2	249	**480**	896	477	**890**	1634
4.44	**40**	104.0	32.2	**90**	194.0	254	**490**	914	482	**900**	1652
5.00	**41**	105.8	32.8	**91**	195.8				488	**910**	1670
5.56	**42**	107.6	33.3	**92**	197.6				493	**920**	1688
6.11	**43**	109.4	33.9	**93**	199.4				499	**930**	1706
6.67	**44**	111.2	34.4	**94**	201.2				504	**940**	1724
7.22	**45**	113.0	35.0	**95**	203.0				510	**950**	1742
7.78	**46**	114.8	35.6	**96**	204.8				516	**960**	1760
8.33	**47**	116.6	36.1	**97**	206.6				521	**970**	1778
8.89	**48**	118.4	36.7	**98**	208.4				527	**980**	1796
9.44	**49**	120.2	37.2	**99**	210.2				532	**990**	1814
			37.8	**100**	212.0				538	**1000**	1832

NOTE.—The numbers in bold face type refer to the temperature either in degrees Centigrade or Fahrenheit which it is desired to convert into the other scale. If converting from degrees Fahrenheit to degrees Centigrade the equivalent temperature will be found in the left column, while if converting from degrees Centigrade to degrees Fahrenheit, the answer will be found in the column on the right.

TABLE 6

FOR THE CALCULATION OF THE WANTAGE (EMPTY SPACE) IN HORIZONTAL CYLINDRICAL TANKS (TANK CARS, CRYSTALLIZERS, ETC.), IN U.S. GALLONS AND CUBIC FEET

(Let W = wantage; D = diameter of tank; I = depth of empty space ("inches out"); L = mean length of tank; F = area of segment, $W = (D^2 \times F \times L) \div 231$, wantage in U. S. gallons, or dividing by 1728 gives wantage in cubic feet. All dimensions are in inches.) See page 596.

$I \div D$	F	$I \div D$	F	$I \div D$	F	$I \div D$	F	$I \div D$	F
.001	.00004	.051	.01512	.101	.04148	.151	.07459	.201	.11262
.002	.00012	.052	.01556	.102	.04208	.152	.07531	.202	.11343
.003	.00022	.053	.01601	.103	.04269	.153	.07603	.203	.11423
.004	.00034	.054	.01646	.104	.04310	.154	.07675	.204	.11503
.005	.00047	.055	.01691	.105	.04391	.155	.07747	.205	.11584
.006	.00062	.056	.01737	.106	.04452	.156	.07820	.206	.11665
.007	.00078	.057	.01783	.107	.04514	.157	.07892	.207	.11746
.008	.00095	.058	.01830	.108	.04575	.158	.07965	.208	.11827
.009	.00113	.059	.01877	.109	.04638	.159	.08038	.209	.11908
.010	.00133	.060	.01924	.110	.04700	.160	.08111	.210	.11990
.011	.00153	.061	.01972	.111	.04763	.161	.08185	.211	.12071
.012	.00175	.062	.02020	.112	.04826	.162	.08258	.212	.12153
.013	.00197	.063	.02068	.113	.04889	.163	.08332	.213	.12235
.014	.00220	.064	.02117	.114	.04953	.164	.08406	.214	.12317
.015	.00244	.065	.02165	.115	.05016	.165	.08480	.215	.12394
.016	.00268	.066	.02215	.116	.05080	.166	.08554	.216	.12481
.017	.00294	.067	.02265	.117	.05145	.167	.08629	.217	.12563
.018	.00320	.068	.02315	.118	.05209	.168	.08704	.218	.12646
.019	.00347	.069	.02336	.119	.05274	.169	.08779	.219	.12728
.020	.00375	.070	.02417	.120	.05338	.170	.08853	.220	.12811
.021	.00403	.071	.02468	.121	.05404	.171	.08929	.221	.12894
.022	.00432	.072	.02519	.122	.05469	.172	.09004	.222	.12977
.023	.00462	.073	.02571	.123	.05534	.173	.09080	.223	.13060
.024	.00492	.074	.02624	.124	.05600	.174	.09155	.224	.13144
.025	.00523	.075	.02676	.125	.05666	.175	.09231	.225	.13227
.026	.00555	.076	.02729	.126	.05733	.176	.09307	.226	.13311
.027	.00587	.077	.02782	.127	.05799	.177	.09384	.227	.13394
.028	.00619	.078	.02835	.128	.05866	.178	.09460	.228	.13478
.029	.00653	.079	.02889	.129	.05933	.179	.09537	.229	.13562
.030	.00686	.080	.02943	.130	.06000	.180	.09613	.230	.13646
.031	.00721	.081	.02997	.131	.06067	.181	.09690	.231	.13731
.032	.00756	.082	.03052	.132	.06135	.182	.09767	.232	.13815
.033	.00791	.083	.03107	.133	.06203	.183	.09845	.233	.13900
.034	.00827	.084	.03162	.134	.06271	.184	.09922	.234	.13984
.035	.00864	.085	.03218	.135	.06339	.185	.10000	.235	.14069
.036	.00901	.086	.03274	.136	.06407	.186	.10077	.236	.14154
.037	.00938	.087	.03330	.137	.06476	.187	.10155	.237	.14239
.038	.00976	.088	.03387	.138	.06545	.188	.10233	.238	.14324
.039	.01015	.089	.03444	.139	.06614	.189	.10312	.239	.14409
.040	.01054	.090	.03501	.140	.06683	.190	.10390	.240	.14494
.041	.01093	.091	.03558	.141	.06753	.191	.10468	.241	.14580
.042	.01133	.092	.03616	.142	.06822	.192	.10547	.242	.14665
.043	.01173	.093	.03674	.143	.06892	.193	.10626	.243	.14751
.044	.01214	.094	.03732	.144	.06962	.194	.10705	.244	.14837
.045	.01255	.095	.03790	.145	.07033	.195	.10784	.245	.14923
.046	.01297	.096	.03849	.146	.07103	.196	.10864	.246	.15009
.047	.01339	.097	.03908	.147	.07174	.197	.10943	.247	.15095
.048	01382	.098	.03968	.148	.07245	.198	.11023	.248	.15182
.049	.01425	.099	.04027	.149	.07316	.199	.11102	.249	.15268
.050	.01468	.100	.04087	.150	.07387	.200	.11182	.250	.15356

TABLE 6 (*Continued*)

CALCULATION OF WANTAGE IN CYLINDRICAL TANKS

$I \div D$	F	$I \div D$	F	$I \div D$	F	$I \div D$	F	$I \div D$	F
.251	.15441	.301	.19908	.351	.24593	.401	.29435	.451	.34378
.252	.15528	.302	.20000	.352	.24689	.402	.29533	.452	.34477
.253	.15615	.303	.20092	.353	.24784	.403	.29631	.453	.34577
.254	.15702	.304	.20184	.354	.24880	.404	.29729	.454	.34676
.255	.15789	.305	.20276	.355	.24976	.405	.29827	.455	.34776
.256	.15876	.306	.20368	.356	.25071	406	.29925	.456	.34875
.257	.15964	.307	.20460	.357	.25167	.407	.30024	.457	.34975
.258	.16051	.308	.20553	.358	.25263	.408	30122	.458	.35075
.259	.16139	.309	20645	.359	25359	.409	.30220	.459	.35174
.260	.16226	.310	.20738	.360	.25455	410	.30319	.460	.35274
.261	.16314	.311	.20830	.361	.25551	411	.30417	.461	.35374
.262	.16402	.312	.20923	.362	.25647	.412	.30515	.462	.35474
.263	.16490	.313	.21015	.363	.25743	.413	.30614	.463	.35573
.264	.16578	.314	.21108	.364	.25839	.414	.30712	.464	.35673
.265	.16666	.315	.21201	.365	.25936	.415	.30811	.465	.35773
.266	.16755	.316	.21294	.366	.26032	.416	.30909	.466	.35872
.267	.16844	.317	.21387	.367	.26128	.417	.31008	.467	.35972
.268	.16931	.318	.21480	.368	.26225	.418	.31107	.468	.36072
.269	.17020	.319	.21573	.369	.26321	.419	.31205	.469	.36172
.270	.17109	.320	.21667	.370	.26418	.420	.31304	.470	.36272
.271	.17197	.321	.21760	.371	.26514	.421	.31403	.471	.36371
.272	.17287	.322	.21853	.372	.26611	.422	.31502	.472	.36471
.273	.17376	.323	.21947	.373	.26708	.423	.31600	.473	.36571
.274	.17465	.324	.22040	.374	.26804	.424	.31699	.474	.36671
.275	.17554	.325	.22134	.375	.26901	.425	.31798	.475	.36771
.276	.17643	.326	.22228	.376	.26998	.426	.31897	.476	.36871
.277	.17733	.327	.22321	.377	.27095	.427	.31996	.477	.36971
.278	.17822	.328	.22415	.378	.27192	.428	.32095	.478	.37071
.279	.17912	.329	.22509	.379	.27289	.429	.32194	.479	.3717
280	.18002	.330	.22603	.380	.27386	.430	.32293	.480	.3727
281	.18092	.331	.22697	.381	.27483	.431	.32391	.481	.3737
.282	.18182	.332	.22791	.382	.27580	.432	.32490	.482	.3747
.283	.18272	.333	.22886	.383	.27677	.433	.32590	.483	.3757
.284	.18361	.334	.22980	.384	.27775	.434	.32689	.484	.3767
.285	.18452	.335	.23074	.385	.27872	.435	.32788	.485	.3777
.286	.18542	.336	.23169	.386	.27969	.436	.32887	.486	.3787
.287	.18633	.337	.23263	.387	.28067	.437	.32987	.487	.3797
.288	.18723	.338	.23358	.388	.28164	.438	.33086	.488	.3807
.289	.18814	.339	.23453	.389	.28262	.439	.33185	.489	.3817
.290	.18905	.340	.23547	.390	.28359	.440	.33284	.490	.3827
.291	.18995	.341	.23642	.391	.28457	.441	.33384	.491	.3837
.292	.19086	.342	.23737	.392	.28554	.442	.33483	.492	.3847
.293	.19177	.343	.23832	.393	.28652	.443	.33582	.493	.3857
.294	.19268	.344	.23927	.394	.28750	.444	.33682	.494	.3867
.295	.19360	.345	.24022	.395	.28848	.445	.33781	.495	.3877
.296	.19451	.346	.24117	.396	.28945	.446	.33880	.496	.3887
.297	.19542	.347	.24212	.397	.29043	.447	.33980	.497	.3897
.298	.19634	.348	.24307	.398	.29141	.448	.34079	.498	.3907
.299	.19725	.349	.24403	.399	.29239	.449	.34179	.499	.3917
.300	.19817	.350	.24498	.400	.29337	.450	.34278	.500	.3927

TABLE 7

DENSITY (IN GRAMS PER MILLILITER) OF WATER AT TEMPERATURES FROM 0° TO 102° C.*

Temperature, Degrees C.	Density	Temperature, Degrees C.	Density	Temperature, Degrees C.	Density
0	0.99987	35	0.99406	70	0.97781
1	.99993	36	.99371	71	.97723
2	.99997	37	.99336	72	.97666
3	.99999	38	.99299	73	.97607
4	1.00000	39	.99262	74	.97548
5	.99999	40	.99224	75	.97489

TABLE 8

Weight of 1 Cubic Foot and 1 Gallon (U. S. A. 231 Cu. In.) of Water [1]

Based on the water densities of P. Chappuis (Bureau International des Poids et Mesures, Travaux et Mémoires, XIII; 1907) for 0° to 40° C. and of M. Thiesen (Wis. Abh. der Physikalisch-Technischen Reichsanstalt, 4, No. 1; 1904) for 41° to 100° C. The weights in air are for dry air at the same temperature as the water up to 40° C. and at a (corrected) barometric pressure of 760 mm. and against brass weights of 8.4 density at 0° C. Above 40° C. the temperature of the air is assumed to be 20° C. The volumetric computations are based on the relation that 1 liter = 1.000027 cubic decimeters, and 1 cubic decimeter = 61.023378 cubic inches.

Temperature ° C.	Weight in Air			
	1 Cubic Foot		1 Gallon	
	Grams	Pounds	Grams	Pounds
0	28280.254	62.3472	3780.520	8.33461
1	82.033	.3511	0.758	.33613
2	83.321	.3540	0.930	.33551
3	84.121	.3557	1.037	.33575
4	84.458	.3565	1.082	.33585
5	84.345	.3562	1.067	.33582
6	83.784	.3550	0.992	.33565
7	82.804	.3528	0.861	.33536
8	81.413	.3498	0.675	.33495
9	79.618	.3458	0.435	.33442
10	77.441	.3410	0.144	.33378
11	74.853	.3358	3779.798	.33302
12	71.898	.3288	9.403	.33215
13	68.584	.3215	8.960	.33117
14	64.933	.3134	8.472	.33009
15 *	60.946	.3046	7.939	.32892
16 †	56.630	.2951	7.362	.32765
17	51.985	.2849	6.741	.32628
18	47.040	.2740	6.080	.32482
19	41.766	.2624	5.375	.32327
20	36.193	.2501	4.630	.32162
21	30.321	.2371	3.845	.31989
22	24.157	.2235	3.021	.31808
23	17.694	.2093	2.157	.31617
24	10.954	.1944	1.256	.31419
* $15\frac{5}{9}$ (60° F.)	28258.580	62.2994	3777.623	8.32820
† $16\frac{2}{3}$ (62° F.)	28253.57	62.2884	3776.953	8.32670

[1] Calculated by the U. S. Bureau of Standards, Washington, January, 1915.

TABLE 8 (*Continued*)

WEIGHT OF 1 CUBIC FOOT AND 1 GALLON

Temperature ° C.	Weight in Air			
	1 Cubic Foot		1 Gallon	
	Grams	Pounds	Grams	Pounds
25	28203.930	62.1789	3770.317	8.31212
26	28196.629	.1629	3769.341	.30996
27	89.059	.1462	8.329	.30773
28	81.234	.1289	7.283	.30543
29	73.140	.1111	6.201	.30304
30	64.799	.0927	5.086	.30058
31	56.212	.0737	3.938	.29805
32	47.377	.0543	2.757	.29545
33	38.311	.0343	1.545	.29278
34	29.005	.0138	0.301	.29003
35	19.475	61.9926	3759.027	.28723
36	09.735	.9713	7.725	.28436
37	28099.771	.9493	6.393	.28142
38	89.596	.9269	5.033	.27842
39	79.215	.9040	3.645	.27536
40	68.645	.8807	2.232	.27225
41	55.68	.8520	0.50	.2684
42	44.62	.828	3749.02	.2652
43	33.30	.803	7.51	.2618
44	21.69	.777	5.96	.2584
45	09.80	.751	4.36	.2549
46	27997.90	.725	2.78	.2514
47	86.01	.698	1.18	.2479
48	73.54	.671	3739.52	.2442
49	61.09	.644	7.85	.2405
50	48.34	.615	6.15	.2368
51	35.60	.587	4.45	.2330
52	22.29	.558	2.67	.2291
53	09.26	.529	0.93	.2253
54	27895.66	.499	3729.11	.2213
55	82.07	.469	7.29	.2173
56	68.20	.439	5.44	.2132
57	55.17	.410	3.70	.2093
58	40.15	.377	1.69	.2049
59	26.00	.346	3719.80	.2007
60	11.55	.314	7.86	.1965
61	27796.83	.281	5.90	.1921
62	82.10	.249	3.93	.1878
63	67.09	.216	1.92	.1834
64	51.80	.182	3709.88	.1789

TABLE 8 (*Continued*)

WEIGHT OF 1 CUBIC FOOT AND 1 GALLON

Temperature ° C.	Weight in Air			
	1 Cubic Foot		1 Gallon	
	Grams	Pounds	Grams	Pounds
65	27736.50	61.148	3707.83	8.1744
66	21.21	.115	5.79	.1699
67	05.64	.080	3.70	.1653
68	27689.79	.046	1.58	.1606
69	73.91	.010	3699.46	.1559
70	57.78	60.975	7.31	.1512
71	41.35	.939	5.11	.1463
72	25.21	.903	2.95	.1416
73	08.50	.866	0.72	.1366
74	27591.79	.829	3688.49	.1317
75	75.08	.793	3686.25	.1268
76	57.81	.754	3.94	.1217
77	40.81	.717	1.67	.1167
78	23.53	.679	3679.36	.1116
79	05.97	.640	7.01	.1064
80	27488.42	.602	4.67	.1012
81	70.58	.562	2.28	.0960
82	52.74	.523	3669.90	.0907
83	34.90	.484	7.51	.0855
84	16.77	.444	5.09	.0801
85	27398.36	.403	2.63	.0747
86	79.96	.362	0.17	.0693
87	61.26	.321	3657.67	.0638
88	42.57	.280	5.17	.0583
89	23.59	.238	2.63	.0527
90	04.62	.196	0.10	.0471
91	27285.65	.154	3647.56	.0415
92	66.38	.112	4.99	.0358
93	46.85	.069	2.37	.0301
94	27.31	.026	3639.76	.0243
95	07.76	59.983	7.15	.0185
96	27187.94	.939	4.50	.0127
97	67.84	.895	1.81	.0068
98	48.01	.851	3629.16	.0009
99	27.62	.806	6.44	7.9949
100	07.51	.762	3.75	.9889

TABLE 9

For the Calibration of Flasks to True Cubic Centimeters at 20° C.*

[This table gives the apparent weight for temperature between 15° and 30° C., humidity 50 per cent, unreduced barometer reading 76 cm., of certain volumes of water weighed with brass weights. The table may be conveniently employed to determine definite volumes of water for calibrating instruments. The air is assumed to be at the same temperature as the water.]

Temp. ° C.	2000 ml.	1000 ml.	500 ml.	400 ml.	300 ml.	250 ml.	150 ml.
15	1996.11	998.05	499.03	399.22	299.42	249.51	149.71
16	1995.80	997.90	498.95	399.16	299.37	249.48	149.68
17	1995.48	997.74	498.87	399.10	299.32	249.43	149.66
18	1995.13	997.56	498.78	399.03	299.27	249.39	149.63
19	1994.76	997.38	498.69	398.95	299.21	249.34	149.61
20	1994.36	997.18	498.59	398.87	299.15	249.30	149.58
21	1993.95	996.97	498.49	398.79	299.09	249.24	149.55
22	1993.51	996.76	498.38	398.70	299.03	249.19	149.51
23	1993.06	996.53	498.26	398.61	298.96	249.13	149.48
24	1992.58	996.29	498.15	398.52	298.89	249.07	149.44
25	1992.09	996.04	498.02	398.42	298.81	249.01	149.41
26	1991.57	995.79	497.89	398.31	208.74	248.95	149.37
27	1991.04	995.52	497.76	398.21	298.66	248.88	149.33
28	1990.49	995.24	497.62	398.10	298.57	248.81	149.29
29	1989.92	994.96	497.48	397.98	298.49	248.74	149.24
30	1989.33	994.66	497.33	397.87	298.40	248.67	149.20

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* From *U. S. Bur. Standards Circ. C 440*, 1942, p. 612.

TABLE 10

TABLES OF CORRECTIONS FOR DETERMINING THE TRUE CAPACITIES OF FLASKS FROM THE WEIGHT OF WATER IN AIR *

(Following data are assumed as approximating ordinary conditions: Observed barometric pressure = 76 cm.; relative humidity = 50 per cent; coefficient of expansion of glass = 0.000025 per degree C.)

INDICATED CAPACITY 50 ML.

Temp. ° C.	Tenths of Degrees									
	0	1	2	3	4	5	6	7	8	9
15	0.104	0.104	0.105	0.106	0.106	0.107	0.107	0.108	0.109	0.109
16	.110	.111	.111	.112	.113	.113	.114	.115	.116	.116
17	.117	.118	.118	.119	.120	.121	.121	.122	.123	.124
18	.124	.125	.126	.127	.128	.128	.129	.130	.131	.132
19	.132	.133	.134	.135	.136	.137	.137	.138	.139	.140
20	.141	.142	.143	.144	.144	.145	.146	.147	.148	.149
21	.150	.151	.152	.153	.154	.155	.156	.157	.158	.159
22	.160	.161	.162	.163	.164	.165	.166	.167	.168	.169
23	.170	.171	.172	.173	.174	.175	.176	.177	.178	.179
24	.180	.182	.183	.184	.185	.186	.187	.188	.189	.190
25	.192	.193	.194	.195	.196	.197	.199	.200	.201	.202
26	.203	.204	.206	.207	.208	.209	.210	.212	.213	.214
27	.215	.216	.218	.219	.220	.222	.223	.224	.225	.226
28	.228	.229	.230	.232	.233	.234	.236	.237	.238	.240
29	.241	.242	.244							

INDICATED CAPACITY 100 ML.

Temp. ° C.	Tenths of Degrees									
	0	1	2	3	4	5	6	7	8	9
15	0.207	0.208	0.210	0.211	0.212	0.213	0.215	0.216	0.217	0.219
16	.220	.221	.223	.224	.225	.227	.228	.230	.231	.232
17	.234	.235	.237	.238	.240	.241	.243	.244	.246	.247
18	.249	.250	.252	.253	.255	.257	.258	.260	.261	.263
19	.265	.266	.268	.270	.272	.273	.275	.277	.278	.280
20	.282	.284	.285	.287	.289	.291	.293	.294	.296	.298
21	.300	.302	.304	.306	.308	.310	.311	.314	.315	.317
22	.319	.321	.323	.325	.327	.329	.331	.333	.336	.338
23	.340	.342	.344	.346	.348	.350	.352	.354	.357	.359
24	.361	.363	.365	.368	.370	.372	.374	.376	.379	.381
25	.383	.386	.388	.390	.392	.395	.397	.399	.402	.404
26	.406	.409	.411	.414	.416	.418	.421	.423	.426	.428
27	.431	.433	.436	.438	.440	.443	.446	.448	.451	.453
28	.456	.458	.461	.463	.466	.469	.471	.474	.476	.479
29	.482	.484	.487							

The tables of corrections give for each nominal capacity and observed temperature the amounts to be added to the apparent weight (in air against brass weights) of the water contained by the flask to give the capacity in True Cubic Centimeters at 20° C. Example: Apparent weight of water at 22.3° = 99.68; adding correction 0.325 = 100.005, the actual capacity.

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* From Circular No. 19, U. S. Bureau of Standards, April 1, 1914.

TABLE 11

VOLUME OF SUCROSE SOLUTIONS AT DIFFERENT TEMPERATURES *

(*U.S. Bur. Standards Circ. C 440* (1942), pp. 648–649)

(Volume at 20° C. = 1.0000)

Percentage of Sucrose by Weight

Temperature, °C.	0	5	10	15	20	25	30	35	40	50	60	70	75
0	0.9984	0.9976	0.9969	0.9964	0.9958	0.9954	0.9949	0.9945	0.9941	0.9934	0.9929	0.9926	0.9927
5	.9982	.9978	.9974	.9970	.9966	.9963	.9960	.9957	.9954	.9949	.9946	.9944	.9945
10	.9985	.9983	.9981	.9978	.9976	.9974	.9972	.9970	.9969	.9966	.9963	.9962	.9964
15	.9991	.9990	.9989	.9988	.9987	.9986	.9985	.9984	.9984	.9982	.9981	.9981	.9982
20	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000
25	1.0012	1.0012	1.0013	1.0014	1.0014	1.0015	1.0016	1.0017	1.0018	1.0015	1.0019	1.0020	1.0019
30	1.0026	1.0026	1.0027	1.0029	1.0030	1.0032	1.0033	1.0035	1.0036	1.0038	1.0039	1.0040	1.0038
35	1.0042	1.0043	1.0044	1.0046	1.0048	1.0050	1.0052	1.0054	1.0056	1.0058	1.0060	1.0061	1.0058
40	1.0060	1.0061	1.0063	1.0065	1.0068	1.0070	1.0072	1.0074	1.0076	1.0079	1.0081	1.0082	1.0078
45	1.0080	1.0081	1.0084	1.0086	1.0089	1.0091	1.0094	1.0096	1.0098	1.0100	1.0102	1.0103	1.0099
50	1.0102	1.0104	1.0106	1.0109	1.0112	1.0115	1.0117	1.0119	1.0120	1.0123	1.0125	1.0125	1.0121
55	1.0126	1.0128	1.0131	1.0134	1.0137	1.0139	1.0141	1.0143	1.0144	1.0147	1.0148	1.0148	1.0143
60	1.0152	1.0155	1.0158	1.0161	1.0163	1.0165	1.0167	1.0168	1.0169	1.0171	1.0172	1.0171	1.0166
65	1.0179	1.0184	1.0186	1.0189	1.0191	1.0194	1.0196	1.0196	1.0200	1.0199	1.0198	1.0189	1.0190
70	1.0209	1.0213	1.0215	1.0218	1.0218	1.0221	1.0223	1.0224	1.0227	1.0225	1.0223	1.0215	1.0214
75	1.0241	1.0243	1.0244	1.0247	1.0248	1.0250	1.0251	1.0252	1.0254	1.0252	1.0250	1.0241	1.0238
80	1.0274	1.0273	1.0275	1.0277	1.0279	1.0280	1.0281	1.0281	1.0283	1.0280	1.0277	1.0268	1.0263
85	1.0308	1.0307	1.0309	1.0309	1.0312	1.0312	1.0312	1.0312	1.0312	1.0310	1.0305	1.0294	1.0289
90	1.0342	1.0342	1.0344	1.0344	1.0347	1.0346	1.0345	1.0344	1.0343	1.0340	1.0334	1.0322	1.0316
95	1.0376	1.0379	1.0380	1.0380	1.0382	1.0381	1.0379	1.0377	1.0375	1.0371	1.0363	1.0351	1.0343
100	1.0411	1.0417	1.0418	1.0417	1.0417	1.0415	1.0413	1.0412	1.0409	1.0403	1.0393	1.0380	1.0370

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* Factors for concentrations from 0 to 70 per cent at temperatures from 0° to 60° C. computed from Plato's density tables (*Kaiserlichen Normal-Eichungs-Kommission, Wiss. Abh. 2* [1900]), remaining factors computed from Th. Gerlach, *Z. Ver. deut. Zuckerind.*, **13**, 320 (1863).

TABLE 12

BOILING POINT RISE OF SUGAR SOLUTIONS *

ELEVATION OF BOILING POINT (BOILING POINT RISE) OF SUGAR SOLUTIONS ABOVE THAT OF WATER

For vapor pressure 14.696 lb. per sq. in.; 760 mm. of mercury; boiling point of water 100° C. †

Solids per 100 grams of Water, Given in Grams	Brix	100° Purity	90° Purity ‡	80° Purity ‡	70° Purity ‡
50	33.33	0.74	0.86	1.01	1.21
100	50.00	1.79	2.11	2.43	2.89
150	60.00	2.98	3.46	3.96	4.64
200	66.67	4.25	4.85	5.54	6.42
250	71.43	5.57	6.26	7.14	8.19
300	75.00	6.88	7.65	8.71	9.94
350	77.78	8.18	9.02	10.24	11.65
400	80.00	9.44	10.33	11 73	13.30
450	81.82	10.65	11.60	13.15	14.88
500	83.33	11.81	12.81	14.50	16.39
550	84.62	12.93	13.96	15.79	17.84
600	85.71	13.98	15.06	17.01	19.20
650	86.67	14.98	16.09	18.16	20.49
700	87.50	15.93	17.08	19.26	21.72
750	88.24	16.84	18.01	20.30	22.88
800	88.89	17.69	18.90	21.38	23.99
850	89.47	18.51	19.74	22.21	25.03
900	90.00	19.27	20.53	23.09	26.03
950	90.48	20.01	21.29	23.92	26.97
1000	90.91	20.70	22.00	24.72	27.86
1050	91.30	21.36	22.68	25.47	28.71
1100	91.67	21.99	23.33	26.18	29.52
1150	92.00	22.58	23.94	26.87	30.28
1200	92.31	23.16	24.54	27.51	31.02
1250	92.59	23.70	25.09	28.13	31.72
1300	92.86	24.22	25.63	28.72	32.38
1350	93.10	24.71	26.14	29.28	33.03
1400	93.33	25.18	26.63	29.81	33.63
1450	93.55	25.64	27.09	30.33	34.21
1500	93.75	26.07	27.54	30.82	34.77
1550	93.94	26.49	27.97	31.30	35.31
1600	94.12	26.88	28.39	31.75	35.83

* Condensed from *U.S. Bur. Standards Circ. C 440*, 1942, pp. 694–701. See also discussion of theory, *ibid.*, pp. 365–370.

† The boiling point is dependent on the pressure at which the boiling takes place. See Holven, *Ind. Eng. Chem.*, April, 1936, p. 452; *ibid.*, October, 1942, p. 1234.

‡ Values in the table for impure sugar solutions are based on determinations made on product in which the impurity was beet molasses.

TABLE 13

SPECIFIC GRAVITIES OF COMMON ACIDS AND ALKALIES. AQUEOUS SOLUTIONS

(From *International Critical Tables*, 1928)

Direct interpolation may be used for fractions of per cents

Per Cent Material by Weight	HCl	HNO_3	H_2SO_4	H_3PO_4	NaOH	KOH	NH_3
	20°/4°	20°/4°	20°/4°	20°/4°	20°/4°	15°/4°	20°/4°
1	1.0032	1.00364	1.0051	1.0038	1.0095	1.0083	0.9939
2	1.0082	1.00909	1.0118	1.0092	1.0207	1.0175	0.9895
3	....	1.01457	1.0184	....	1.0318	1.0267	
4	1.0181	1.02008	1.0250	1.0200	1.0428	1.0359	0.9811
5	....	1.02563	1.0317	....	1.0538	1.0452	
6	1.0279	1.03122	1.0385	1.0309	1.0648	1.0544	0.9730
7	....	1.0369	1.0453	....	1.0758	1.0637	
8	1.0376	1.0427	1.0522	1.0420	1.0869	1.0730	0.9651
9	....	1.0485	1.0591	....	1.0979	1.0824	
10	1.0474	1.0543	1.0661	1.0532	1.1089	1.0918	0.9575
11	....	1.0602	1.0731	....	....	1.1013	
12	1.0574	1.0661	1.0802	1.0647	1.1309	1.1108	0.9501
13	....	1.0721	1.0874	....	....	1.1203	
14	1.0675	1.0781	1.0947	1.0764	1.1530	1.1299	0.9430
15	....	1.0842	1.1020	....	....	1.1396	
16	1.0776	1.0903	1.1094	1.0884	1.1751	1.1493	0.9362
17	....	1.0964	1.1168	....	....	1.1590	
18	1.0878	1.1026	1.1243	1.1008	1.1972	1.1688	0.9295
19	....	1.1088	1.1318	....	....	1.1786	
20	1.0980	1.1150	1.1394	1.1134	1.2191	1.1884	0.9229
21	....	1.1213	1.1471	....	....	1.1984	
22	1.1083	1.1276	1.1548	1.1263	1.2411	1.2083	0.9164
23	....	1.1340	1.1626	....	....	1.2184	
24	1.1187	1.1404	1.1704	1.1395	1.2629	1.2285	0.9101
25	....	1.1469	1.1783	....	....	1.2387	
26	1.1290	1.1534	1.1862	1.1529	1.2848	1.2489	0.9040
27	....	1.1600	1.1942	....	....	1.2592	
28	1.1392	1.1666	1.2023	1.1665	1.3064	1.2695	0.8980
29	....	1.1733	1.2104	....	....	1.2800	
30	1.1493	1.1800	1.2185	1.1805	1.3279	1.2905	0.8920
31	....	1.1867	1.2267	....	....	1.3010	
32	1.1593	1.1934	1.2349	....	1.3490	1.3117	
33	....	1.2002	1.2432	....	....	1.3224	
34	1.1691	1.2071	1.2515	....	1.3696	1.3331	
35	....	1.2140	1.2599	1.216	....	1.3440	
36	1.1789	1.2205	1.2684	....	1.3900	1.3549	
37	....	1.2270	1.2769	....	....	1.3659	
38	1.1885	1.2325	1.2855	....	1.4101	1.3769	
39	....	1.2399	1.2941	....	....	1.3879	
40	1.1980	1.2463	1.3028	1.254	1.4300	1.3991	
45	....	1.2783	1.3476	1.293	....	1.4558	
50	....	1.3100	1.3951	1.355	1.5253		
55	....	1.3393	1.4453	1.379			
60	....	1.3667	1.4983	1.426			
70	....	1.4134	1.6105				
80	....	1.4521	1.7272				
90	....	1.4826	1.8144				
100	....	1.5217	1.8305				

TABLE 14

SHOWING THE AMOUNT OF CaO IN MILK OF LIME OF VARIOUS DENSITIES AT 15° C.

(From Blatner's Table)

Degree Brix	Degree Baumé	Weight of one Liter, Milk of Lime, Grams	CaO per Liter, Grams	Per Cent CaO	Degree Brix	Degree Baumé	Weight of One Liter, Milk of Lime, Grams	CaO per Liter, Grams	Per Cent CaO
1.8	1	1007	7.5	0.745	29	16	1125	159	14.13
3.6	2	1014	16.5	1.64	30.8	17	1134	170	15
5.4	3	1022	26	2.54	32.7	18	1142	181	15.85
7.2	4	1029	36	3.5	34.6	19	1152	193	16.75
9	5	1037	46	4.43	36.4	20	1162	206	17.72
10.8	6	1045	56	5.36	38.3	21	1171	218	18.61
12.6	7	1052	65	6.18	40.1	22	1180	229	19.4
14.4	8	1060	75	7.08	42	23	1190	242	20.34
16.2	9	1067	84	7.87	43.9	24	1200	255	21.25
18	10	1075	94	8.74	45.8	25	1210	268	22.15
19.8	11	1083	104	9.6	47.7	26	1220	281	23.03
21.7	12	1091	115	10.54	49.6	27	1231	295	23.96
23.5	13	1100	126	11.45	51.5	28	1241	309	24 9
25.3	14	1108	137	12 35	53 5	29	1252	324	25.87
27.2	15	1116	148	13.26	55.4	30	1263	339	26.84

TABLE 15

SHOWING THE SOLUBILITY OF LIME IN SOLUTIONS OF SUGAR

Sugar in 100 Parts Water	Density of Sirup	Density after Saturation with Lime	100 Parts of the Residue Dried at 120° C. Contain:	
			Lime	Sugar
40	1.122	1.179	21	79
35	1.110	1.166	20.5	79.5
30	1.096	1.148	20.1	79.9
25	1.082	1.128	19.8	80.2
20	1.068	1.104	18.8	81.2
15	1.052	1.080	18.5	81.5
10	1.036	1.053	18.1	81.9
5	1.018	1.026	15.3	84.7

TABLE 16

Showing the Percentage of Lead Acetate in Solutions of the Salt, of Different Densities, at 15° C. (Gerlach)

Specific Gravity	Per Cent of the Salt	Specific Gravity	Per Cent of the Salt	Specific Gravity	Per Cent of the Salt
1.0127	2	1.1384	20	1.2768	36
1.0255	4	1.1544	22	1.2966	38
1.0386	6	1.1704	24	1.3163	40
1.0520	8	1.1869	26	1.3376	42
1.0654	10	1.2040	28	1.3588	44
1.0796	12	1.2211	30	1.3810	46
1.0939	14	1.2395	32	1.4041	48
1.1084	16	1.2578	34	1.4271	50
1.1234	18				

TABLE 17

Showing the Solubility of Barium Oxide in Sugar Solutions

(Pellet and Sencier, *La fabrication du sucre*, 1, 186)

Sucrose per 100 Cc.	Baryta (BaO) per 100 Cc.	Baryta (BaO) Per Cent Sucrose
2.5	4.59	18.3
5	5.46	10.9
7.5	6.56	8.7
10	7.96	7.7
12.5	9.41	7.5
15	10.00	6.6
20	10.90	5.4
25	12.90	5.1
30	14.68	4.9

TABLE 18

Showing the Solubility of Certain Salts in Water in the Presence of Sucrose

(Jacobsthal, *Zeit. Rübenzuckerind*, 18, 649; taken from Sidersky's *Traité d'analyse des matières sucrées*, p. 11)

Solution containing	5% Sucrose, Grams	10% Sucrose, Grams	15% Sucrose, Grams	20% Sucrose, Grams	25% Sucrose, Grams
Calcium sulphate.......	2.095	1.946	1.593	1.539	1.333
Calcium carbonate.....	0.027	0.036	0.024	0.022	0.008
Calcium oxalate........	0.033	0.047	0.012	0.008	0.001
Calcium phosphate.....	0.029	0.028	0.014	0.018	0.005
Calcium citrate........	1.813	1.578	1.505	1.454	1.454
Magnesium carbonate...	0.317	0.199	0.194	0.213	0.284

TABLE 19

SHOWING THE SOLUBILITY OF SUGAR IN ALCOHOL AT 17.5° C.

(OTTO SCHREFELD)

(*Zeit. Rübenzuckerind.*, **44**, 970)

Alcohol Per Cent by Weight	Sucrose Per Cent	Sucrose in Grams in 100 Grams of the Mixture of Alcohol and Water Solution
0	66 20	195 8
5*	64 25	179 7
10*	62 20	164 5
15	60 40	152 5
20*	58 55	141 2
25	56 20	128 3
30	54 05	117 8
35	51 25	105 3
40	47 75	91 3
45	43 40	76 6
50	38 55	62 7
55	32 80	48 8
60	26 70	36 4
65	19 50	24 2
70	12 25	13 9
75	7 20	7 7
80	4 05	4 2
85	2 10	2 1
90	0 95	0 09
95	0 15	0 01
Absolute	0 00	0 00

* Calculated.

TABLE 20

SOLUBILITY OF SUCROSE IN WATER (HERZFELD) *

Temp. °C.	Sucrose in 100 Grams of Solution	Sucrose Dissolved by 100 Grams of Water	Temp. °C.	Sucrose in 100 Grams of Solution	Sucrose Dissolved by 100 Grams of Water
0	64.18	179 2	50	72.25	260.4
2	64.45	181 3	52	72.63	265.4
4	64.73	183.5	54	73.01	270.5
6	65.01	185.8	56	73.39	275.8
8	65.29	188.1	58	73.78	281.4
10	65.58	190.5	60	74.18	287.3
12	65.88	193.1	62	74.58	293.4
14	66.18	195.7	64	74.98	299.7
16	66.51	198.3	66	75.38	306.2
18	66.78	201.0	68	75.80	313.2
20	67.09	203.9	70	76.22	320.5
22	67.41	206.8	72	76.64	328.1
24	67.73	209.9	74	77.06	335.9
26	68.05	213.0	76	77.48	344.0
28	68.37	216.2	78	77.92	252.9
30	68.70	219.5	80	78.36	362.2
32	69.04	223.0	82	78.80	371.7
34	69.38	226.6	84	79.24	381.7
36	69.72	230.3	86	79.69	392.4
38	70.06	234.0	88	80.15	403.8
40	70.42	238.1	90	80.61	415.7
42	70.78	242.2	92	81.07	428.3
44	71.14	246.5	94	81.53	441.4
46	71.50	250.9	96	82.01	455.9
48	71.87	255.5	98	82.49	471.1
			100	82.97	487.2

* See Table 20*a* for relationship to work of other investigators.

TABLE 20*a*

SOLUBILITY OF SUCROSE IN PURE WATER

(Various Investigators) *

(Grams of sugar per 100 grams of water)

Temperature	Flourens 1876	Herzfeld 1892	Horsin-Deon	Scott-Macfie	Lebedev 1908	Mondain-Monval 1924	Grut 1936	Hruby-Kasjanov 1936
°C.								
0	183	179.2	183	163				
0.9						181		
10	190	190.5	190	179				
15.8						196		
20	199	203.9	199	197			201	
25.6						211		
30	213	219.5	213	216	220		218	
30.5						218		
40	231	238.1	231	239			238	
50	255	260.4	255	266	258		263	260
60	285	287.3	283	296			293	
70	319	320.5	319	333	330		330	
80	362	362.1	362	376			390	373
90	415	415.7	415	430				
100	471	487.2	485	497				

* Compiled by R. Hruby and V. Kasjanov, *Intern Sugar J.*, January, 1940, p. 21. Herzfeld's values up to 50° C. are supported by those of other investigators, but at higher temperatures recent workers, as shown above, find considerably higher figures. The results in Tables 20 and 20*a* are all for pure sucrose. For impure sugar solutions Hruby's results show that with declining purity the solubility of sucrose at first falls, reaching a minimum at about 92° purity, and then rises again.

TABLE 21

INCREASE IN VOLUME WHEN SUCROSE IS DISSOLVED IN WATER AT 20° C. (GRAMS/100 ML.)

(*Bur. Standards Circ. C 440*, 1942, p. 642)

Grams of Sucrose in 100 Ml. of Water	Resultant Solution			Grams of Sucrose in 100 Ml. of Water	Resultant Solution		
	Sucrose by weight (Brix)	Specific gravity 20°/4° C.	Increase in volume ml.		Sucrose by weight (Brix)	Specific gravity 20°/4° C.	Increase in volume ml.
1	2	3	4	1	2	3	4
5	4.7699	1.01694	3.078	90	47.4125	1.21546	56.174
10	9.1055	1.03446	6.165	100	50.0442	1.22981	62.483
15	13.0635	1.05093	9.259	110	52.4250	1.24301	68.802
20	16.6912	1.06645	12.357	120	54.5893	1.25520	75.130
25	20.0283	1.08109	15.461	130	56.5652	1.26649	81.465
30	23.1083	1.09491	18.570	140	58.3763	1.27696	87.808
35	25.9599	1.10799	21.683	150	60.0424	1.28671	94.157
40	28.6075	1.12037	24.801	160	61.5803	1.29579	100.513
45	31.0723	1.13212	27.922	170	63.0042	1.30429	106.873
50	33.3726	1.14327	31.048	180	64.3263	1.31225	113.239
55	35.5243	1.15387	34.177	190	65.5572	1.31972	119.609
60	37.5414	1.16396	37.310	200	66.7059	1.32675	125.984
65	39.4361	1.17356	40.447	210	67.7805	1.33337	132.362
70	41.2193	1.18273	43.587	220	68.7880	1.33961	138.744
75	42.9004	1.19147	46.729	230	69.7343	1.34551	145.129
80	44.4881	1.19983	49.874	240	70.6249	1.35110	151.517

(*Example.* One hundred milliliters of water at 20° C. is taken and 130 grams of sucrose is dissolved therein. The resultant solution at 20° C. has increased in volume 81.465 ml. (column 4), the total volume being 100 + 81.465 = 181.465 ml.)

TABLE 21*a*

INCREASE IN VOLUME WHEN SUCROSE IS DISSOLVED IN WATER AT 20° C. (POUNDS PER GALLON)

(*Bur. Standards Circ. C 440*, 1942, p. 643)

Pounds Sucrose in 1 Gal. of Water	Resultant Solution			Pounds Sucrose in 1 Gal. of Water	Resultant Solution		
	Sucrose by weight (Brix)	Specific gravity 20°/4° C.	Increase in volume		Sucrose by weight (Brix)	Specific gravity 20°/4° C.	Increase in volume
1	2	3	4	1	2	3	4
0.1	1.1862	1.0028	0.007	7	45.6606	1.2061	0.523
.2	2.3443	1.0074	.015	8	48.9881	1.2240	.599
.3	3.4760	1.0118	.022	9	51.9816	1.2403	.674
.4	4.5816	1.0162	.029	10	54.5539	1.2550	.750
.5	5.6622	1.0205	.037	11	56.9049	1.2684	.826
.6	6.7186	1.0247	.044	12	59.0246	1.2807	.902
.7	7.7515	1.0289	.052	13	60.9456	1.2920	.978
.8	8.7619	1.0331	.059	14	62.6945	1.3024	1.055
.9	9.7503	1.0371	.067	15	64.2935	1.3121	1.131
1	10.7175	1.0411	.074	16	65.7611	1.3210	1.207
2	19.3602	1.0781	.148	17	67.1128	1.3292	1.283
3	26.4772	1.1104	.223	18	68.3618	1.3370	1.360
4	32.4399	1.1387	.297	19	69.5194	1.3442	1.436
5	37.5080	1.1638	.373	20	70.5953	1.3509	1.513
6	41.8688	1.1861	.448				

(*Example.* One gallon of water at 20° C. is taken and 5 lb. of sugar (sucrose) is dissolved therein. The resultant solution at 20° C. has increased in volume 0.373 gal. (column 4), the total volume being 1 + 0.373 = 1.373 gal.)

TABLE 22

SHOWING THE CONTRACTION OF INVERT SUGAR ON DISSOLVING IN WATER; ALSO, THE CONTRACTION OF CANE SUGAR SOLUTIONS ON INVERSION

(From *Manuel Agenda*, Gallois and Dupont)

Per Cent Sugar	Volume	Contraction	Specific Gravity	
			Cane-sugar Solution	Invert-sugar Solution
0	1.00000	0.00000	1.0000	1.0000
5	.99863	0.00137	1.0203	1.0206
10	.99744	0.00256	1.0413	1.0418
15	.99639	0.00361	1.0630	1.0631
20	.99546	0.00454	1.0854	1.0856
25	.99462	0.00538	1.1086	1.1086

TABLE 23

SHOWING THE EVAPORATION OF WATER IN CONCENTRATING JUICE TO SIRUP (SPENCER)

(Percentages by weight, see p. 634)

Brix of Juice	Degree Brix of the Sirup and the Evaporation in Percentage Terms of the Juice									
	52.0	52.5	53.0	53.5	54.0	54.5	55.0	55.5	56.0	56.5
11.0	78.8	79.0	79.2	79.4	79.6	79.8	80.0	80.2	80.4	80.5
.2	78.4	78.6	78.8	79.0	79.2	79.4	79.6	79.8	80.0	80.2
.4	78.0	78.2	78.5	78.7	78.9	79.0	79.3	79.5	79.7	79.8
.6	77.7	77.9	78.1	78.3	78.5	78.7	78.9	79.1	79.3	79.5
.8	77.3	77.5	77.7	78.0	78.2	78.4	78.6	78.5	79.0	79.1
12.0	76.9	77.1	77.4	77.6	77.8	78.0	78.2	78.4	78.6	78.8
.2	76.5	76.7	77.0	77.2	77.4	77.6	77.8	78.0	78.2	78.4
.4	76.1	76.3	76.6	76.8	77.0	77.2	77.5	77.7	77.9	78.1
.6	75.8	76.0	76.3	76.5	76.7	76.9	77.2	77.4	77.6	77.8
.8	75.4	75.6	75.9	76.1	76.3	76.5	76.8	77.0	77.2	77.4
13.0	75.0	75.2	75.5	75.7	75.9	76.1	76.4	76.6	76.8	77.0
.2	74.6	74.8	75.1	75.3	75.5	75.7	76.0	76.2	76.4	76.6
.4	74.2	74.4	74.7	74.9	75.1	75.3	75.6	75.8	76.0	76.2
.6	73.9	74.1	74.4	74.6	74.8	75.0	75.2	75.4	75.6	75.8
.8	73.5	73.7	74.0	74.2	74.4	74.6	74.8	75.0	75.2	75.4
14.0	73.1	73.3	73.6	73.8	74.1	74.3	74.6	74.8	75.0	75.2
.2	72.7	72.9	73.2	73.4	73.7	73.9	74.2	74.4	74.6	74.8
.4	72.3	72.5	72.9	73.1	73.3	73.6	73.8	74.0	74.2	74.4
.6	72.0	72.1	72.5	72.7	72.9	73.2	73.5	73.7	74.0	74.2
.8	71.6	71.8	72.1	72.3	72.6	72.8	73.2	73.4	73.6	73.8
15.0	71.2	71.4	71.7	71.9	72.2	72.5	72.8	73.0	73.2	73.4
.2	70.8	71.0	71.3	71.5	71.8	72.1	72.4	72.6	72.8	73.0
.4	70.4	70.6	70.9	71.1	71.4	71.8	72.1	72.3	72.5	72.7
.6	70.0	70.3	70.6	70.8	71.0	71.4	71.7	71.9	72.1	72.3
.8	69.6	69.9	70.2	70.4	70.7	71.0	71.3	71.5	71.7	71.9
16.0	69.2	69.5	69.8	70.1	70.4	70.7	70.9	71.2	71.4	71.7
.2	68.8	69.1	69.4	69.7	70.0	70.2	70.5	70.8	71.1	71.3
.4	68.4	68.7	69.1	69.4	69.6	69.9	70.2	70.4	70.7	70.9
.6	68.0	68.3	68.7	69.0	69.3	69.5	69.8	70.1	70.4	70.6
.8	67.7	68.0	68.3	68.6	68.9	69.2	69.4	69.7	70.0	70.3
17.0	67.3	67.6	67.9	68.2	68.5	68.8	69.1	69.4	69.7	69.9
.2	66.9	67.2	67.5	67.8	68.1	68.4	68.8	69.1	69.4	69.6
.4	66.5	66.8	67.1	67.4	67.7	68.0	68.4	68.7	69.1	69.3
.6	66.2	66.5	66.8	67.1	67.4	67.7	68.0	68.3	68.6	68.8
.8	65.8	66.1	66.4	66.7	67.0	67.3	67.7	68.0	68.3	68.5
18.0	65.4	65.7	66.0	66.4	66.7	67.0	67.3	67.6	67.9	68.2
.2	65.0	65.3	65.6	66.0	66.3	66.6	66.9	67.2	67.5	67.8
.4	64.6	64.9	65.2	65.6	65.9	66.2	66.6	66.9	67.2	67.5
.6	64.2	64.5	64.8	65.2	65.5	65.8	66.2	66.5	66.8	67.1
.8	63.8	64.1	64.4	64.8	65.1	65.4	65.8	66.1	66.4	66.7
19.0	63.4	63.8	64.2	64.5	64.8	65.1	65.4	65.8	66.1	66.4
.2	63.0	63.4	63.8	64.1	64.4	64.7	65.0	65.4	65.7	66.0
.4	62.6	63.0	63.4	63.7	64.0	64.3	64.7	65.1	65.4	65.7
.6	62.3	62.7	63.1	63.4	63.7	64.0	64.3	64.7	65.0	65.3
.8	61.9	62.3	62.7	63.0	63.3	63.6	64.0	64.4	64.7	65.0
20.0	61.5	61.9	62.3	62.6	63.0	63.3	63.6	64.0	64.3	64.6
.2	61.1	61.5	61.9	62.2	62.6	62.9	63.2	63.6	63.9	64.2
.4	60.7	61.1	61.5	61.8	62.2	62.5	62.9	63.3	63.6	63.9
.6	60.4	60.8	61.2	61.5	61.9	62.2	62.6	63.0	63.3	63.6
.8	60.0	60.4	60.8	61.1	61.5	61.8	62.2	62.6	62.9	63.2
21.0	59.6	60.0	60.4	60.8	61.1	61.5	61.8	62.2	62.5	62.8

TABLE 23 (*Continued*)

SHOWING THE EVAPORATION OF WATER IN CONCENTRATING JUICE TO SIRUP (SPENCER)

Brix of Juice	Degree Brix of the Sirup and the Evaporation in Percentage Terms of the Juice									
	57.0	57.5	58.0	58.5	59.0	59.5	60.0	60.5	61.0	61.5
11.0	80.7	80.9	81.0	81.2	81.4	81.5	81.7	81.8	82.0	82.1
.2	80.3	80.5	80.7	80.9	81.1	81.2	81.4	81.5	81.7	81.8
.4	80.0	80.2	80.4	80.5	80.7	80.8	81.0	81.2	81.3	81.5
.6	79.6	79.8	80.1	80.2	80.3	80.5	80.7	80.8	81.0	81.1
.8	79.3	79.5	79.7	79.9	80.0	80.2	80.3	80.5	80.7	80.8
12.0	79.0	79.2	79.3	79.5	79.7	79.9	80.0	80.2	80.4	80.5
.2	78.6	78.8	79.0	79.1	79.3	79.5	79.7	79.9	80.0	80.1
.4	78.3	78.5	78.6	78.8	78.9	79.2	79.3	79.5	79.7	79.8
.6	78.0	78.2	78.3	78.5	78.6	78.8	79.0	79.2	79.4	79.5
.8	77.6	77.8	77.9	78.1	78.3	78.5	78.7	78.8	79.0	79.2
13.0	77.2	77.4	77.6	77.8	78.0	78.2	78.3	78.5	78.7	78.9
.2	76.8	77.0	77.2	77.4	77.6	77.8	78.0	78.2	78.4	78.5
.4	76.4	76.6	76.9	77.1	77.3	77.5	77.7	77.9	78.0	78.2
.6	76.0	76.2	76.5	76.8	77.0	77.2	77.3	77.5	77.5	77.9
.8	75.7	75.8	76.2	76.4	76.6	76.8	77.0	77.2	77.4	77.5
14.0	75.4	75.6	75.9	76.1	76.3	76.5	76.7	76.9	77.0	77.2
.2	75.0	75.2	75.5	75.7	75.9	76.1	76.3	76.5	76.7	76.9
.4	74.6	74.8	75.2	75.4	75.6	75.8	76.0	76.2	76.4	76.6
.6	74.4	74.6	74.8	75.0	75.2	75.5	75.7	75.9	76.1	76.3
.8	74.0	74.2	74.5	74.7	74.9	75.1	75.3	75.7	75.7	75.9
15.0	73.7	73.9	74.2	74.4	74.6	74.8	75.0	75.2	75.4	75.6
.2	73.2	73.4	73.8	74.0	74.2	74.5	74.7	74.9	75.1	75.3
.4	72.9	73.1	73.5	73.7	73.9	74.1	74.3	74.5	74.8	74.9
.6	72.5	72.7	73.1	73.3	73.5	73.8	74.0	74.2	74.4	74.6
.8	72.1	72.3	72.8	73.0	73.2	73.5	73.7	73.9	74.1	74.3
16.0	71.9	72.1	72.4	72.6	72.9	73.1	73.3	73.6	73.8	74.0
.2	71.5	71.7	72.1	72.3	72.5	72.8	73.0	73.2	73.5	73.6
.4	71.1	71.3	71.7	72.0	72.2	72.4	72.7	72.9	73.1	73.3
.6	70.9	71.1	71.4	71.6	71.9	72.1	72.3	72.5	72.8	73.0
.8	70.5	70.8	71.0	71.3	71.5	71.8	72.0	72.2	72.5	72.7
17.0	70.2	70.4	70.7	71.0	71.2	71.5	71.7	71.9	72.1	72.4
.2	69.8	70.0	70.3	70.6	70.8	71.1	71.3	71.6	71.8	72.0
.4	69.5	69.7	70.0	70.3	70.5	70.8	71.0	71.2	71.5	71.7
.6	69.0	69.2	69.7	69.9	70.2	70.4	70.7	70.9	71.1	71.4
.8	68.7	68.9	69.3	69.6	69.8	70.1	70.3	70.6	70.8	71.1
18.0	68.4	68.7	69.0	69.2	69.5	69.8	70.0	70.3	70.5	70.7
.2	68.0	68.3	68.6	68.9	69.1	69.4	69.7	69.9	70.1	70.4
.4	67.7	68.0	68.3	68.5	68.8	69.1	69.3	69.6	69.8	70.1
.6	67.3	67.6	67.9	68.2	68.5	68.7	69.0	69.2	69.5	69.8
.8	67.0	67.3	67.6	67.9	68.1	68.4	68.7	68.9	69.1	69.4
19.0	66.7	67.0	67.2	67.5	67.8	68.1	68.3	68.6	68.8	69.1
.2	66.3	66.6	66.9	67.2	67.5	67.7	68.0	68.2	68.5	68.8
.4	66.0	66.3	66.5	66.8	67.1	67.4	67.7	67.9	68.2	68.5
.6	65.6	65.9	66.2	66.5	66.8	67.0	67.3	67.6	67.8	68.1
.8	65.3	65.6	65.9	66.2	66.4	66.7	67.0	67.3	67.5	67.8
20.0	64.9	65.2	65.5	65.8	66.1	66.4	66.7	67.0	67.2	67.5
.2	64.5	64.8	65.2	65.5	65.7	66.0	66.3	66.6	66.9	67.2
.4	64.2	64.5	64.8	65.1	65.4	65.7	66.0	66.3	66.6	66.8
.6	63.9	64.2	64.5	64.8	65.1	65.4	65.7	65.9	66.2	66.5
.8	63.5	63.8	64.1	64.4	64.7	65.1	65.3	65.6	65.9	66.2
21.0	63.2	63.5	63.8	64.1	64.4	64.7	65.0	65.3	65.6	65.8

TABLE 24

Showing the Evaporation of Water in Concentrating Juice to Sirup (Spencer)

(Percentages by volume, see p. 634)

Brix of Juice	Degree Brix of the Sirup and the Evaporation in Percentage Terms of the Juice																				
	53.0	53.5	54.0	54.5	55.0	55.5	56.0	56.5	57.0	57.5	58.0	58.5	59.0	59.5	60.0	60.5	61.0	61.5	62.0	62.5	63.0
10.0	84.3	84.5	84.7	84.9	85.0	85.2	85.3	85.5	85.7	85.9	86.0	86.2	86.3	86.5	86.6	86.7	86.8	86.9	87.1	87.3	87.4
.5	83.5	83.7	83.9	84.1	84.3	84.5	84.6	84.8	85.0	85.2	85.3	85.4	85.6	85.8	85.9	86.0	86.1	86.2	86.4	86.6	86.7
11.0	82.7	82.9	83.1	83.3	83.5	83.7	83.8	84.0	84.2	84.4	84.6	84.7	84.9	85.0	85.1	85.3	85.4	85.6	85.8	86.0	86.1
.5	81.9	82.1	82.3	82.5	82.7	82.9	83.0	83.3	83.5	83.7	83.9	84.0	84.1	84.3	84.4	84.6	84.7	84.9	85.1	85.3	85.4
12.0	81.0	81.2	81.4	81.6	81.9	82.1	82.2	82.5	82.7	82.9	83.1	83.3	83.4	83.6	83.7	83.9	84.1	84.3	84.4	84.6	84.7
.5	80.2	80.4	80.6	80.9	81.1	81.3	81.5	81.8	82.0	82.2	82.4	82.5	82.7	82.9	83.0	83.2	83.4	83.6	83.7	83.9	84.1
13.0	79.3	79.6	79.8	80.1	80.3	80.5	80.7	81.0	81.2	81.4	81.6	81.8	82.0	82.2	82.3	82.5	82.7	82.9	83.1	83.3	83.5
.5	78.5	78.8	79.0	79.3	79.5	79.7	79.9	80.2	80.4	80.6	80.8	81.1	81.3	81.5	81.6	81.8	82.0	82.2	82.4	82.6	82.8
14.0	77.7	77.9	78.1	78.4	78.7	78.9	79.1	79.4	79.6	79.8	80.0	80.3	80.5	80.7	80.9	81.1	81.3	81.5	81.7	82.0	82.2
.5	76.9	77.1	77.3	77.6	77.9	78.1	78.3	78.7	78.9	79.1	79.3	79.6	79.8	80.0	80.2	80.4	80.6	80.8	81.0	81.3	81.4
15.0	76.0	76.3	76.5	76.8	77.1	77.3	77.5	77.8	78.1	78.3	78.5	78.8	79.0	79.2	79.4	79.6	79.8	80.1	80.3	80.5	80.7
.5	75.2	75.5	75.7	76.0	76.3	76.5	76.7	77.0	77.3	77.5	77.7	78.1	78.3	78.5	78.7	78.9	79.1	79.4	79.6	79.8	80.0
16.0	74.3	74.6	74.9	75.2	75.4	75.6	75.9	76.2	76.5	76.8	77.0	77.3	77.5	77.8	78.0	78.2	78.4	78.7	78.9	79.1	79.3
.5	73.5	73.8	74.1	74.4	74.6	74.9	75.1	75.4	75.7	76.0	76.3	76.5	76.8	77.1	77.3	77.5	77.7	78.0	78.2	78.4	78.6
17.0	72.6	72.9	73.3	73.6	73.8	74.0	74.3	74.6	74.9	75.2	75.5	75.7	76.0	76.3	76.5	76.8	77.0	77.3	77.5	77.7	77.9
.5	71.8	72.1	72.4	72.7	73.0	73.3	73.5	73.8	74.1	74.4	74.7	75.0	75.3	75.6	75.8	76.1	76.3	76.6	76.8	77.0	77.2
18.0	70.9	71.2	71.5	71.8	72.1	72.4	72.7	73.0	73.3	73.6	73.9	74.2	74.5	74.8	75.1	75.4	75.6	75.8	76.0	76.3	76.5
.5	70.0	70.3	70.7	71.0	71.3	71.6	71.9	72.2	72.5	72.8	73.1	73.5	73.7	74.1	74.4	74.6	74.9	75.1	75.3	75.6	75.8
19.0	69.1	69.5	69.8	70.1	70.5	70.8	71.1	71.4	71.7	72.0	72.3	72.7	72.9	73.3	73.6	73.9	74.2	74.4	74.6	74.9	75.1
.5	68.2	68.6	68.9	69.3	69.6	69.9	70.3	70.5	70.9	71.2	71.5	71.9	72.2	72.6	72.9	73.1	73.4	73.7	73.9	74.2	74.4
20.0	67.3	67.7	68.0	68.4	68.8	69.0	69.4	69.7	70.1	70.5	70.8	71.1	71.4	71.8	72.1	72.4	72.7	73.0	73.2	73.5	73.7
.5	66.4	66.8	67.2	67.6	68.1	68.3	68.5	68.9	69.3	69.7	70.0	70.3	70.6	71.0	71.3	71.6	71.9	72.2	72.4	72.7	73.0
21.0	65.5	65.9	66.3	66.8	67.3	67.5	67.8	68.2	68.5	68.8	69.2	69.6	69.9	70.2	70.5	70.8	71.1	71.4	71.7	72.0	72.3
.5	64.6	65.0	65.4	65.9	66.5	66.8	67.1	67.4	67.7	68.0	68.4	68.8	69.1	69.4	69.7	70.0	70.3	70.6	70.9	71.2	71.5

TABLE 25

Table of Pounds of Water Evaporated per Cubic Foot of Sugar Solution at 20°/20° C. (F. M. Gibson) *

Brix After Evaporation	Brix of Solution Before Evaporation									
	10	11	12	13	14	15	16	17	18	19
51	52.10	51.03	49.96	48.87	47.78	46.67	45.56	44.44	43.31	42.17
52	52.35	51.31	50.25	49.19	48.13	47.04	45.96	44.87	43.76	42.65
53	52.58	51.57	40.54	49.50	48.46	47.41	46.35	45.28	44.20	43.12
54	52.81	51.82	50.81	49.80	48.78	47.75	46.72	45.68	44.62	43.57
55	53.03	52.06	51.07	50.08	49.10	48.09	47.08	46.06	45.02	43.99
56	53.23	52.29	51.33	50.37	49.39	48.41	47.42	46.42	45.42	44.41
57	53.23	52.29	51.33	50.37	49.39	48.41	47.42	46.42	45.42	44.81
58	53.64	52.73	51.81	50.89	49.96	49.02	48.07	47.12	46.16	45.19
59	53.82	52.94	52.04	51.14	50.23	49.31	48.38	47.46	46.51	45.57
60	54.01	53.14	52.26	51.38	50.49	49.59	48.68	47.78	46.85	45.92
61	54.19	53.34	52.48	51.61	50.75	49.86	48.98	48.08	47.18	46.27
62	54.36	53.58	52.69	51.84	50.99	50.13	49.25	48.58	47.50	46.61
63	54.52	53.71	52.88	52.06	51.23	50.38	49.53	48.68	47.81	46.94
64	54.68	53.88	53.08	52.27	51.45	50.62	49.79	48.96	48.10	47.26
65	54.84	54.06	53.27	52.47	51.67	50.86	50.04	49.23	48.40	47.56
66	54.99	54.22	53.45	52.67	51.89	51.09	50.30	49.49	48.68	47.86
67	55.13	54.39	53.63	52.87	52.10	51.32	50.54	49.75	48.95	48.15
68	55.28	54.54	53.80	53.05	52.30	51.53	50.77	49.99	49.21	48.43
69	55.42	54.70	53.97	53.23	52.50	51.75	50.99	50.23	49.47	48.70
70	55.55	54.85	54.13	53.41	52.69	51.95	51.21	50.47	49.72	48.97
71	55.68	54.99	54.29	53.58	52.87	52.15	51.43	50,70	49.96	49.22
72	55.81	55.13	54.44	53.74	53.06	52.35	51.64	50.92	50.20	49.47
73	55.93	55.26	54.59	53.91	53.23	52.53	51.84	51.13	50.42	49.72
74	56.05	55.40	54.74	54.07	53.40	52.72	52.04	51.35	50.65	49.95
75	56.17	55.52	54.88	54.22	53.56	52.90	52.23	51.55	50.87	50.19
76	56.28	55.65	55.01	54.37	53.73	53.07	52.41	51.75	51.08	50.41
77	56.39	55.77	55.15	54.52	53.89	53.24	52.59	51.94	51.28	50.62
78	56.50	55.90	55.28	54.66	54.04	53.41	52.77	52.13	51.48	50.84
79	56.60	56.01	55.41	54.79	54.19	53.56	52.95	52.31	51.68	51.05
80	56.71	56.12	55.53	54.93	54.33	53.72	53.11	52.49	51.87	51.25
81	56.81	56.23	55.65	55.06	54.48	53.87	53.28	52.67	52.06	51.44
82	56.90	56.34	55.77	55.19	54.62	54.03	53.44	52.84	52.24	51.64
83	57.00	56.45	55.88	55.32	54.75	54.17	53.59	53.01	52.41	51.83
84	57.10	56.55	55.99	55.44	54.88	54.31	53.74	53.17	52.59	52.01
85	57.19	56.65	56.11	55.56	55.01	54.45	53.90	53.33	52.75	52.19
86	57.27	56.75	56.22	55.67	55.14	54.59	54.04	53.48	52.92	52.36
87	57.36	56.85	56.31	55.79	55.26	54.72	54.18	53.63	53.08	52.53
88	57.45	56.94	56.42	55.90	55.38	54.85	54.32	53.78	53.24	52.70
89	57.53	57.03	56.52	56.01	55.50	54.98	54.45	53.93	53.40	52.86
90	57.61	57.12	56.62	56.12	55.61	55.10	54.59	54.07	53.54	53.02
91	57.69	57.20	56.71	56.22	55.73	55.22	54.72	54.21	53.69	53.18
92	57.77	57.29	56.81	56.32	55.84	55.34	54.84	54.34	53.83	53.33
93	57.84	57.37	56.90	56.42	55.95	55.45	54.97	54.47	53.98	53.48
94	57.91	57.46	56.99	56.52	56.05	55.57	55.09	54.60	54.11	53.63
95	57.99	57.53	57.08	56.61	56.15	55.68	55.21	54.73	54.25	53.77
96	58.06	57.61	57.16	56.71	56.26	55.79	55.33	54.85	54.38	53.91
97	58.13	57.69	57.25	56.80	56.36	55.90	53.44	54.97	54.51	54.04
98	58.20	57.77	57.33	56.89	56.45	56.00	55.55	55.09	54.63	54.18
99	58.26	57.84	57.41	56.98	56.55	56.10	55.66	55.21	54.76	54.31
100	58.33	57.91	57.49	57.06	56.64	56.20	55.77	55.33	54.88	54.44

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* *Sugar Industry Engineers Proceedings*, April, 1942. For method of use with Norris Tables see p. 717.

TABLE 25 (*Continued*)

TABLE OF POUNDS OF WATER EVAPORATED PER CUBIC FOOT OF SUGAR SOLUTION AT 20°/20° C. (F. M. GIBSON)

Brix After Evaporation	Brix of Solution Before Evaporation									
	20	21	22	23	24	25	26	27	28	29
51	41.01	39.86	38.69	37.51	36.32	35.12	33.91	32.69	31.46	30.22
52	41.53	40.40	39.25	38.10	36.95	35.77	34.59	33.40	32.19	30.99
53	42.01	40.91	39.80	38.67	37.54	36.39	35.24	34.08	32.91	31.72
54	42.49	41.41	40.32	39.22	38.12	36.99	35.87	34.73	33.59	32.44
55	42.94	41.89	40.82	39.75	38.67	37.58	36.48	35.37	34.25	33.12
56	43.38	42.35	41.31	40.26	39.20	38.14	37.06	35.98	34.88	33.78
57	43.80	42.80	41.78	40.75	39.72	38.67	37.63	36.56	35.49	34.41
58	44.21	43.22	42.23	41.22	40.22	39.20	38.17	37.13	36.08	35.03
59	44.60	43.64	42.67	41.69	40.70	39.70	38.69	37.68	36.65	35.63
60	44.99	44.04	43.09	42.13	41.17	40.18	39.20	38.21	37.20	36.20
61	45.35	44.43	43.50	42.56	41.62	40.66	39.70	38.72	37.74	36.75
62	45.71	44.81	43.90	42.97	42.05	41.11	40.17	39.22	38.26	37.29
63	46.06	45.18	44.28	43.38	42.47	41.55	40.63	39.70	38.76	37.81
64	46.39	45.53	44.65	43.77	42.88	41.98	41.07	40.16	39.24	38.32
65	46.72	45.87	45.01	44.15	43.28	42.39	41.51	40.61	39.71	38.81
66	47.03	46.20	45.36	44.51	43.66	42.79	41.93	41.05	40.17	39.28
67	47.34	46.52	45.70	44.87	44.03	43.19	42.33	41.47	40.61	39.74
68	47.63	46.84	46.03	45.21	44.40	43.57	42.73	41.88	41.03	40.18
69	47.92	47.14	46.35	45.55	44.75	43.93	43.11	42.29	41.45	40.61
70	48.20	47.43	46.67	45.87	45.08	44.29	43.49	42.68	41.86	41.03
71	48.47	47.72	46.95	46.19	45.42	44.63	43.85	43.05	42.25	41.44
72	48.73	47.99	47.25	46.50	45.74	44.96	44.20	43.42	42.63	41.84
73	48.99	48.27	47.53	46.79	46.05	45.30	44.54	43.77	43.00	42.23
74	49.25	48.53	47.81	47.09	46.36	45.62	44.87	44.13	43.36	42.60
75	49.48	48.79	48.08	47.37	46.65	45.93	45.20	44.46	43.72	42.97
76	49.72	49.04	48.34	47.65	46.94	46.23	45.51	44.79	44.06	43.33
77	49.96	49.28	48.60	47.91	47.22	46.52	45.82	45.11	44.40	43.68
78	50.18	49.52	48.85	48.17	47.50	46.81	46.12	45.42	44.72	44.01
79	50.39	49.75	49.09	48.43	47.77	47.09	46.41	45.73	45.04	44.34
80	50.61	49.97	49.33	48.68	48.03	47.36	46.70	46.02	45.34	44.66
81	50.82	50.19	49.56	48.92	48.28	47.63	46.97	46.32	45.64	44.98
82	51.02	50.41	49.78	49.16	48.53	47.89	47.24	46.59	45.94	45.28
83	51.22	50.62	50.00	49.39	48.77	38.14	47.51	46.87	46.22	45.58
84	51.41	50.82	50.22	49.61	49.01	48.39	47.77	47.14	46.51	45.88
85	51.60	51.02	50.43	49.83	49.23	48.63	48.02	47.41	46.78	46.16
86	51.78	51.21	50.64	50.05	49.46	48.86	48.27	47.66	47.05	46.44
87	51.97	51.40	50.83	50.26	49.68	49.09	48.50	47.91	47.31	46.71
88	52.14	51.59	51.03	50.46	49.90	49.32	48.74	48.16	47.56	46.98
89	52.32	51.77	51.22	50.67	50.11	49.54	48.97	48.39	47.81	47.23
90	52.49	51.95	51.41	50.86	50.31	49.75	49.19	48.63	48.06	47.49
91	52.65	52.12	51.59	51.06	50.52	49.97	49.42	48.86	48.29	47.73
92	52.81	52.29	51.77	51.24	50.71	50.17	49.63	49.08	48.53	47.98
93	52.96	52.46	51.94	51.42	50.90	50.37	49.84	49.30	48.76	48.22
94	53.12	52.62	52.12	51.60	51.09	50.57	50.04	49.52	48.98	48.45
95	53.28	52.78	52.28	51.78	51.28	50.76	50.25	49.73	49.20	48.67
96	53.42	52.94	52.45	51.95	51.46	50.95	50.45	49.94	49.42	48.89
97	53.57	53.09	52.61	52.12	51.64	51.14	50.64	50.13	49.62	49.11
98	53.71	53.24	52.76	52.29	51.81	51.32	50.83	50.33	49.83	49.33
99	53.85	53.39	52.92	52.45	51.98	51.50	51.01	50.53	50.03	49.54
100	53.98	53.53	53.07	52.61	52.14	51.67	51.19	50.71	50.23	49.74

TABLE 25 (*Continued*)

TABLE OF POUNDS OF WATER EVAPORATED PER CUBIC FOOT OF SUGAR SOLUTION AT 20°/20° C. (F. M. GIBSON)

Brix After Evaporation	Brix of Solution Before Evaporation									
	50	51	52	53	54	55	56	57	58	59
81	29.38	28.56	27.73	26.90	26.05	25.20	24.34	23.47	22.59	21.71
82	29.95	29.14	28.34	27.52	26.69	25.85	25.01	24.15	23.29	22.43
83	30.52	29.72	28.93	28.12	27.31	26.48	25.65	24.82	23.97	23.12
84	31.07	30.29	29.51	28.72	27.91	27.10	26.28	25.46	24.63	23.79
85	31.61	30.84	30.07	29.29	28.50	27.71	26.91	26.09	25.27	24.46
86	32.13	31.38	30.62	29.85	29.08	28.30	27.51	26.71	25.91	25.10
87	32.65	31.90	31.16	30.40	29.64	28.88	28.10	27.31	26.52	25.73
88	33.14	32.42	31.68	30.94	30.20	29.44	28.67	27.91	27.13	26.34
89	33.64	32.92	32.20	31.47	30.74	29.99	29.24	28.49	27.72	26.95
90	34.11	33.41	32.70	31.98	31.26	30.53	29.79	29.05	28.30	27.53
91	34.58	33.89	33.29	32.49	31.78	31.06	30.33	29.60	28.86	28.11
92	35.04	34.36	33.68	32.98	32.28	31.58	30.86	30.14	29.41	28.68
93	35.49	34.82	34.15	33.46	32.78	32.08	31.38	30.67	29.95	29.23
94	35.93	35.27	34.60	33.94	33.25	32.57	31.88	31.18	30.48	29.77
95	36.36	35.71	35.05	34.40	33.73	33.06	32.37	31.69	30.99	30.29
96	36.78	36.14	35.50	34.85	34.19	33.53	32.86	32.19	31.50	30.81
97	37.19	36.56	35.93	35.29	34.64	33.99	33.33	32.67	32.00	31.32
98	37.60	36.98	36.35	35.73	35.09	34.45	33.80	33.15	32.48	31.82
99	37.99	37.38	36.77	36.15	35.52	34.89	34.25	33.61	32.95	32.30
100	38.38	37.78	37.18	36.57	35.95	35.33	34.70	34.06	33.42	32.78

Brix After Evaporation	Brix of Solution Before Evaporation									
	60	61	62	63	64	65	66	67	68	69
81	20.82	19.92	19.01	18.09	17.17	16.23	15.29	14.33	13.37	12.40
82	21.55	20.66	19.77	18.86	17.96	17.04	16.11	15.17	14.22	13.27
83	22.25	21.39	20.51	19.62	18.72	17.82	16.91	15.99	15.06	14.12
84	22.94	22.09	21.23	20.35	19.48	18.59	17.69	16.79	15.87	14.95
85	23.62	22.78	21.93	21.07	20.21	19.34	18.45	17.57	16.66	15.75
86	24.28	23.45	22.62	21.77	20.92	20.07	19.20	18.32	17.44	16.55
87	24.92	24.11	23.29	22.46	21.63	20.78	19.93	19.07	18.20	17.32
88	25.55	24.75	23.95	23.13	22.31	21.48	20.64	19.79	18.94	18.07
89	26.16	25.38	24.59	23.78	22.98	22.16	21.33	20.50	19.66	18.81
90	26.77	25.99	25.21	24.43	23.63	22.83	22.02	21.20	20.36	19.53
91	27.36	26.60	25.83	25.05	24.27	23.48	22.68	21.87	21.05	20.24
92	27.93	27.19	26.43	25.66	24.89	24.12	23.33	22.53	21.74	20.93
93	28.49	27.76	27.01	26.27	25.51	24.74	23.97	23.19	22.40	21.61
94	29.05	28.33	27.59	26.85	26.10	25.35	24.59	23.82	23.05	22.27
95	29.59	28.88	28.16	27.42	26.69	25.95	25.21	24.44	23.68	22.91
96	30.12	29.42	28.71	27.99	27.26	26.54	25.80	25.06	24.30	23.55
97	30.63	29.94	29.24	28.54	27.83	27.11	26.39	25.65	24.91	24.17
98	31.14	30.46	29.77	29.07	28.38	27.67	26.96	26.23	25.50	24.77
99	31.63	30.96	30.29	29.60	28.92	28.22	27.52	26.81	26.09	25.36
100	32.12	31.47	30.80	30.13	29.45	28.76	28.07	27.37	26.66	25.95

TABLE 26

CONDENSER WATER

Weight per unit weight of water evaporated from juice
R. S. Norris' tables extended
For Single Effects (Vacuum pans)

Temperature ° C. of Injection-water	Temperature of Water Coming Out of Condenser in ° C.															
	30	32	34	36	38	40	42	44	46	48	50	52	54	56	58	60
5	24.7	22.8	21.3	19.9	18.7	17.6	16.7	15.8	15.0	14.3	13.7	13.1	12.6	12.1	11.6	11.2
6	25.6	23.7	22.0	20.0	19.2	18.1	17.1	16.2	15.4	14.7	14.0	13.4	12.8	12.3	11.8	11.4
7	26.7	24.6	22.8	21.2	19.8	18.6	17.6	16.6	15.8	15.0	14.3	13.7	13.1	12.5	12.0	11.6
8	27.9	25.6	23.6	21.9	20.4	19.2	18.0	17.0	16.1	15.3	14.6	13.9	13.3	12.8	12.3	11.8
9	29.2	26.8	24.5	22.7	21.1	19.8	18.6	17.5	16.6	15.7	14.9	14.2	13.6	13.0	12.5	12.0
10	30.6	27.8	25.5	23.5	21.8	20.4	19.1	18.0	17.0	16.1	15.3	14.6	13.9	13.3	12.7	12.2
11	32.1	29.1	26.5	24.4	22.6	21.0	19.7	18.5	17.4	16.5	15.7	14.9	14.2	13.6	13.0	12.5
12	33.9	30.5	27.7	25.4	23.4	21.8	20.3	19.0	17.9	16.9	16.0	15.2	14.5	13.9	13.3	12.7
13	35.8	32.0	29.0	26.5	24.3	22.5	21.0	19.6	18.4	17.4	16.4	15.6	14.8	14.2	13.5	12.9
14	38.0	33.7	30.4	27.6	25.3	23.4	21.7	20.2	19.0	17.9	16.9	16.0	15.2	14.5	13.8	13.2
15	40.4	35.7	31.9	28.9	26.4	24.2	22.5	20.9	19.6	18.4	17.3	16.4	15.6	14.8	14.1	13.5
16	43.2	37.8	33.6	30.3	27.5	25.2	23.3	21.6	20.2	18.9	17.8	16.8	15.9	15.1	14.4	13.8
17	46.5	40.3	35.6	31.8	28.8	26.3	24.2	22.4	20.8	19.5	18.3	17.3	16.3	15.5	14.7	14.1
18	50.3	43.1	37.8	33.5	30.2	27.4	25.1	23.2	21.6	20.1	18.9	17.7	16.8	15.9	15.1	14.4
19	54.8	46.3	40.2	35.4	31.7	28.7	26.2	24.1	22.3	20.8	19.4	18.3	17.2	16.3	15.4	14.7
20	60.1	50.1	43.0	37.6	33.4	30.1	27.3	25.1	23.1	21.5	20.0	18.8	17.7	16.7	15.8	15.0
21	66.7	54.6	46.2	40.0	35.3	31.6	28.6	26.1	24.0	22.2	20.7	19.3	18.2	17.2	16.2	15.4
22	74.9	59.9	49.9	42.8	37.5	33.3	30.0	27.2	25.0	23.1	21.4	20.0	18.7	17.6	16.7	15.8
23	85.5	66.5	54.4	46.0	39.9	35.2	31.5	28.5	26.0	23.9	22.2	20.6	19.3	18.1	17.1	16.2
24	99.6	74.7	59.7	49.8	42.7	37.3	33.2	29.9	27.2	24.9	23.0	21.3	19.9	18.7	17.6	16.6
25	119.3	85.2	66.3	53.2	45.9	39.8	35.1	31.4	28.4	25.9	23.9	22.1	20.6	19.2	18.1	17.0
26	148.8	98.2	74.4	59.5	49.6	42.5	37.2	33.1	29.8	27.1	24.8	22.9	21.3	19.8	18.6	17.5
27	198.1	118.9	84.9	65.8	54.0	45.7	39.6	35.0	31.3	28.3	25.8	23.8	22.0	20.5	19.2	18.0
28	......	148.3	98.9	74.2	59.3	49.4	42.4	37.1	33.0	29.7	27.0	24.7	22.8	21.2	19.8	18.5
29	......	197.4	118.5	84.6	65.6	53.9	45.6	39.5	34.8	31.2	28.2	25.8	23.7	21.9	20.4	19.1

30	......	......	147.8	98.6	73.9	59.1	49.3	42.2	37.0	32.9	29.6	26.9	24.6	22.7	21.1	19.7
31	......	......	195.8	118.1	84.4	65.6	53.7	45.4	39.3	34.7	31.1	28.2	25.7	23.6	21.9	20.3
32	......	......	......	147.3	98.2	73.7	58.9	49.2	42 1	36.8	32.7	29.5	26.8	24.5	22.7	21.0
33	......	......	......	196.1	117.6	84.1	65.2	53.4	45 3	39.2	34.6	31.0	28.0	25.6	23.5	21.8
34	......	......	......	......	146.8	97.9	73.4	58.7	48.9	41.9	36.7	32.6	29.4	26.7	24.4	22.6
35	......	......	......	......	195.6	117.2	83.8	65.1	53.3	45.1	39.1	34.5	30.9	27.9	25.5	23.4
36	......	......	......	......	......	146.2	97.6	73.2	58.5	48.7	41.8	36.6	32.5	29.3	26.6	24.4
37	......	......	......	......	......	194.8	116.8	83.5	64.9	53.1	44.9	38 9	34.4	30.7	27.8	25.4
38	......	......	......	......	......	......	145.8	97.2	72.9	58.3	48.6	41.7	36.5	32.4	29.2	26.5
39	......	......	......	......	......	......	194.1	116.5	83.2	64.7	52.9	44.8	38.8	34.2	30.6	27.7
40	......	......	......	......	......	......	......	145.3	96.9	72.7	58.1	48.5	41.5	36.6	32.3	29.1
41	......	......	......	......	......	......	......	193.5	116 0	82.9	64.5	52.8	44.6	38.7	34.1	30.5
42	......	......	......	......	......	......	......	......	144.7	96 5	72 4	57.9	48.3	41.4	36.2	32.2
43	......	......	......	......	......	......	......	......	192.7	115.5	82.7	64.3	52.6	44.5	38.5	34.0
44	......	......	......	......	......	......	......	......	......	144 3	96.3	72.2	57.7	48.1	41.2	36.1
45	......	......	......	......	......	......	......	......	......	192.1	115.3	82.4	64.1	52.4	44.3	38.4
46	......	......	......	......	......	......	......	......	......	......	143.8	96 0	71 9	57.5	47.9	41.1
47	......	......	......	......	......	......	......	......	......	......	191.4	114.9	82.1	63.8	52.2	44.2
48												143 4	95 6	71.7	57.3	47.8
49												190 8	114.5	81.4	63.6	52.0
50												......	142.9	95.2	71.4	57.1
51												......	190.1	114.0	81.5	63.4
52												......	......	142.2	94.9	71.2
53												......	......	189.4	113.5	81.2
54												......	......	......	141.8	94.6

CALCULATION OF THE CONDENSER-WATER

To calculate the condenser-water (W) for other pressures than 25 inches (basis of the tables) and for other evaporator combinations than single-, triple- and quadruple-effect, use the following formula, factors and constants:

$$W=\frac{\text{Constant}-\text{T. }^\circ\text{ C. of condenser-water}}{\text{T. }^\circ\text{ C. of condenser-water}-\text{T. }^\circ\text{ C. of injection-water}}+1.$$

The value of W must be multiplied by the factor appropriate to the evaporator combination. (*Continued on p.* 714.)

TABLE 26 (*Continued*)

CONDENSER WATER

For Triple Effects

Temperature ° C. of Injection-water	Temperature of Water Coming Out of Condensers in ° C.															
	30	32	34	36	38	40	42	44	46	48	50	52	54	56	58	60
5	9.1	8.4	7.9	7.4	6.5	6.5	6.2	5.8	5.6	5.3	4.9	4.9	4.7	4.5	4.3	4.2
6	9.5	8.7	8.1	7.6	7.1	6.7	6.3	6.0	5.7	5.4	5.2	5.0	4.7	4.6	4.4	4.2
7	9.9	9.1	8.4	7.8	7.3	6.9	6.5	6.1	5.8	5.5	5.3	5.1	4.8	4.6	4.5	4.3
8	10.3	9.5	8.7	8.1	7.6	7.1	6.7	6.3	6.0	5.7	5.4	5.2	4.9	4.7	4.5	4.4
9	10.8	9.9	9.1	8.4	7.8	7.3	6.9	6.5	6.1	5.8	5.5	5.3	5.0	4.8	4.6	4.4
10	11.3	10.3	9.4	8.7	8.1	7.5	7.1	6.7	6.3	6.0	5.7	5.4	5.1	4.9	4.7	4.5
11	11.9	10.8	9.8	9.0	8.4	7.8	7.3	6.8	6.5	6.1	5.8	5.5	5.3	5.0	4.8	4.6
12	12.5	11.3	10.2	9.4	8.7	8.1	7.5	7.0	6.6	6.3	5.9	5.6	5.4	5.1	4.9	4.7
13	13.2	11.8	10.7	9.8	9.0	8.3	7.8	7.3	6.8	6.4	6.1	5.8	5.5	5.2	5.0	4.8
14	14.0	12.5	11.2	10.2	9.4	8.6	8.0	7.5	7.0	6.6	6.2	5.9	5.6	5.4	5.1	4.9
15	15.0	13.2	11.8	10.7	9.8	9.0	8.3	7.7	7.2	6.8	6.4	6.1	5.8	5.5	5.2	5.0
16	16.0	14.0	12.4	11.2	10.2	9.3	8.6	8.0	7.5	7.0	6.6	6.2	5.9	5.6	5.3	5.1
17	17.2	14.9	13.2	11.8	10.6	9.7	8.9	8.3	7.7	7.2	6.8	6.4	6.0	5.7	5.5	5.2
18	18.6	15.9	14.0	12.4	11.2	10.1	9.3	8.6	8.0	7.4	7.0	6.6	6.2	5.9	5.6	5.3
19	20.3	17.1	14.9	13.1	11.7	10.6	9.7	8.9	8.3	7.7	7.2	6.8	6.4	6.0	5.7	5.4
20	22.2	18.5	15.9	13.9	12.4	11.1	10.1	9.3	8.6	8.0	7.4	7.0	6.5	6.2	5.9	5.6
21	24.7	20.2	17.1	14.8	13.1	11.7	10.6	9.7	8.9	8.2	7.7	7.2	6.7	6.4	6.0	5.7
22	27.7	22.2	18.5	15.8	13.9	12.3	11.1	10.1	9.2	8.5	7.9	7.4	6.9	6.5	6.2	5.8
23	31.6	24.6	20.1	17.0	14.8	13.0	11.7	10.5	9.6	8.9	8.2	7.6	7.2	6.7	6.3	6.0
24	36.8	27.6	22.1	18.4	15.8	13.8	12.3	11.1	10.1	9.2	8.5	7.9	7.4	6.9	6.5	6.1
25	44.1	31.5	24.5	20.1	17.0	14.7	13.0	11.6	10.5	9.6	8.8	8.2	7.6	7.1	6.7	6.3
26	55.1	36.7	27.5	22.0	18.4	15.7	13.8	12.2	11.0	10.0	9.2	8.5	7.9	7.4	6.9	6.5
27	73.3	44.0	31.4	24.4	20.0	16.9	14.7	12.9	11.6	10.5	9.6	8.8	8.2	7.6	7.1	6.9
28	.....	54.9	36.6	27.4	22.0	18.3	15.7	13.7	12.2	11.0	10.0	9.2	8.6	7.9	7.3	6.9
29	......	......	43.8	31.3	24.3	19.9	16.9	14.6	12.9	11.5	10.4	9.5	8.8	8.1	7.6	7.1

30	......	......	54.7	36.5	27.4	21.9	18 2	15.6	13.7	12.2	11.0	10.0	9.1	8.4	7.8	7.2
31	......	......	72.9	43.7	31.2	24.3	19.9	16.8	14.5	12.8	11.5	10.4	9.5	8.7	8.1	7.5
32				54.9	36.3	27.3	21.8	18.2	15.6	13.6	12.1	10.9	9.9	9.1	8.4	7.8
33				72.6	43.5	31.1	24.1	19.8	16.8	14.5	12.8	11.5	10.3	9.5	8.7	8.1
34				......	54.3	36.2	27.1	21.7	18.1	15.3	13.6	12.0	10.9	9.9	9.0	8.4
35				......	72.3	43.4	30 9	24.1	19.7	16.8	14.5	12.7	11.4	10.3	9.4	8.7
36				......	......	54.1	36.1	27.1	21 6	18.0	15 4	13.5	12.0	10.8	9.8	9.0
37				......	......	72.1	43.3	30.9	24.0	19.6	16.6	14.4	12.7	11.3	10.3	9.4
38				......	......	......	53.9	35.9	27.0	21.5	18.0	15.4	13.5	11.9	10.8	9.8
39				......	......	......	71.8	43 1	30.8	23.9	19 5	16.5	14.3	12.6	11.3	10.3
40				......	......	......	......	53.8	35.8	26.9	21 5	17.9	15.3	13.5	11.9	10.8
41				......	......	......	......	71.6	42 9	30.7	23.8	19.5	16.5	14.3	12.6	11.2
42				......	......	......	......	......	53.6	35.7	26.8	21 4	17.8	15.3	13.4	11.8
43				......	......	......	......	......	71.4	42.8	30.6	23.8	19.5	16.5	14.2	12.5
44					......	......	......	......	......	53.4	35 6	26.7	21.3	17.8	15.2	13.3
45					......	......	......	......	......	71.1	42.7	30.5	23.7	19.4	16.4	14.2
46					......	......	......	......	......	......	53.3	35.5	26.6	21.2	17.7	15.1
47					......	......	......	......	......	......	70.9	42.5	30.4	23.6	19.3	16.3
48					......	......	......	......	......	......	......	53.1	35 3	26.5	21.2	17.6
49					......	......	......	......	......	......	......	70.7	42.4	30.3	23.5	19.2
50													52.9	35.2	26.4	21.1
51													70.4	42.2	30.2	23.4
52													......	52.6	35.1	26.3
53													......	70.1	42.1	30.0
54													......	......	52.4	35.0

Constants

Inches	Calories
22	625.9
23	624 4
24	623.1
25	621.3
26	619.5
27	616.8
28	613.4

Factors

Single effect	1.000
Double effect	0.534
Triple effect	0.370
Quadruple effect	0.284
Quintuple effect	0.235

METHOD OF USING NORRIS' TABLES

Calculate the evaporation from clarified juice to sirup (E) by the following formula, in which B is the Brix of the juice and b that of the sirup:

$$E = (B - b) \div B \times 100, \text{ in terms of the juice}$$

The next stages of the calculations are best illustrated by an example in quadruple-effect evaporation (continued on page 716):

TABLE 26 (*Continued*)

CONDENSER WATER

For Quadruple Effects

Temperature ° C. of Injection-water	Temperature of Water Coming Out of Condenser in ° C.															
	30	32	34	36	38	40	42	44	46	48	50	52	54	56	58	60
5	7.0	6.5	6.0	5.6	5.3	5.0	4.7	4.5	4.3	4.1	3.9	3.7	3.6	3.4	3.3	3.2
6	7.3	6.7	6.2	5.8	5.5	5.1	4.9	4.6	4.4	4.2	4.0	3.8	3.6	3.5	3.4	3.2
7	7.6	7.0	6.5	6.0	5.6	5.3	5.0	4.7	4.5	4.3	4.1	3.9	3.7	3.6	3.4	3.3
8	7.9	7.3	6.7	6.2	5.8	5.4	5.1	4.8	4.6	4.4	4.1	4.0	3.8	3.6	3.5	3.3
9	8.3	7.6	7.0	6.4	6.0	5.6	5.3	5.0	4.7	4.5	4.2	4.0	3.9	3.7	3.6	3.4
10	8.7	7.9	7.2	6.7	6.2	5.8	5.4	5.1	4.8	4.6	4.3	4.1	3.9	3.8	3.6	3.5
11	9.1	8.3	7.5	6.9	6.4	6.0	5.6	5.3	5.0	4.7	4.4	4.2	4.0	3.9	3.7	3.5
12	9.3	8.7	7.9	7.2	6.7	6.2	5.8	5.4	5.1	4.8	4.6	4.3	4.1	3.9	3.8	3.6
13	10.2	9.1	8.2	7.5	6.9	6.4	6.0	5.6	5.2	4.9	4.7	4.4	4.2	4.0	3.8	3.7
14	10.8	9.6	8.6	7.8	7.2	6.6	6.2	5.7	5.4	5.1	4.8	4.5	4.3	4.1	3.9	3.7
15	11.5	10.1	9.1	8.2	7.5	6.9	6.4	5.9	5.6	5.2	4.9	4.7	4.4	4.2	4.0	3.8
16	12.3	10.7	9.6	8.6	7.8	7.2	6.6	6.1	5.7	5.4	5.1	4.8	4.5	4.3	4.1	3.9
17	13.2	11.4	10.1	9.0	8.2	7.5	6.9	6.4	5.9	5.5	5.2	4.9	4.6	4.4	4.2	4.0
18	14.3	12.2	10.7	9.5	8.6	7.8	7.1	6.6	6.1	5.7	5.4	5.0	4.8	4.5	4.3	4.1
19	15.6	13.2	11.4	10.1	9.0	8.1	7.4	6.8	6.3	5.9	5.5	5.2	4.9	4.6	4.4	4.2
20	17.1	14.2	12.2	10.7	9.5	8.5	7.8	7.1	6.6	6.1	5.7	5.3	5.0	4.7	4.5	4.3
21	18.9	15.5	13.1	11.4	10.0	9.0	8.1	7.4	6.8	6.3	5.9	5.5	5.2	4.9	4.6	4.4
22	21.3	17.0	14.2	12.2	10.6	9.5	8.5	7.7	7.1	6.5	6.1	5.7	5.3	5.0	4.7	4.5
23	24.3	18.9	15.4	13.1	11.3	10.0	8.9	8.1	7.4	6.8	6.3	5.9	5.5	5.1	4.9	4.6
24	28.3	21.2	17.0	14.1	12.1	10.6	9.4	8.5	7.7	7.1	6.5	6.1	5.7	5.3	5.0	4.7
25	33.9	24.2	18.8	15.4	13.0	11.3	10.0	8.9	8.1	7.4	6.8	6.3	5.8	5.5	5.1	4.8
26	42.3	28.2	21.1	16.9	14.1	12.1	10.6	9.4	8.5	7.7	7.0	6.5	6.0	5.6	5.3	5.0
27	56.3	33.8	24.1	18.7	15.3	13.0	11.3	9.9	8.9	8.0	7.3	6.8	6.3	5.8	5.4	5.1
28	......	42.2	28.1	21.1	16.9	14.0	12.0	10.5	9.4	8.4	7.7	7.0	6.5	6.0	5.6	5.3
29	......	......	33.6	24.0	18.6	15.3	12.9	11.2	9.9	8.9	8.0	7.3	6.7	6.2	5.8	5.4

30	......	......	42.0	28.0	21.0	16.8	14.0	12.0	10.5	9.3	8.4	7.6	7.0	6.5	6.0	5.6
31	......	......	56.0	33.6	24.0	18.6	15.3	12.9	11.1	9.9	8.8	8.0	7.3	6.7	6.2	5.8
32	......	......	......	41.9	27.9	20.9	16.7	13.9	11.9	10.4	9.3	8.4	7.6	7.0	6.5	6.0
33	......	......	......	55.3	33.4	23.9	18.5	15.2	12.8	11.1	9.8	8.8	7.9	7.3	6.7	6.2
34	......	......	......	......	41.7	27.9	20.8	16.7	13.9	11.9	10.4	9.3	8.4	7.6	7.0	6.5
35	......	......	......	......	55.3	33.3	23.7	18.5	15.1	12.8	11.1	9.8	8.8	7.9	7.3	6.7
36	......	......	......	......	......	41.5	27.7	20.8	16.6	13.8	11.9	10.4	9.2	8.3	7.6	7.0
37	......	......	......	......	......	55.2	33.2	23.7	18.4	15.1	12.7	11.0	9.8	8.7	7.9	7.3
38	......	......	......	......	......	......	41.4	27.6	20.7	16.5	13.8	11.8	10.4	9.2	8.3	7.6
39	......	......	......	......	......	......	55.1	33.1	23.6	18.4	15.0	12.7	11.0	9.7	8.7	7.9
40	......	......	......	......	......	......	......	41.4	27.5	20.5	16.5	13.8	11.8	10.4	9.2	8.3
41	......	......	......	......	......	......	......	55.0	32.9	23.5	18.3	15.0	12.6	11.0	9.7	8.7
42								......	41.1	27.4	20.5	16.4	13.7	11.9	10.3	9.2
43								......	54.8	32.8	23.5	18.2	14.9	12.6	10.9	9.7
44								......	......	41.0	27.3	20.5	16.4	13.6	11.7	10.2
45								......	......	54.6	32.7	23.4	18.2	14.9	12.6	10.8
46								......	......	......	40.9	27.2	20.4	16.3	13.6	11.7
47								......	......	......	54.4	32.6	23.3	18.1	14.8	12.5
48								......	......	......	......	40.7	27.1	20.3	16.3	13.5
49								......	......	......	......	54.2	32.5	23.1	18.0	14.7
50								......	......	......	......	......	40.6	27.0	20.2	16.2
51								......	......	......	......	......	54.0	32.4	23.1	18.0
52								......	......	......	......	......	......	40.4	26.9	20.2
53								......	......	......	......	......	......	53.8	32.2	23.0
54								......	......	......	......	......	......	......	40.3	26.8

Weight of mixed juice.............1500 tons
Temperature of injection-water.... 35° C.
Temperature of condenser-water.... 50° C.
Brix of the juice.................. 15
Brix of the sirup.................. 60

$E = [(60-15) \div 60] \times 100 = 75$ per cent of weight of juice; $1500 \times .75 = 1125$ tons of water evaporated; referring to the third section of the table, opposite 35° and under 50° we have 11.1, the condenser water per unit of water evaporated; $1125 \times 11.1 = 12{,}488$ tons of condenser-water.

Note. **Gibson's table 25, p. 709, pounds of water evaporated per cubic foot of primary material may be used in conjunction with Norris' tables given in the preceding pages. For example, a strike of 1000 cu. ft. of sirup (or liquor) of 60° Brix is evaporated in a vacuum pan to a massecuite of 92° Brix. From Gibson's table, p. 711, the pounds of water evaporated per cubic foot is 27.93 lb. With the water temperatures given above we have (Table 26) 39.1 lb. condenser water per pound of evaporation. Then 39.1 × 27.93 = 920.6 lb. condenser water per cu. ft. × 1000 = 920,600 lb. condenser water per strike. Similarly Gibson's table may be used in determining the pounds of condenser water needed in evaporating a given number of cubic feet of juice or sweet water in multiple effect.**

TABLE 27

DEGREES BRIX, SPECIFIC GRAVITY, AND DEGREES BAUMÉ OF SUGAR SOLUTIONS[1] AT 20° C.

Degrees Brix or Per Cent Sucrose by Weight	Specific Gravity at 20°/4° C.	Specific Gravity at 20°/20° C.	Degrees Baumé (Modulus 145)	Degrees Brix or Per Cent Sucrose by Weight	Specific Gravity at 20°/4° C.	Specific Gravity at 20°/20° C.	Degrees Baumé (Modulus 145)
0.0	0.99823	1.00000	0.00	**5.0**	1.01785	1.01965	2.79
.1	0.99862	1.00039	0.06	.1	1.01825	1.02005	2.85
.2	0.99901	1.00078	0.11	.2	1.01865	1.02045	2.91
.3	0.99940	1.00117	0.17	.3	1.01905	1.02085	2.96
.4	0.99979	1.00155	0.22	.4	1.01945	1.02125	3.02
0.5	1.00017	1.00194	0.28	**5.5**	1.01985	1.02165	3.07
.6	1.00056	1.00233	0.34	.6	1.02025	1.02206	3.13
.7	1.00095	1.00272	0.39	.7	1.02065	1.02246	3.18
.8	1.00134	1.00311	0.45	.8	1.02105	1.02286	3.24
.9	1.00173	1.00350	0.51	.9	1.02145	1.02321	3.30
1.0	1.00212	1.00389	0.56	**6.0**	1.02186	1.02366	3.35
.1	1.00251	1.00428	0.62	.1	1.02226	1.02407	3.41
.2	1.00290	1.00467	0.67	.2	1.02266	1.02447	3.46
.3	1.00329	1.00506	0.73	.3	1.02306	1.02487	3.52
.4	1.00368	1.00545	0.79	.4	1.02346	1.02527	3.57
1.5	1.00406	1.00584	0.84	**6.5**	1.02387	1.02568	3.63
.6	1.00445	1.00623	0.90	.6	1.02427	1.02608	3.69
.7	1.00484	1.00662	0.95	.7	1.02467	1.02648	3.74
.8	1.00523	1.00701	1.01	.8	1.02508	1.02689	3.80
.9	1.00562	1.00740	1.07	.9	1.02548	1.02729	3.85
2.0	1.00602	1.00779	1.12	**7.0**	1.02588	1.02770	3.91
.1	1.00641	1.00818	1.18	.1	1.02629	1.02810	3.96
.2	1.00680	1.00853	1.23	.2	1.02669	1.02851	4.02
.3	1.00719	1.00897	1.29	.3	1.02710	1.02892	4.08
.4	1.00758	1.00936	1.34	.4	1.02750	1.02932	4.13
2.5	1.00797	1.00976	1.40	**7.5**	1.02791	1.02973	4.19
.6	1.00836	1.01015	1.46	.6	1.02832	1.03013	4.24
.7	1.00876	1.01054	1.51	.7	1.02872	1.03054	4.30
.8	1.00915	1.01093	1.57	.8	1.02913	1.03095	4.35
.9	1.00954	1.01133	1.62	.9	1.02954	1.03136	4.41
3.0	1.00993	1.01172	1.68	**8.0**	1.02994	1.03176	4.46
.1	1.01033	1.01211	1.74	.1	1.03035	1.03217	4.52
.2	1.01072	1.01251	1.79	.2	1.03076	1.03258	4.58
.3	1.01112	1.01290	1.85	.3	1.03116	1.03299	4.63
.4	1.01151	1.01330	1.90	.4	1.03157	1.03340	4.69
3.5	1.01190	1.01369	1.96	**8.5**	1.03198	1.03381	4.74
.6	1.01230	1.01409	2.02	.6	1.03239	1.03422	4.80
.7	1.01269	1.01448	2.07	.7	1.03280	1.03463	4.85
.8	1.01309	1.01488	2.13	.8	1.03321	1.03504	4.91
.9	1.01348	1.01528	2.18	.9	1.03362	1.03545	4.96
4.0	1.01388	1.01567	2.24	**9.0**	1.03403	1.03586	5.02
.1	1.01428	1.01607	2.29	.1	1.03444	1.03627	5.07
.2	1.01467	1.01647	2.35	.2	1.03485	1.03668	5.13
.3	1.01507	1.01687	2.40	.3	1.03526	1.03709	5.19
.4	1.01547	1.01726	2.46	.4	1.03567	1.03750	5.24
4.5	1.01586	1.01766	2.52	**9.5**	1.03608	1.03792	5.30
.6	1.01626	1.01806	2.57	.6	1.03649	1.03833	5.35
.7	1.01666	1.01846	2.63	.7	1.03691	1.03874	5.41
.8	1.01706	1.01886	2.68	.8	1.03732	1.03915	5.46
.9	1.01746	1.01926	2.74	.9	1.03773	1.03957	5.52

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[1] **From Bureau Standards Circular 44 (1918).**

TABLE 27 (*Continued*)

DEGREES BRIX, SPECIFIC GRAVITY, AND DEGREES BAUMÉ OF SUGAR SOLUTIONS AT 20° C.

Degrees Brix or Per Cent Sucrose by Weight	Specific Gravity at 20°/4° C.	Specific Gravity at 20°/20° C.	Degrees Baumé (Modulus 145)	Degrees Brix or Per Cent Sucrose by Weight	Specific Gravity at 20°/4° C.	Specific Gravity at 20°/20° C.	Degrees Baumé (Modulus 145)
10.0	1.03814	1.03998	5.57	**15.0**	1.05916	1.06104	8.34
.1	1.03856	1.04039	5.63	.1	1.05959	1.06147	8.40
.2	1.03897	1.04081	5.68	.2	1.06002	1.06190	8.45
.3	1.03938	1.04122	5.74	.3	1.06045	1.06233	8.51
.4	1.03980	1.04164	5.80	.4	1.06088	1.06276	8.56
10.5	1.04021	1.04205	5.85	**15.5**	1.06131	1.06319	8.62
.6	1.04063	1.04247	5.91	.6	1.06174	1.06362	8.67
.7	1.04104	1.04288	5.96	.7	1.06217	1.06405	8.73
.8	1.04146	1.04330	6.02	.8	1.06260	1.06448	8.78
.9	1.04187	1.04371	6.07	.9	1.06303	1.06491	8.84
11.0	1.04229	1.04413	6.13	**16.0**	1.06346	1.06534	8.89
.1	1.04270	1.04455	6.18	.1	1.06389	1.06577	8.95
.2	1.04312	1.04497	6.24	.2	1.06432	1.06621	9.00
.3	1.04354	1.04538	6.30	.3	1.06476	1.06664	9.06
.4	1.04395	1.04580	6.35	.4	1.06519	1.06707	9.11
11.5	1.04437	1.04622	6.41	**16.5**	1.06562	1.06751	9.17
.6	1.04479	1.04664	6.46	.6	1.06605	1.06794	9.22
.7	1.04521	1.04706	6.52	.7	1.06649	1.06837	9.28
.8	1.04562	1.04747	6.57	.8	1.06692	1.06881	9.33
.9	1.04604	1.04789	6.63	.9	1.06736	1.06924	9.39
12.0	1.04646	1.04831	6.68	**17.0**	1.06779	1.06968	9.45
.1	1.04688	1.04873	6.74	.1	1.06822	1.07011	9.50
.2	1.04730	1.04915	6.79	.2	1.06866	1.07055	9.56
.3	1.04772	1.04957	6.85	.3	1.06909	1.07098	9.61
.4	1.04814	1.04999	6.90	.4	1.06953	1.07142	9.67
12.5	1.04856	1.05041	6.96	**17.5**	1.06996	1.07186	9.72
.6	1.04898	1.05084	7.02	.6	1.07040	1.07229	9.78
.7	1.04940	1.05126	7.07	.7	1.07084	1.07273	9.83
.8	1.04982	1.05168	7.13	.8	1.07127	1.07317	9.89
.9	1.05024	1.05210	7.18	.9	1.07171	1.07361	9.94
13.0	1.05066	1.05252	7.24	**18.0**	1.07215	1.07404	10.00
.1	1.05109	1.05295	7.29	.1	1.07258	1.07448	10.05
.2	1.05151	1.05337	7.35	.2	1.07302	1.07492	10.11
.3	1.05193	1.05379	7.40	.3	1.07346	1.07536	10.16
.4	1.05236	1.05422	7.46	.4	1.07390	1.07580	10.22
13.5	1.05278	1.05464	7.51	**18.5**	1.07434	1.07624	10.27
.6	1.05320	1.05506	7.57	.6	1.07478	1.07668	10.33
.7	1.05363	1.05549	7.62	.7	1.07522	1.07712	10.38
.8	1.05405	1.05591	7.68	.8	1.07566	1.07756	10.44
.9	1.05448	1.05634	7.73	.9	1.07610	1.07800	10.49
14.0	1.05490	1.05677	7.79	**19.0**	1.07654	1.07844	10.55
.1	1.05532	1.05719	7.84	.1	1.07698	1.07888	10.60
.2	1.05575	1.05762	7.90	.2	1.07742	1.07932	10.66
.3	1.05618	1.05804	7.96	.3	1.07786	1.07977	10.71
.4	1.05660	1.05847	8.01	.4	1.07830	1.08021	10.77
14.5	1.05703	1.05890	8.07	**19.5**	1.07874	1.08065	10.82
.6	1.05746	1.05933	8.12	.6	1.07919	1.08110	10.88
.7	1.05788	1.05975	8.18	.7	1.07963	1.08154	10.93
.8	1.05831	1.06018	8.23	.8	1.08007	1.08198	10.99
.9	1.05874	1.06061	8.29	.9	1.08052	1.08243	11.04

TABLE 27 (*Continued*)

DEGREES BRIX, SPECIFIC GRAVITY, AND DEGREES BAUMÉ OF SUGAR SOLUTIONS AT 20° C.

Degrees Brix or Per Cent Sucrose by Weight	Specific Gravity at 20°/4° C.	Specific Gravity at 20°/20° C.	Degrees Baumé (Modulus 145)	Degrees Brix or Per Cent Sucrose by Weight	Specific Gravity at 20°/4° C.	Specific Gravity at 20°/20° C.	Degrees Baumé (Modulus 145)
20.0	1.08096	1.08287	11.10	**25.0**	1.10356	1.10551	13.84
.1	1.08140	1.08332	11.15	.1	1.10402	1.10597	13.89
.2	1.08185	1.08376	11.21	.2	1.10448	1.10643	13.95
.3	1.08229	1.08421	11.26	.3	1.10494	1.10689	14.00
.4	1.08274	1.08465	11.32	.4	1.10540	1.10736	14.06
20.5	1.08318	1.08510	11.37	**25.5**	1.10586	1.10782	14.11
.6	1.08363	1.08554	11.43	.6	1.10632	1.10828	14.17
.7	1.08407	1.08599	11.48	.7	1.10679	1.10874	14.22
.8	1.08452	1.08644	11.54	.8	1.10725	1.10921	14.28
.9	1.08497	1.08689	11.59	.9	1.10771	1.10967	14.33
21.0	1.08541	1.08733	11.65	**26.0**	1.10818	1.11014	14.39
.1	1.08586	1.08778	11.70	.1	1.10864	1.11060	14.44
.2	1.08631	1.08823	11.76	.2	1.10910	1.11106	14.49
.3	1.08676	1.08868	11.81	.3	1.10957	1.11153	14.55
.4	1.08720	1.08913	11.87	.4	1.11003	1.11200	14.60
21.5	1.08765	1.08958	11.92	**26.5**	1.11050	1.11246	14.66
.6	1.08810	1.09003	11.98	.6	1.11096	1.11293	14.71
.7	1.08855	1.09048	12.03	.7	1.11143	1.11339	14.77
.8	1.08900	1.09093	12.09	.8	1.11190	1.11386	14.82
.9	1.08945	1.09138	12.14	.9	1.11236	1.11433	14.88
22.0	1.08990	1.09183	12.20	**27.0**	1.11283	1.11480	14.93
.1	1.09035	1.09228	12.25	.1	1.11330	1.11526	14.99
.2	1.09080	1.09273	12.31	.2	1.11376	1.11573	15.04
.3	1.09125	1.09318	12.36	.3	1.11423	1.11620	15.09
.4	1.09170	1.09364	12.42	.4	1.11470	1.11667	15.15
22.5	1.09216	1.09409	12.47	**27.5**	1.11517	1.11714	15.20
.6	1.09261	1.09454	12.52	.6	1.11564	1.11761	15.26
.7	1.09306	1.09499	12.58	.7	1.11610	1.11808	15.31
.8	1.09351	1.09545	12.63	.8	1.11657	1.11855	15.37
.9	1.09397	1.09590	12.69	.9	1.11704	1.11902	15.42
23.0	1.09442	1.09636	12.74	**28.0**	1.11751	1.11949	15.48
.1	1.09487	1.09681	12.80	.1	1.11798	1.11996	15.53
.2	1.09533	1.09727	12.85	.2	1.11845	1.12043	15.59
.3	1.09578	1.09772	12.91	.3	1.11892	1.12090	15.64
.4	1.09624	1.09818	12.96	.4	1.11940	1.12138	15.69
23.5	1.09669	1.09863	13.02	**28.5**	1.11987	1.12185	15.75
.6	1.09715	1.09909	13.07	.6	1.12034	1.12232	15.80
.7	1.09760	1.09954	13.13	.7	1.12081	1.12280	15.86
.8	1.09806	1.10000	13.18	.8	1.12128	1.12327	15.91
.9	1.09851	1.10046	13.24	.9	1.12176	1.12374	15.97
24.0	1.09897	1.10092	13.29	**29.0**	1.12223	1.12422	16.02
.1	1.09943	1.10137	13.35	.1	1.12270	1.12469	16.08
.2	1.09989	1.10183	13.40	.2	1.12318	1.12517	16.13
.3	1.10034	1.10229	13.46	.3	1.12365	1.12564	16.18
.4	1.10080	1.10275	13.51	.4	1.12413	1.12612	16.24
24.5	1.10126	1.10321	13.57	**29.5**	1.12460	1.12659	16.29
.6	1.10172	1.10367	13.62	.6	1.12508	1.12707	16.35
.7	1.10218	1.10413	13.67	.7	1.12556	1.12755	16.40
.8	1.10264	1.10459	13.73	.8	1.12603	1.12802	16.46
.9	1.10310	1.10505	13.78	.9	1.12651	1.12850	16.51

TABLE 27 (*Continued*)

DEGREES BRIX, SPECIFIC GRAVITY, AND DEGREES BAUMÉ OF SUGAR SOLUTIONS AT 20° C.

Degrees Brix or Per Cent Sucrose by Weight	Specific Gravity at 20°/4° C.	Specific Gravity at 20°/20° C.	Degrees Baumé (Modulus 145)	Degrees Brix or Per Cent Sucrose by Weight	Specific Gravity at 20°/4° C.	Specific Gravity at 20°/20° C.	Degrees Baumé (Modulus 145)
30.0	1.12698	1.12898	16.57	**35.0**	1.15128	1.15331	19.28
.1	1.12746	1.12946	16.62	.1	1.15177	1.15381	19.33
.2	1.12794	1.12993	16.67	.2	1.15226	1.15430	19.38
.3	1.12842	1.13041	16 73	.3	1.15276	1.15480	19.44
.4	1.12890	1.13089	16.78	.4	1.15326	1.15530	19.49
30.5	1.12937	1.13137	16.84	**35.5**	1.15375	1.15579	19.55
.6	1.12985	1.13185	16.89	.6	1.15425	1.15629	19.60
.7	1.13033	1.13233	16.95	.7	1.15475	1.15679	19.65
.8	1.13081	1.13281	17.00	.8	1.15524	1.15729	19.71
.9	1.13129	1.13329	17.05	.9	1.15574	1.15778	19.76
31.0	1.13177	1.13378	17.11	**36.0**	1.15624	1.15828	19.81
.1	1.13225	1.13426	17.16	.1	1.15674	1.15878	19.87
.2	1.13274	1.13474	17.22	.2	1.15724	1.15928	19.92
.3	1.13322	1.13522	17.27	.3	1.15773	1.15978	19.98
.4	1.13370	1.13570	17.33	.4	1.15823	1.16028	20.03
31.5	1.13418	1.13619	17.38	**36.5**	1.15873	1.16078	20.08
.6	1.13466	1.13667	17.43	.6	1.15923	1.16128	20.14
.7	1.13515	1.13715	17.49	.7	1.15973	1.16178	20.19
.8	1.13563	1.13764	17.54	.8	1.16023	1.16228	20.25
.9	1.13611	1.13812	17.60	.9	1.16073	1.16279	20.30
32.0	1.13660	1.13861	17.65	**37.0**	1.16124	1.16329	20.35
.1	1.13708	1.13909	17.70	.1	1.16174	1.16379	20.41
.2	1.13756	1.13958	17.76	.2	1.16224	1.16430	20.46
.3	1.13805	1.14006	17.81	.3	1.16274	1.16480	20.52
.4	1.13853	1.14055	17.87	.4	1.16324	1.16530	20.57
32.5	1.13902	1.14103	17.92	**37.5**	1.16375	1.16581	20.62
.6	1.13951	1.14152	17.98	.6	1.16425	1.16631	20.68
.7	1.13999	1.14201	18.03	.7	1.16476	1.16682	20.73
.8	1.14048	1.14250	18.08	.8	1.16526	1.16732	20.78
.9	1.14097	1.14298	18.14	.9	1.16576	1.16783	20.84
33.0	1.14145	1.14347	18.19	**38.0**	1.16627	1.16833	20.89
.1	1.14194	1.14396	18.25	.1	1.16678	1.16884	20.94
.2	1.14243	1.14445	18.30	.2	1.16728	1.16934	21.00
.3	1.14292	1.14494	18.36	.3	1.16779	1.16985	21.05
.4	1.14340	1.14543	18.41	.4	1.16829	1.17036	21.11
33.5	1.14389	1.14592	18.46	**38.5**	1.16880	1.17087	21.16
.6	1.14438	1.14641	18.52	.6	1.16931	1.17138	21.21
.7	1.14487	1.14690	18.57	.7	1.16982	1.17188	21.27
.8	1.14536	1.14739	18.63	.8	1.17032	1.17239	21.32
.9	1.14585	1.14788	18.68	.9	1.17083	1.17290	21.38
34.0	1.14634	1.14837	18.73	**39.0**	1.17134	1.17341	21.43
.1	1.14684	1.14886	18.79	.1	1.17185	1.17392	21.48
.2	1.14733	1.14936	18.84	.2	1.17236	1.17443	21.54
.3	1.14782	1.14985	18.90	.3	1.17287	1.17494	21.59
.4	1.14831	1.15034	18.95	.4	1.17338	1.17545	21.64
34.5	1.14880	1.15084	19.00	**39.5**	1.17389	1.17596	21.70
.6	1.14930	1.15133	19.06	.6	1.17440	1.17648	21.75
.7	1.14979	1.15183	19.11	.7	1.17491	1.17699	21.80
.8	1.15029	1.15232	19.17	.8	1.17542	1.17750	21.86
.9	1.15078	1.15282	19.22	.9	1.17594	1.17802	21.91

TABLE 27 (*Continued*)

DEGREES BRIX, SPECIFIC GRAVITY, AND DEGREES BAUMÉ OF SUGAR SOLUTIONS AT 20° C.

Degrees Brix or Per Cent Sucrose by Weight	Specific Gravity at 20°/4°.C.	Specific Gravity at 20°/20° C.	Degrees Baumé (Modulus 145)	Degrees Brix or Per Cent Sucrose by Weight	Specific Gravity at 20°/4° C.	Specific Gravity at 20°/20° C.	Degrees Baumé (Modulus 145)
40.0	1.17645	1.17853	21 97	**45.0**	1.20254	1.20467	24.63
.1	1.17696	1.17904	22.02	.1	1.20307	1.20520	24.69
.2	1.17747	1.17956	22.07	.2	1.20360	1.20573	24.74
.3	1.17799	1.18007	22.13	.3	1.20414	1.20627	24.79
.4	1.17850	1.18058	22.18	.4	1.20467	1.20680	24.85
40.5	1.17901	1.18110	22.23	**45.5**	1.20520	1.20733	24.90
.6	1.17953	1.18162	22.29	.6	1.20573	1.20787	24.95
.7	1.18004	1.18213	22.34	.7	1.20627	1.20840	25.01
.8	1.18056	1.18265	22.39	.8	1.20680	1.20894	25.06
.9	1.18108	1.18316	22.45	.9	1.20734	1.20947	25.11
41.0	1.18159	1.18368	22.50	**46.0**	1.20787	1.21001	25.17
.1	1.18211	1.18420	22.55	.1	1.20840	1.21054	25.22
.2	1.18262	1.18472	22.61	.2	1.20894	1.21108	25.27
.3	1.18314	1.18524	22.66	.3	1.20948	1.21162	25.32
.4	1.18366	1.18575	22.72	.4	1.21001	1.21215	25.38
41.5	1.18418	1.18627	22.77	**46.5**	1.21055	1.21269	25.43
.6	1.18470	1.18679	22.82	.6	1.21109	1.21323	25.48
.7	1.18522	1.18731	22.88	.7	1.21162	1.21377	25.54
.8	1.18573	1.18783	22.93	.8	1.21216	1.21431	25.59
.9	1.18625	1.18835	22.98	.9	1.21270	1.21484	25.64
42.0	1.18677	1.18887	23.04	**47.0**	1.21324	1.21538	25.70
.1	1.18729	1.18939	23.09	.1	1.21378	1.21592	25.75
.2	1.18781	1.18992	23.14	.2	1.21432	1.21646	25.80
.3	1.18834	1.19044	23.20	.3	1.21486	1.21700	25.86
.4	1.18886	1.19096	23.25	.4	1.21540	1.21755	25.91
42.5	1.18938	1.19148	23.30	**47.5**	1.21594	1.21809	25.96
.6	1.18990	1.19201	23.36	.6	1.21648	1.21863	26.01
.7	1.19042	1.19253	23.41	.7	1.21702	1.21917	26.07
.8	1.19095	1.19305	23.46	.8	1.21756	1.21971	26.12
.9	1.19147	1.19358	23.52	.9	1.21810	1.22026	26.17
43.0	1.19199	1.19410	23.57	**48.0**	1.21864	1.22080	26.23
.1	1.19252	1.19463	23.62	.1	1.21918	1.22134	26.28
.2	1.19304	1.19515	23.68	.2	1.21973	1.22189	26.33
.3	1.19356	1.19568	23.73	.3	1.22027	1.22243	26.38
.4	1.19409	1.19620	23.78	.4	1.22082	1.22298	26.44
43.5	1.19462	1.19673	23.84	**48.5**	1.22136	1.22352	26.49
.6	1.19514	1.19726	23.89	.6	1.22190	1.22406	26.54
.7	1.19567	1.19778	23.94	.7	1.22245	1.22461	26.59
.8	1.19619	1.19831	24.00	.8	1.22300	1.22516	26.65
.9	1.19672	1.19884	24.05	.9	1.22354	1.22570	26.70
44.0	1.19725	1.19936	24.10	**49.0**	1.22409	1.22625	26.75
.1	1.19778	1.19989	24.16	.1	1.22463	1.22680	26.81
.2	1.19830	1.20042	24.21	.2	1.22518	1.22735	26.86
.3	1.19883	1.20095	24.26	.3	1.22573	1.22789	26.91
.4	1.19936	1.20148	24.32	.4	1.22627	1.22844	26.96
44.5	1.19989	1.20201	24.37	**49.5**	1.22682	1.22899	27.02
.6	1.20042	1.20254	24.42	.6	1.22737	1.22954	27.07
.7	1.20095	1.20307	24.48	.7	1.22792	1.23009	27.12
.8	1.20148	1.20360	24.53	.8	1.22847	1.23064	27.18
.9	1.20201	1.20414	24.58	.9	1.22902	1.23119	27.23

TABLE 27 (*Continued*)

DEGREES BRIX, SPECIFIC GRAVITY, AND DEGREES BAUMÉ OF SUGAR SOLUTIONS AT 20° C.

Degrees Brix or Per Cent Sucrose by Weight	Specific Gravity at 20°/4° C.	Specific Gravity at 20°/20° C.	Degrees Baumé (Modulus 145)	Degrees Brix or Per Cent Sucrose by Weight	Specific Gravity at 20°/4° C.	Specific Gravity at 20°/20° C.	Degrees Baumé (Modulus 145)
50.0	1.22957	1.23174	27.28	**55.0**	1.25754	1.25976	29.90
.1	1.23012	1.23229	27.33	.1	1.25810	1.26033	29.95
.2	1.23067	1.23284	27.39	.2	1.25867	1.26090	30.00
.3	1.23122	1.23340	27.44	.3	1.25924	1.26147	30.06
.4	1.23177	1.23395	27.49	.4	1.25982	1.26204	30.11
50.5	1.23232	1.23450	27.54	**55.5**	1.26039	1.26261	30.16
.6	1.23287	1.23506	27.60	.6	1.26096	1.26319	30.21
.7	1.23343	1.23561	27.65	.7	1.26153	1.26376	30.26
.8	1.23398	1.23616	27.70	.8	1.26210	1.26433	30.32
.9	1.23453	1.23672	27.75	.9	1.26267	1.26490	30.37
51.0	1.23508	1.23727	27.81	**56.0**	1.26324	1.26548	30.42
.1	1.23564	1.23782	27.86	.1	1.26382	1.26605	30.47
.2	1.23619	1.23838	27.91	.2	1.26439	1.26663	30.52
.3	1.23675	1.23894	27.96	.3	1.26496	1.26720	30.57
.4	1.23730	1.23949	28.02	.4	1.26554	1.26778	30.63
51.5	1.23786	1.24005	28.07	**56.5**	1.26611	1.26835	30.68
.6	1.23841	1.24060	28.12	.6	1.26669	1.26893	30.73
.7	1.23897	1.24116	28.17	.7	1.26726	1.26950	30.78
.8	1.23953	1.24172	28.23	.8	1.26784	1.27008	30.83
.9	1.24008	1.24228	28.28	.9	1.26841	1.27066	30.89
52.0	1.24064	1.24284	28.33	**57.0**	1.26899	1.27123	30.94
.1	1.24120	1.24339	28.38	.1	1.26956	1.27181	30.99
.2	1.24176	1.24395	28.44	.2	1.27014	1.27239	31.04
.3	1.24232	1.24451	28.49	.3	1.27072	1.27297	31.09
.4	1.24287	1.24507	28.54	.4	1.27130	1.27355	31.15
52.5	1.24343	1.24563	28.59	**57.5**	1.27188	1.27413	31.20
.6	1.24399	1.24619	28.65	.6	1.27246	1.27471	31.25
.7	1.24455	1.24675	28.70	.7	1.27304	1.27529	31.30
.8	1.24511	1.24731	28.75	.8	1.27361	1.27587	31.35
.9	1.24567	1.24788	28.80	.9	1.27419	1.27645	31.40
53.0	1.24623	1.24844	28.86	**58.0**	1.27477	1.27703	31.46
.1	1.24680	1.24900	28.91	.1	1.27535	1.27761	31.51
.2	1.24736	1.24956	28.96	.2	1.27594	1.27819	31.56
.3	1.24792	1.25013	29.01	.3	1.27652	1.27878	31.61
.4	1.24848	1.25069	29.06	.4	1.27710	1.27936	31.66
53.5	1.24905	1.25126	29.12	**58.5**	1.27768	1.27994	31.71
.6	1.24961	1.25182	29.17	.6	1.27826	1.28052	31.76
.7	1.25017	1.25238	29.22	.7	1.27884	1.28111	31.82
.8	1.25074	1.25295	29.27	.8	1.27943	1.28169	31.87
.9	1.25130	1.25351	29.32	.9	1.28001	1.28228	31.92
54.0	1.25187	1.25408	29.38	**59.0**	1.28060	1.28286	31.97
.1	1.25243	1.25465	29.43	.1	1.28118	1.28345	32.02
.2	1.25300	1.25521	29.48	.2	1.28176	1.28404	32.07
.3	1.25356	1.25578	29.53	.3	1.28235	1.28462	32.13
.4	1.25413	1.25635	29.59	.4	1.28294	1.28520	32.18
54.5	1.25470	1.25692	29.64	**59.5**	1.28352	1.28579	32.23
.6	1.25526	1.25748	29.69	.6	1.28411	1.28638	32.28
.7	1.25583	1.25805	29.74	.7	1.28469	1.28697	32.33
.8	1.25640	1.25862	29.80	.8	1.28528	1.28755	32.38
.9	1.25697	1.25919	29.85	.9	1.28587	1.28814	32.43

TABLE 27 (*Continued*)

DEGREES BRIX, SPECIFIC GRAVITY, AND DEGREES BAUMÉ OF SUGAR SOLUTIONS AT 20° C.

Degrees Brix or Per Cent Sucrose by Weight	Specific Gravity at 20°/4° C.	Specific Gravity at 20°/20° C.	Degrees Baumé (Modulus 145)	Degrees Brix or Per Cent Sucrose by Weight	Specific Gravity at 20°/4° C.	Specific Gravity at 20°/20° C.	Degrees Baumé (Modulus 145)
60.0	1.28646	1.28873	32.49	**65.0**	1.31633	1.31866	35.04
.1	1.28704	1.28932	32.54	.1	1.31694	1.31927	35.09
.2	1.28763	1.28991	32.59	.2	1.31755	1.31988	35.14
.3	1.28822	1.29050	32.64	.3	1.31816	1.32049	35.19
.4	1.28881	1.29109	32.69	.4	1.31877	1.32110	35.24
60.5	1.28940	1.29168	32.74	**65.5**	1.31937	1.32171	35.29
.6	1.28999	1.29227	32.79	.6	1.31998	1.32232	35.34
.7	1.29058	1.29286	32.85	.7	1.32059	1.32293	35.39
.8	1.29117	1.29346	32.90	.8	1.32120	1.32354	35.45
.9	1.29176	1.29405	32.95	.9	1.32181	1.32415	35.50
61.0	1.29235	1.29464	33.00	**66.0**	1.32242	1.32476	35.55
.1	1.29295	1.29523	33.05	.1	1.32304	1.32538	35.60
.2	1.29354	1.29583	33.10	.2	1.32365	1.32599	35.65
.3	1.29413	1.29642	33.15	.3	1.32426	1.32660	35.70
.4	1.29472	1.29701	33.20	.4	1.32487	1.32722	35.75
61.5	1.29532	1.29761	33.26	**66.5**	1.32548	1.32783	35.80
.6	1.29591	1.29820	33.31	.6	1.32610	1.32844	35.85
.7	1.29651	1.29880	33.36	.7	1.32671	1.32906	35.90
.8	1.29710	1.29940	33.41	.8	1.32732	1.32967	35.95
.9	1.29770	1.29999	33.46	.9	1.32794	1.33029	36.00
62.0	1.29829	1.30059	33.51	**67.0**	1.32855	1.33090	36.05
.1	1.29889	1.30118	33.56	.1	1.32917	1.33152	36.10
.2	1.29948	1.30178	33.61	.2	1.32978	1.33214	36.15
.3	1.30008	1.30238	33.67	.3	1.33040	1.33275	36.20
.4	1.30068	1.30298	33.72	.4	1.33102	1.33337	36.25
62.5	1.30127	1.30358	33.77	**67.5**	1.33163	1.33399	36.30
.6	1.30187	1.30418	33.82	.6	1.33225	1.33460	36.35
.7	1.30247	1.30477	33.87	.7	1.33287	1.33523	36.40
.8	1.30307	1.30537	33.92	.8	1.33348	1.33584	36.45
.9	1.30367	1.30597	33.97	.9	1.33410	1.33646	36.50
63.0	1.30427	1.30657	34.02	**68.0**	1.33472	1.33708	36.55
.1	1.30487	1.30718	34.07	.1	1.33534	1.33770	36.61
.2	1.30547	1.30778	34.12	.2	1.33596	1.33832	36.66
.3	1.30607	1.30838	34.18	.3	1.33658	1.33894	36.71
.4	1.30667	1.30898	34.23	.4	1.33720	1.33957	36.76
63.5	1.30727	1.30958	34.28	**68.5**	1.33782	1.34019	36.81
.6	1.30787	1.31019	34.33	.6	1.33844	1.34081	36.86
.7	1.30848	1.31079	34.38	.7	1.33906	1.34143	36.91
.8	1.30908	1.31139	34.43	.8	1.33968	1.34205	36.96
.9	1.30968	1.31200	34.48	.9	1.34031	1.34268	37.01
64.0	1.31028	1.31260	34.53	**69.0**	1.34093	1.34330	37.06
.1	1.31088	1.31320	34.58	.1	1.34155	1.34392	37.11
.2	1.31149	1.31381	34.63	.2	1.34217	1.34455	37.16
.3	1.31209	1.31441	34.68	.3	1.34280	1.34517	37.21
.4	1.31270	1.31502	34.74	.4	1.34342	1.34580	37.26
64.5	1.31330	1.31563	34.79	**69.5**	1.34405	1.34642	37.31
.6	1.31391	1.31623	34.84	.6	1.34467	1.34705	37.36
.7	1.31452	1.31684	34.89	.7	1.34530	1.34768	37.41
.8	1.31512	1.31745	34.94	.8	1.34592	1.34830	37.46
.9	1.31573	1.31806	34.99	.9	1.34655	1.34893	37.51

TABLE 27 (*Continued*)

DEGREES BRIX, SPECIFIC GRAVITY, AND DEGREES BAUMÉ OF SUGAR SOLUTIONS AT 20° C.

Degrees Brix or Per Cent Sucrose by Weight	Specific Gravity at 20°/4 C°.	Specific Gravity at 20°/20° C.	Degrees Baumé (Modulus 145)	Degrees Brix or Per Cent Sucrose by Weight	Specific Gravity at 20°/4° C.	Specific Gravity at 20°/20° C.	Degrees Baumé (Modulus 145)
70.0	1.34717	1.34956	37.56	**75.0**	1.37897	1.38141	40.03
.1	1.34780	1.35019	37.61	.1	1.37962	1.38206	40.08
.2	1.34843	1.35081	37.66	.2	1.38026	1.38270	40.13
.3	1.34906	1.35144	37.71	.3	1.38091	1.38335	40.18
.4	1.34968	1.35207	37.76	.4	1.38156	1.38400	40.23
70.5	1.35031	1.35270	37.81	**75.5**	1.38220	1.38465	40.28
.6	1.35094	1.35333	37.86	.6	1.38285	1.38530	40.33
.7	1.35157	1.35396	37.91	.7	1.38350	1.38595	40.38
.8	1.35220	1.35459	37.96	.8	1.38415	1.38660	40.43
.9	1.35283	1.35522	38.01	.9	1.38480	1.38725	40.48
71.0	1.35346	1.35585	38.06	**76.0**	1.38545	1.38790	40.53
.1	1.35409	1.35648	38.11	.1	1.38610	1.38855	40.57
.2	1.35472	1.35711	38.16	.2	1.38675	1.38920	40.62
.3	1.35535	1.35775	38.21	.3	1.38740	1.38985	40.67
.4	1.35598	1.35838	38.26	.4	1.38805	1.39050	40.72
71.5	1.35661	1.35901	38.30	**76.5**	1.38870	1.39115	40.77
.6	1.35724	1.35964	38.35	.6	1.38935	1.39180	40.82
.7	1.35788	1.36028	38.40	.7	1.39000	1.39246	40.87
.8	1.35851	1.36091	38.45	.8	1.39065	1.39311	40.92
.9	1.35914	1.36155	38.50	.9	1.39130	1.39376	40.97
72.0	1.35978	1.36218	38.55	**77.0**	1.39196	1.39442	41.01
.1	1.36041	1.36282	38.60	.1	1.39261	1.39507	41.06
.2	1.36105	1.36346	38.65	.2	1.39326	1.39573	41.11
.3	1.36168	1.36409	38.70	.3	1.39392	1.39638	41.16
.4	1.36232	1.36473	38.75	.4	1.39457	1.39704	41.21
72.5	1.36295	1.36536	38.80	**77.5**	1.39523	1.39769	41.26
.6	1.36359	1.36600	38.85	.6	1.39588	1.39835	41.31
.7	1.36423	1.36664	38.90	.7	1.39654	1.39901	41.36
.8	1.36486	1.36728	38.95	.8	1.39719	1.39966	41.40
.9	1.36550	1.36792	39.00	.9	1.39785	1.40032	41.45
73.0	1.36614	1.36856	39.05	**78.0**	1.39850	1.40098	41.50
.1	1.36678	1.36919	39.10	.1	1.39916	1.40164	41.55
.2	1.36742	1.36983	39.15	.2	1.39982	1.40230	41.60
.3	1.36805	1.37047	39.20	.3	1.40048	1.40295	41.65
.4	1.36869	1.37111	39.25	.4	1.40113	1.40361	41.70
73.5	1.36933	1.37176	39.30	**78.5**	1.40179	1.40427	41.74
.6	1.36997	1.37240	39.35	.6	1.40245	1.40493	41.79
.7	1.37061	1.37304	39.39	.7	1.40311	1.40559	41.84
.8	1.37125	1.37368	39.44	.8	1.40377	1.40625	41.89
.9	1.37189	1.37432	39.49	.9	1.40443	1.40691	41.94
74.0	1.37254	1.37496	39.54	**79.0**	1.40509	1.40758	41.99
.1	1.37318	1.37561	39.59	.1	1.40575	1.40824	42.03
.2	1.37382	1.37625	39.64	.2	1.40641	1.40890	42.08
.3	1.37446	1.37689	39.69	.3	1.40707	1.40956	42.13
.4	1.37510	1.37754	39.74	.4	1.40774	1.41023	42.18
74.5	1.37575	1.37818	39.79	**79.5**	1.40840	1.41089	42.23
.6	1.37639	1.37883	39.84	.6	1.40906	1.41155	42.28
.7	1.37704	1.37947	39.89	.7	1.40972	1.41222	42.32
.8	1.37768	1.38012	39.94	.8	1.41039	1.41288	42.37
.9	1.37833	1.38076	39.99	.9	1.41105	1.41355	42.42

TABLE 27 (*Continued*)

DEGREES BRIX, SPECIFIC GRAVITY, AND DEGREES BAUMÉ OF SUGAR SOLUTIONS AT 20° C.

Degrees Brix or Per Cent Sucrose by Weight	Specific Gravity at 20°/4° C.	Specific Gravity at 20°/20° C.	Degrees Baumé (Modulus 145)	Degrees Brix or Per Cent Sucrose by Weight	Specific Gravity at 20°/4° C.	Specific Gravity at 20°/20° C.	Degrees Baumé (Modulus 145)
80.0	1.41172	1.41421	42.47	**85.0**	1.44539	1.44794	44.86
.1	1.41238	1.41488	42.52	.1	1.44607	1.44863	44.91
.2	1.41304	1 41554	42.57	.2	1.44675	1.44931	44.95
.3	1.41371	1.41621	42.61	.3	1.44744	1.45000	45.00
.4	1.41437	1.41688	42.66	.4	1.44812	1.45068	45.05
80.5	1.41504	1.41754	42.71	**85.5**	1.44881	1.45137	45.09
.6	1.41571	1.41821	42.76	.6	1.44949	1.45205	45.14
.7	1.41637	1.41888	42.81	.7	1.45018	1.45274	45.19
.8	1.41704	1.41955	42.85	.8	1.45086	1.45343	45.24
.9	1.41771	1.42022	42.90	.9	1.45154	1.45411	45.28
81.0	1.41837	1.42088	42.95	**86.0**	1.45223	1.45480	45.33
.1	1.41904	1.42155	43.00	.1	1.45292	1.45549	45.38
.2	1.41971	1.42222	43.05	.2	1.45360	1.45618	45.42
.3	1 42038	1.42289	43.10	.3	1.45429	1.45686	45.47
.4	1.42105	1.42356	43.14	.4	1.45498	1.45755	45.52
81.5	1.42172	1.42423	43.19	**86.5**	1.45567	1.45824	45.57
.6	1.42239	1.42490	43.24	.6	1.45636	1.45893	45.61
.7	1.42306	1.42558	43.29	.7	1.45704	1.45962	45.66
.8	1.42373	1.42625	43.33	.8	1.45773	1.46031	45.71
.9	1.42440	1.42692	43.38	.9	1.45842	1.46100	45.75
82.0	1.42507	1.42759	43.43	**87.0**	1.45911	1.46170	45.80
.1	1.42574	1.42827	43.48	.1	1.45980	1.46239	45.85
.2	1.42642	1.42894	43.53	.2	1.46050	1.46308	45.89
.3	1.42709	1.42961	43.57	.3	1.46119	1.46377	45.94
.4	1.42776	1.43029	43.62	.4	1.46188	1.46446	45.99
82.5	1.42844	1.43096	43.67	**87.5**	1.46257	1.46516	46.03
.6	1.42911	1.43164	43.72	.6	1.46326	1.46585	46.08
.7	1.42978	1.43231	43.77	.7	1.46395	1.46654	46.13
.8	1.43046	1.43298	43.81	.8	1.46464	1.46724	46.17
.9	1.43113	1.43366	43.86	.9	1.46534	1.46793	46.22
83.0	1.43181	1.43434	43.91	**88.0**	1.46603	1.46862	46.27
.1	1.43248	1.43502	43.96	.1	1.46673	1.46932	46.31
.2	1.43316	1.43569	44.00	.2	1.46742	1.47002	46.36
.3	1.43384	1.43637	44.05	.3	1.46812	1.47071	46.41
.4	1.43451	1.43705	44.10	.4	1.46881	1.47141	46.45
83.5	1.43519	1.43773	44.15	**88.5**	1.46950	1.47210	46.50
.6	1.43587	1.43841	44.19	.6	1.47020	1.47280	46.55
.7	1.43654	1.43908	44.24	.7	1.47090	1.47350	46.59
.8	1.43722	1.43976	44.29	.8	1.47159	1.47420	46.64
.9	1.43790	1.44044	44.34	.9	1.47229	1.47489	46.69
84.0	1.43858	1.44112	44.38	**89.0**	1.47299	1.47559	46.73
.1	1.43926	1.44180	44.43	.1	1.47368	1.47629	46.78
.2	1.43994	1.44249	44.48	.2	1.47438	1.47699	46.83
.3	1.44062	1.44317	44.53	.3	1.47508	1.47769	46.87
.4	1.44130	1.44385	44.57	.4	1.47578	1.47839	46.92
84.5	1.44198	1.44453	44.62	**89.5**	1.47648	1.47909	46.97
.6	1.44266	1.44521	44.67	.6	1.47718	1.47979	47.01
.7	1.44334	1.44590	44.72	.7	1.47788	1.48049	47.06
.8	1.44402	1.44658	44.76	.8	1.47858	1.48119	47.11
.9	1.44470	1.44726	44.81	.9	1.47928	1.48189	47.15

TABLE 27 (*Continued*)

DEGREES BRIX, SPECIFIC GRAVITY, AND DEGREES BAUMÉ OF SUGAR SOLUTIONS AT 20° C.

Degrees Brix or Per Cent Sucrose by Weight	Specific Gravity at 20°/4° C.	Specific Gravity at 20°/20° C.	Degrees Baumé (Modulus 145)	Degrees Brix or Per Cent Sucrose by Weight	Specific Gravity at 20°/4° C.	Specific Gravity at 20°/20° C.	Degrees Baumé (Modulus 145)
90.0	1.47998	1.48259	47.20	**95.0**	1.51546	1.51814	49.49
.1	1.48068	1.48330	47.24	.1	1.51617	1.51886	49.53
.2	1.48138	1.48400	47.29	.2	1.51689	1.51958	49.58
.3	1.48208	1.48470	47.34	.3	1.51761	1.52030	49.62
.4	1.48278	1.48540	47.38	.4	1.51833	1.52102	49.67
90.5	1.48348	1.48611	47.43	**95.5**	1.51905	1.52174	49.71
.6	1.48419	1.48681	47.48	.6	1.51977	1.52246	49.76
.7	1.48489	1.48752	47.52	.7	1.52049	1.52318	49.80
.8	1.48559	1.48822	47.57	.8	1.52121	1.52390	49.85
.9	1.48630	1.48893	47.61	.9	1.52193	1.52463	49.90
91.0	1.48700	1.48963	47.66	**96.0**	1.52266	1.52535	49.94
.1	1.48771	1.49034	47.71	.1	1.52338	1.52607	49.98
.2	1.48841	1.49104	47.75	.2	1.52410	1.52680	50.03
.3	1.48912	1.49175	47.80	.3	1.52482	1.52752	50.08
.4	1.48982	1.49246	47.84	.4	1.52555	1.52824	50.12
91.5	1.49053	1.49316	47.89	**96.5**	1.52627	1.52897	50.16
.6	1.49123	1.49387	47.94	.6	1.52699	1.52969	50.21
.7	1.49194	1.49458	47.98	.7	1.52772	1.53042	50.25
.8	1.49265	1.49529	48.03	.8	1.52844	1.53114	50.30
.9	1.49336	1.49600	48.08	.9	1.52917	1.53187	50.34
92.0	1.49406	1.49671	48.12	**97.0**	1.52989	1.53260	50.39
.1	1.49477	1.49741	48.17	.1	1.53062	1.53332	50.43
.2	1.49548	1.49812	48.21	.2	1.53134	1.53405	50.48
.3	1.49619	1.49883	48.26	.3	1.53207	1.53478	50.52
.4	1.49690	1.49954	48.30	.4	1.53279	1.53551	50.57
92.5	1.49761	1.50026	48.35	**97.5**	1.53352	1.53623	50.61
.6	1.49832	1.50097	48.40	.6	1.53425	1.53696	50.66
.7	1.49903	1.50168	48.44	.7	1.53498	1.53769	50.70
.8	1.49974	1.50239	48.49	.8	1.53570	1.53842	50.75
.9	1.50045	1.50310	48.53	.9	1.53643	1.53915	50.79
93.0	1.50116	1.50381	48.58	**98.0**	1.53716	1.53988	50.84
.1	1.50187	1.50453	48.62	.1	1.53789	1.54061	50.88
.2	1.50258	1.50524	48.67	.2	1.53862	1.54134	50.93
.3	1.50329	1.50595	48.72	.3	1.53935	1.54207	50.97
.4	1.50401	1.50667	48.76	.4	1.54008	1.54280	51.02
93.5	1.50472	1.50738	48.81	**98.5**	1.54081	1.54353	51.06
.6	1.50543	1.50810	48.85	.6	1.54154	1.54426	51.10
.7	1.50615	1.50881	48.90	.7	1.54227	1.54499	51.15
.8	1.50686	1.50952	48.94	.8	1.54300	1.54573	51.19
.9	1.50757	1.51024	48.99	.9	1.54373	1.54646	51.24
94.0	1.50829	1.51096	49.03	**99.0**	1.54446	1.54719	51.28
.1	1.50900	1.51167	49.08	.1	1.54519	1.54793	51.33
.2	1.50972	1.51239	49.12	.2	1.54593	1.54866	51.37
.3	1.51044	1.51311	49.17	.3	1.54666	1.54939	51.42
.4	1.51115	1.51382	49.22	.4	1.54739	1.55013	51.46
94.5	1.51187	1.51454	49.26	**99.5**	1.54813	1.55087	51.50
.6	1.51258	1.51526	49.31	.6	1.54886	1.55160	51.55
.7	1.51330	1.51598	49.35	.7	1.54960	1.55234	51.59
.8	1.51402	1.51670	49.40	.8	1.55033	1.55307	51.64
.9	1.51474	1.51742	49.44	.9	1.55106	1.55381	51.68
				100.0	1.55180	1.55454	51.73

TABLE 28

TEMPERATURE CORRECTIONS TO READINGS OF BRIX HYDROMETERS (STANDARD AT 20° C.) *

(This table is calculated using the data on thermal expansion of sugar solutions by Plato,† assuming the instrument to be of Jena 16^{III} glass. The table should be used with caution and only for approximate results when the temperature differs much from the standard temperature or from the temperature of the surrounding air.)

Temperature ° C.	Observed Per Cent of Sugar													
	0	5	10	15	20	25	30	35	40	45	50	55	60	70
	Subtract from Observed Per Cent													
0	0.30	0.49	0.65	0.77	0.89	0.99	1.08	1.16	1.24	1.31	1.37	1.41	1.44	1.49
5	.36	.47	.56	.65	.73	.80	.86	.91	.97	1.01	1.05	1.08	1.10	1.14
10	.32	.38	.43	.48	.52	.57	.60	.64	.67	.70	.72	.74	.75	.77
11	.31	.35	.40	.44	.48	.51	.55	.58	.60	.63	.65	.66	.68	.70
12	.29	.32	.36	.40	.43	.46	.50	.52	.54	.56	.58	.59	.60	.62
13	.26	.29	.32	.35	.38	.41	.44	.46	.48	.49	.51	.52	.53	.55
14	.24	.26	.29	.31	.34	.36	.38	.40	.41	.42	.44	.45	.46	.47
15	.20	.22	.24	.26	.28	.30	.32	.33	.34	.36	.36	.37	.38	.39
16	.17	.18	.20	.22	.23	.25	.26	.27	.28	.28	.29	.30	.31	.32
17	.13	.14	.15	.16	.18	.19	.20	.20	.21	.21	.22	.23	.23	.24
18	.09	.10	.10	.11	.12	.13	.13	.14	.14	.14	.15	.15	.15	.16
19	.05	.05	.05	.06	.06	.06	.07	.07	.07	.07	.08	.08	.08	.08

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* From Circular No. 19, 1914, U. S. Bureau of Standards, p. 25.

† Wiss. Abh. der Kaiserlichen Normal-Eichungs-Kommission, 2, p. 140, 1900.

TABLE 28 (*Continued*)

TEMPERATURE CORRECTIONS TO READINGS OF BRIX HYDROMETERS (20° C.)

Temperature ° C.	Observed Per Cent of Sugar													
	0	5	10	15	20	25	30	35	40	45	50	55	60	70
	Add to Observed Per Cent													
21	0.04	0.05	0.06	0.06	0.06	0.07	0.07	0.07	0.07	0.08	0.08	0.08	0.08	0.09
22	.10	.10	.11	.12	.12	.13	.14	.14	.14	.15	.16	.16	.16	.16
23	.16	.16	.17	.17	.19	.20	.21	.21	.22	.23	.24	.24	.24	.24
24	.21	.22	.23	.24	.26	.27	.28	.29	.30	.31	.32	.32	.32	.32
25	.27	.28	.30	.31	.32	.34	.35	.36	.38	.38	.39	.39	.40	.39
26	.33	.34	.36	.37	.40	.40	.42	.44	.46	.47	.47	.48	.48	.48
27	.40	.41	.42	.44	.46	.48	.50	.52	.54	.54	.55	.56	.56	.56
28	.46	.47	.49	.51	.54	.56	.58	.60	.61	.62	.63	.64	.64	.64
29	.54	.55	.56	.59	.61	.63	.66	.68	.70	.70	.71	.72	.72	.72
30	.61	.62	.63	.66	.68	.70	.73	.76	.78	.78	.79	.80	.80	.81
35	.99	1.01	1.02	1.06	1.10	1.13	1.16	1.18	1.20	1.21	1.22	1.22	1.23	1.22
40	1.42	1.45	1.47	1.51	1.54	1.57	1.62	1.62	1.64	1.65	1.65	1.65	1.66	1.65
45	1.91	1.94	1.96	2.00	2.03	2.05	2.07	2.09	2.10	2.10	2.10	2.10	2.10	2.08
50	2.46	2.48	2.50	2.53	2.56	2.57	2.58	2.59	2.59	2.58	2.58	2.57	2.56	2.52
55	3.05	3.07	3.09	3.12	3.12	3.12	3.12	3.11	3.10	3.08	3.07	3.05	3.03	2.97
60	3.69	3.72	3.73	3.73	3.72	3.70	3.67	3.65	3.62	3.60	3.57	3.54	3.50	3.43
65	4.4	4.4	4.4	4.4	4.4	4.4	4.3	4.2	4.2	4.1	4.1	4.0	4.0	3.9
70	5.1	5.1	5.1	5.0	5.0	5.0	4.9	4.8	4.8	4.7	4.7	4.6	4.6	4.4
75	6.1	6.0	6.0	5.9	5.8	5.8	5.7	5.6	5.5	5.4	5.4	5.3	5.2	5.0
80	7.1	7.0	7.0	6.9	6.8	6.7	6.6	6.4	6.3	6.2	6.1	6.0	5.9	5.6

TABLE 29

TEMPERATURE CORRECTIONS TO READINGS OF BAUMÉ HYDROMETERS, NATIONAL BUREAU OF STANDARDS BAUMÉ SCALE FOR SUGAR SOLUTIONS (20° C., MODULUS 145)

(*Bur. Standards Circ. C 440*, 1942, p. 625)

Temperature °C.	Observed Degrees Baumé								
	0	5	10	15	20	25	30	35	40
Subtract from Observed Degrees Baumé									
0	0.17	0.34	0.47	0.57	0.65	0.72	0.77	0.79	0.81
5	.21	.30	.39	.45	.51	.55	.59	.60	.61
10	.18	.23	.28	.32	.36	.38	.40	.41	.42
11	.18	.22	.26	.29	.32	.34	.36	.37	.38
12	.17	.20	.23	.26	.29	.31	.32	.33	.34
13	.15	.18	.20	.23	.25	.27	.28	.29	.29
14	.14	.16	.18	.20	.22	.23	.24	.25	.25
15	.11	.13	.15	.17	.18	.20	.20	.21	.21
16	.10	.11	.13	.14	.15	.15	.16	.17	.17
17	.07	.08	.10	.11	.11	.11	.13	.13	.13
18	.05	.06	.07	.07	.08	.08	.08	.08	.09
19	.03	.03	.03	.03	.04	.04	.04	.04	.05
Add to Observed Degrees Baumé									
21	0.02	0.03	0.03	0.04	0.04	0.04	0.04	0.05	0.05
22	.06	.06	.07	.07	.08	.08	.08	.09	.09
23	.09	.09	.10	.11	.12	.13	.13	.13	.13
24	.12	.13	.14	.15	.16	.17	.17	.17	.17
25	.15	.17	.18	.19	.20	.21	.21	.21	.21
26	.19	.20	.22	.22	.24	.26	.26	.26	.26
27	.23	.23	.25	.27	.29	.30	.30	.30	.30
28	.26	.27	.29	.31	.33	.34	.35	.35	.34
29	.31	.31	.34	.35	.37	.38	.39	.39	.38
30	.35	.35	.38	.39	.42	.43	.44	.43	.43
35	.56	.57	.60	.63	.65	.66	.66	.66	.65
40	.81	.82	.85	.87	.89	.90	.90	.89	.88
45	1.09	1.09	1.12	1.14	1.15	1.15	1.14	1.13	1.11
50	1.40	1.39	1.42	1.42	1.43	1.41	1.40	1.37	1.33
55	1.74	1.72	1.73	1.73	1.71	1.69	1.66	1.62	1.57
60	2.10	2.08	2.06	2.05	2.00	1.97	1.92	1.87	1.82
65	2.5	2.5	2.4	2.4	2.3	2.2	2.2	2.1	2.1
70	2.9	2.8	2.8	2.8	2.6	2.6	2.5	2.4	2.3
75	3.5	3.3	3.2	3.2	3.1	3.0	2.9	2.7	2.6
80	4.0	3.9	3.8	3.7	3.5	3.4	3.3	3.1	2.8

TABLE 30

Weight per Unit Volume and Weight of Solids (Brix) per Unit Volume of Sugar Solutions at 20° C.

(Expanded, 1943, from table of Snyder and Hammond, *U.S. Bur. Standards Circ. C 440*, 1942, p. 644)

Degree Brix	Weight (Pounds) in Air of		Pounds Solids (Brix) per		Degree Brix	Weight (Pounds) in Air of		Pounds Solids (Brix) per	
	1 Cu. Ft.	1 Gal.	1 Cu. Ft.	1 Gal.		1 Cu. Ft.	1 Gal.	1 Cu. Ft.	1 Gal
0.0	62.253	8.322	0.000	0.000	**10.0**	64.744	8.655	6.474	0.866
.2	62.298	8.328	.125	.017	.2	64.796	8.662	6.609	.883
.4	62.350	8.335	.249	.033	.4	64.849	8.669	6.744	.902
.6	62.395	8.341	.374	.050	.6	64 894	8 675	6.879	.820
.8	62.447	8.348	.500	.067	.8	64.946	8.682	7.014	.938
1.0	62.492	8.354	.625	.084	**11.0**	65.006	8.689	7.151	.956
.2	62.545	8.361	.751	.100	.2	65.051	8.696	7.286	.974
.4	62.590	8.367	.076	.117	.4	65.103	8.703	7.422	.992
.6	62.642	8.374	1.002	.134	.6	65.155	8.710	7.558	1.010
.8	62.687	8.380	1.128	.151	.8	65.208	8.717	7.695	1.029
2.0	62.739	8.387	1.255	.168	**12.0**	65.260	8.724	7.831	1.047
.2	62.784	8.393	1.381	.185	.2	65.312	8.731	7.968	1.065
.4	62.836	8.400	1.508	.202	.4	65.365	8.738	8.105	1.083
.6	62.881	8.406	1.635	.219	.6	65.417	8.745	8.243	1.102
.8	62.934	8.413	1.762	.236	.8	65.470	8.752	8.380	1.120
3.0	62.978	8.419	1.889	.253	**13.0**	65.522	8.759	8.518	1.139
.2	63.031	8.426	2.017	.270	.2	65.574	8.766	8.656	1.157
.4	63.076	8.432	2.145	.287	.4	65.627	8.773	8.794	1.176
.6	63.128	8.439	2.273	.304	.6	65.686	8.781	8.933	1.194
.8	63.180	8.446	2.401	.321	.8	65.739	8.788	9.072	1.213
4.0	63.225	8.452	2.529	.338	**14.0**	65.791	8.795	9.211	1.231
.2	63.278	8.459	2.658	.355	.2	65.844	8.802	9.350	1.250
.4	63.323	8.465	2.786	.372	.4	65.896	8.809	9.489	1.268
.6	63.375	8.472	2.915	.390	.6	65.948	8.816	9.628	1.287
.8	63.427	8.479	3.044	.407	.8	66.001	8.823	9.768	1.306
5.0	63.472	8.485	3.174	.424	**15.0**	66.053	8.830	9.908	1.325
.2	63.525	8.492	3.303	.442	.2	66.105	8.837	10.048	1.343
.4	63.577	8.499	3.433	.459	.4	66.165	8.845	10.189	1.362
.6	63.622	8.505	3.563	.476	.6	66.218	8.852	10.330	1.381
.8	63.674	8.512	3.693	.494	.8	66.270	8.859	10.471	1.400
6.0	63.727	8.519	3.824	.511	**16.0**	66.322	8.866	10.612	1.419
.2	63.779	8.526	3.954	.529	.2	66.375	8.873	10.753	1.437
.4	63.834	8.532	4.085	.546	.4	66.434	8.881	10.895	1.456
.6	63.876	8.539	4.216	.564	.6	66.487	8.888	11.037	1.475
.8	63.929	8.546	4.347	.581	.8	66.539	8.895	11.179	1.494
7.0	63.973	8.552	4.478	.599	**17.0**	66.592	8.902	11.321	1.513
.2	64.026	8.559	4.610	.616	.2	66.644	8.909	11.463	1.532
.4	64.078	8.566	4.742	.634	.4	66.704	8.917	11.606	1.551
.6	64.130	8.573	4.874	.652	.6	66.756	8.924	11.749	1.571
.8	64.183	8.580	5.006	.669	.8	66.809	8.931	11.892	1.590
8.0	64.228	8.586	5.138	.687	**18.0**	66.868	8.939	12.036	1.609
.2	64.280	8.593	5.271	.705	.2	66.921	8.946	12.180	1.628
.4	64.332	8.600	5.403	.722	.4	66.973	8.953	12.323	1.647
.6	64.385	8.607	5.537	.740	.6	67.033	8.961	12.468	1.667
.8	64.437	8.614	5.670	.758	.8	67.085	8.968	12.612	1.686
9.0	64.482	8.620	5.803	.776	**19.0**	67.138	8.975	12.756	1.705
.2	64.534	8.627	5.938	.794	.2	67.198	8.983	12.902	1.725
.4	64.587	8.634	6.071	.812	.4	67.250	8.990	13.047	1.744
.6	64.639	8.641	6.205	.829	.6	67.302	8.997	13.191	1.763
.8	64.692	8.648	6.340	.848	.8	67.362	9.005	13.338	1.783

TABLE 30 (*Continued*)

WEIGHT PER UNIT VOLUME AND WEIGHT OF SOLIDS (BRIX) PER UNIT VOLUME OF SUGAR SOLUTIONS AT 20° C.

Degree Brix	Weight (Pounds) in Air of		Pounds Solids (Brix) per		Degree Brix	Weight (Pounds) in Air of		Pounds Solids (Brix) per	
	1 Cu. Ft.	1 Gal.	1 Cu. Ft.	1 Gal.		1 Cu. Ft.	1 Gal.	1 Cu. Ft.	1 Gal.
20.0	67.414	9.012	13.483	1.802	**30.0**	70.287	9.396	21.086	2.819
.2	67.474	9.020	13.630	1.822	.2	70.347	9.404	21.244	2.840
.4	67.527	9.027	13.776	1.842	.4	70.407	9.412	21.404	2.861
.6	67.579	9.034	13.921	1.861	.6	70.466	9.420	21.563	2.883
.8	67.639	9.042	14.069	1.881	.8	70.526	9.428	21.722	2.904
21.0	67.691	9.049	14.215	1.900	**31.0**	70.586	9.436	21.882	2.925
.2	67.751	9.057	14.363	1.920	.2	70.646	9.444	22.042	2.947
.4	67.803	9.064	14.510	1.940	.4	70.706	9.452	22.202	2.968
.6	67.863	9.072	14.658	1.959	.6	70.766	9.460	22.362	2.989
.8	67.916	9.097	14.806	1.979	.8	70.826	9.468	22.523	3.011
22.0	67.975	9.087	14.955	1.999	**32.0**	70.893	9.477	22.686	3.033
.2	68.028	9.094	15.102	2.019	.2	70.953	9.485	22.847	3.054
.4	68.088	9.102	15.252	2.039	.4	71.013	9.493	23.008	3.076
.6	68.140	9.109	15.400	2.059	.6	71.072	9.501	23.169	3.097
.8	68.200	9.117	15.550	2.079	.8	71.132	9.509	23.331	3.119
23.0	68.260	9.125	15.700	2.099	**33.0**	71.192	9.517	23.493	3.141
.2	68.312	9.132	15.848	2.119	.2	71.252	9.525	23.656	3.162
.4	68.372	9.140	15.999	2.139	.4	71.312	9.533	23.818	3.184
.6	68.424	9.147	16.148	2.159	.6	71.372	9.541	23.981	3.206
.8	68.484	9.155	16.299	2.179	.8	71.439	9.550	24.146	3.228
24.0	68.544	9.163	16.451	2.199	**34.0**	71.499	9.558	24.310	3.250
.2	68.596	9.170	16.600	2.219	.2	71.559	9.566	24.473	3.272
.4	68.656	9.178	16.752	2.239	.4	71.618	9.574	24.637	3.293
.6	68.709	9.185	16.902	2.259	.6	71.686	9.583	24.803	3.316
.8	68.768	9.193	17.054	2.280	.8	71.746	9.591	24.968	3.338
25.0	68.828	9.201	17.207	2.300	**35.0**	71.806	9.599	25.132	3.368
.2	68.881	9.208	17.358	2.320	.2	71.865	9.607	25.296	3.382
.4	68.940	9.216	17.511	2.341	.4	71.932	9.616	25.464	3.404
.6	69.000	9.224	17.664	2.361	.6	71.993	9.624	25.630	3.426
.8	69.060	9.232	17.817	2.382	.8	72.052	9.632	25.795	3.448
26.0	69.113	9.239	17.969	2.402	**36.0**	72.112	9.640	25.960	3.470
.2	69.172	9.247	18.123	2.423	.2	72.180	9.649	26.129	3.493
.4	69.232	9.255	18.277	2.443	.4	72.239	9.657	26.295	3.515
.6	69.292	9.263	18.432	2.464	.6	72.299	9.665	26.461	3.537
.8	69.344	9.270	18.584	2.484	.8	72.367	9.674	26.631	3.560
27.0	69.404	9.278	18.739	2.505	**37.0**	72.426	9.682	26.798	3.582
.2	69.464	9.286	18.894	2.526	.2	72.494	9.691	26.968	3.605
.4	69.524	9.294	19.050	2.547	.4	72.554	9.699	27.135	3.627
.6	69.584	9.302	19.205	2.567	.6	72.613	9.707	27.302	3.650
.8	69.636	9.309	19.359	2.588	.8	72.681	9.716	27.473	3.673
28.0	69.696	9.317	19.515	2.609	**38.0**	72.741	9.724	27.642	3.695
.2	69.756	9.325	19.671	2.630	.2	72.808	9.733	27.813	3.718
.4	69.816	9.333	19.828	2.651	.4	72.868	9.741	27.981	3.741
.6	69.876	9.341	19.985	2.672	.6	72.928	9.749	28.150	3.763
.8	69.935	9.349	20.141	2.692	.8	72.995	9.758	28.322	3.786
29.0	69.995	9.357	20.299	2.714	**39.0**	73.055	9.766	28.491	3.809
.2	70.055	9.365	20.456	2.735	.2	73.122	9.775	28.664	3.882
.4	70.107	9.372	20.611	2.755	.4	73.182	9.783	28.834	3.855
.6	70.167	9.380	20.769	2.776	.6	73.249	9.792	29.007	3.878
.8	70.227	9.388	20.928	2.798	.8	73.317	9.801	29.180	3.901

TABLE 30 (*Continued*)

WEIGHT PER UNIT VOLUME AND WEIGHT OF SOLIDS (BRIX) PER UNIT VOLUME OF SUGAR SOLUTIONS AT 20° C.

Degree Brix	Weight (Pounds) in Air of		Pounds Solids (Brix) per		Degree Brix	Weight (Pounds) in Air of		Pounds Solids (Brix) per	
	1 Cu. Ft.	1 Gal.	1 Cu. Ft.	1 Gal.		1 Cu. Ft.	1 Gal.	1 Cu. Ft	1 Gal.
40.0	73.376	9.809	29.350	3.924	**50.0**	76.690	10.252	38.345	5.126
.2	73.444	9.818	29.524	3.947	.2	76.765	10.262	38.536	5.152
.4	73.504	9.826	29.696	3.970	.4	76.832	10.271	38.723	5.177
.6	73.571	9.835	29.870	3.993	.6	76.900	10.280	38.911	5.202
.8	73.631	9.843	30.041	4.016	.8	76.975	10.290	39.103	5.225
41.0	73.698	9.852	30.216	4.039	**51.0**	77.042	10.299	39.291	5.252
.2	73.765	9.861	30.391	4.063	.2	77.109	10.308	39.480	5.278
.4	73.825	9.869	30.564	4.086	.4	77.177	10.317	39.669	5.303
.6	73.893	9.878	30.739	4.109	.6	77.244	10.326	39.858	5.328
.8	73.960	9.887	30.915	4.133	.8	77.319	10.336	40.051	5.354
42.0	74.020	9.895	31.088	4.156	**52.0**	77.386	10.345	40.241	5.379
.2	74.087	9.904	31.265	4.180	.2	77.453	10.354	40.430	5.405
.4	74.154	9.913	31.441	4.203	.4	77.528	10.364	40.625	5.431
.6	74.214	9.921	31.615	4.226	.6	77.595	10.373	40.815	5.456
.8	74.282	9.930	31.793	4.250	.8	77.663	10.382	41.006	5.482
43.0	74.349	9.939	31.970	4.274	**53.0**	77.738	10.392	41.201	5.508
.2	74.416	9.948	32.148	4.298	.2	77.805	10.401	41.392	5.533
.4	74.476	9.956	32.323	4.321	.4	77.872	10.410	41.583	5.559
.6	74.543	9.965	32.501	4.345	.6	77.947	10.420	41.780	5.585
.8	74.611	9.974	32.680	4.369	.8	78.014	10.429	41.972	5.611
44.0	74.678	9.983	32.858	4.392	**54.0**	78.089	10.439	42.168	5.637
.2	74.745	9.992	33.037	4.416	.2	78.156	10.448	42.361	5.663
.4	74.805	10.000	33.213	4.440	.4	78.231	10.458	42.558	5.689
.6	74.873	10.009	33.393	4.464	.6	78.299	10.467	42.751	5.715
.8	74.940	10.018	33.573	4.488	.8	78.366	10.476	42.945	5.741
45.0	75.007	10.027	33.753	4.512	**55.0**	78.441	10.486	43.143	5.767
.2	75.074	10.036	33.933	4.536	.2	78.508	10.495	43.336	5.793
.4	75.142	10.045	34.114	4.560	.4	78.583	10.505	43.535	5.820
.6	75.209	10.054	34.296	4.585	.6	78.658	10.515	43.734	5.846
.8	75.276	10.063	34.476	4.609	.8	78.725	10.524	43.929	5.872
46.0	75.336	10.071	34.655	4.633	**56.0**	78.800	10.534	44.128	5.899
.2	75.404	10.080	34.837	4.657	.2	78.867	10.543	44.323	5.925
.4	75.471	10.089	35.019	4.681	.4	78.942	10.553	44.523	5.952
.6	75.538	10.098	35.201	4.706	.6	79.009	10.562	44.719	5.978
.8	75.606	10.107	35.384	4.730	.8	79.084	10.572	44.920	6.005
47.0	75.673	10.116	35.566	4.754	**57.0**	79.151	10.581	45.116	6.031
.2	75.740	10.125	35.749	4.779	.2	79.226	10.591	45.317	6.058
.4	75.808	10.134	35.933	4.804	.4	79.301	10.601	45.519	6.085
.6	75.875	10.143	36.117	4.828	.6	79.368	10.610	45.716	6.111
.8	75.942	10.152	36.300	4.853	.8	79.443	10.620	45.918	6.138
48.0	76.017	10.162	36.488	4.878	**58.0**	79.518	10.630	46.120	6.165
.2	76.077	10.170	36.669	4.902	.2	79.593	10.640	46.323	6.192
.4	76.144	10.179	36.854	4.927	.4	79.660	10.649	46.521	6.219
.6	76.219	10.189	37.042	4.952	.6	79.735	10.659	46.725	6.246
.8	76.286	10.198	37.228	4.977	.8	79.810	10.669	46.928	6.273
49.0	76.354	10.207	37.413	5.001	**59.0**	79.877	10.678	47.127	6.300
.2	76.421	10.216	37.599	5.026	.2	79.952	10.688	47.332	6.327
.4	76.488	10.225	37.785	5.051	.4	80.027	10.698	47.536	6.355
.6	76.556	10.234	37.972	5.076	.6	80.101	10.708	47.740	6.382
.8	76.623	10.243	38.158	5.101	.8	80.176	10.718	47.945	6.409

TABLE 30 (*Continued*)

WEIGHT PER UNIT VOLUME AND WEIGHT OF SOLIDS (BRIX) PER UNIT VOLUME OF SUGAR SOLUTIONS AT 20° C.

Degree Brix	Weight (Pounds) in Air of		Pounds Solids (Brix) per		Degree Brix	Weight (Pounds) in Air of		Pounds Solids (Brix) per	
	1 Cu. Ft.	1 Gal.	1 Cu. Ft.	1 Gal.		1 Cu. Ft.	1 Gal.	1 Cu. Ft.	1 Gal.
60.0	80.244	10.727	48.146	6.436	**70.0**	84.036	11.234	58.825	7.864
.2	80.318	10.737	48.351	6.464	.2	84.118	11.245	59.050	7.894
.4	80.393	10.747	48.557	6.491	.4	84.193	11.255	59.272	7.924
.6	80.468	10.757	48.763	6.519	.6	84.268	11.265	59.493	7.953
.8	80.543	10.767	48.970	6.546	.8	84.350	11.276	59.720	7.983
61.0	80.618	10.777	49.177	6.574	**71.0**	84.425	11.286	59.942	8.013
.2	80.685	10.786	49.379	6.601	.2	84.507	11.297	60.169	8.043
.4	80.760	10.796	49.587	6.629	.4	84.590	11.308	60.397	8.074
.6	80.834	10.806	49.794	6.656	.6	84.664	11.318	60.619	8.104
.8	80.909	10.816	50.002	6.684	.8	84.747	11.329	60.848	8.134
62.0	80.984	10.826	50.210	6.712	**72.0**	84.822	11.339	61.072	8.164
.2	81.059	10.836	50.419	6.740	.2	84.904	11.350	61.301	8.195
.4	81.134	10.846	50.628	6.768	.4	84.979	11.360	61.525	8.225
.6	81.209	10.856	50.837	6.796	.6	85.061	11.371	61.754	8.255
.8	81.283	10.866	51.046	6.824	.8	85.143	11.382	61.984	8.286
63.0	81.358	10.876	51.256	6.852	**73.0**	85.218	11.392	62.209	8.316
.2	81.443	10.886	51.472	6.881	.2	85.300	11.403	62.440	8.347
.4	81.508	10.896	51.676	6.908	.4	85.383	11.414	62.671	8.378
.6	81.583	10.906	51.887	6.936	.6	85.457	11.424	62.896	8.408
.8	81.657	10.916	52.097	6.964	.8	85.540	11.435	63.129	8.439
64.0	81.732	10.926	52.308	6.993	**74.0**	85.622	11.446	63.360	8.470
.2	81.807	10.936	52.520	7.021	.2	85.697	11.456	63.587	8.500
.4	81.882	10.946	52.732	7.049	.4	85.779	11.467	63.820	8.531
.6	81.957	10.956	52.944	7.078	.6	85.861	11.478	64.052	8.563
.8	82.039	10.967	53.161	7.107	.8	85.944	11.489	64.286	8.594
65.0	82.114	10.977	53.374	7.135	**75.0**	86.018	11.018	64.514	8.624
.2	82.188	10.987	53.587	7.164	.2	86.101	11.510	64.748	8.656
.4	82.263	10.997	53.800	7.192	.4	86.183	11.521	64.982	8.687
.6	82.338	11.007	54.014	7.221	.6	86.265	11.532	65.216	8.718
.8	82.413	11.017	54.227	7.249	.8	86.348	11.543	65.452	8.750
66.0	82.488	11.027	54.442	7.278	**76.0**	86.430	11.554	65.687	8.781
.2	82.570	11.038	54.661	7.307	.2	86.505	11.564	65.917	8.812
.4	82.645	11.048	54.876	7.336	.4	86.587	11.575	66.146	8.842
.6	82.720	11.058	55.092	7.365	.6	86.669	11.586	66.388	8.875
.8	82.794	11.068	55.306	7.393	.8	86.752	11.597	66.626	8.907
67.0	82.877	11.079	55.528	7.423	**77.0**	86.834	11.608	66.862	8.938
.2	82.951	11.089	55.743	7.452	.2	86.916	11.619	67.099	8.970
.4	83.026	11.099	55.960	7.481	.4	86.998	11.630	67.336	9.002
.6	83.109	11.110	56.182	7.510	.6	87.081	11.641	67.575	9.033
.8	83.183	11.120	56.398	7.539	.8	87.163	11.652	67.813	9.065
68.0	83.258	11.130	56.615	7.568	**78.0**	87.238	11.663	68.046	9.096
.2	83.333	11.140	56.833	7.597	.2	87.320	11.673	68.284	9.128
.4	83.415	11.151	57.056	7.627	.4	87.402	11.684	68.523	9.160
.6	83.490	11.161	57.274	7.656	.6	87.485	11.695	68.763	9.192
.8	83.572	11.172	57.498	7.686	.8	87.567	11.706	69.003	9.224
69.0	83.647	11.182	57.716	7.716	**79.0**	87.647	11.717	69.243	9.256
.2	83.722	11.192	57.936	7.745	.2	87.732	11.728	69.484	9.289
.4	83.804	11.203	58.160	7.775	.4	87.821	11.740	69.730	9.322
.6	83.879	11.213	58.380	7.834	.6	87.904	11.751	69.972	9.354
.8	83.961	11.224	58.605	7.834	.8	87.986	11.762	70.213	9.386

TABLE 30 (*Continued*)

WEIGHT PER UNIT VOLUME AND WEIGHT OF SOLIDS (BRIX) PER UNIT VOLUME OF SUGAR SOLUTIONS AT 20° C.

Degree Brix	Weight (Pounds) in Air of		Pounds Solids (Brix) per		Degree Brix	Weight (Pounds) in Air of		Pounds Solids (Brix) per	
	1 Cu. Ft.	1 Gal.	1 Cu. Ft.	1 Gal.		1 Cu. Ft.	1 Gal.	1 Cu. Ft	1 Gal.
80.0	88.068	11.773	70.454	9.418	**88.0**	91.457	12.226	80.482	10.759
.2	88.150	11.784	70.696	9.451	.2	91.547	12.238	80.744	10.794
.4	88.233	11.795	70.939	9.483	.4	91.639	12.249	81.009	10.829
.6	88.315	11.806	71.181	9.516	.6	91.719	12.261	81.263	10.863
.8	88.397	11.817	71.425	9.548	.8	91.808	12.273	81.526	10.898
81.0	88.480	11.828	71.669	9.581	**89.0**	91.891	12.284	81.783	10.933
.2	88.562	11.839	71.912	9.613	.2	91.980	12.296	82.046	10.968
.4	88.652	11.851	72.163	9.647	.4	92.063	12.307	82.304	11.002
.6	88.732	11.862	72.405	9.679	.6	92.153	12.319	82.569	11.038
.8	88.816	11.873	72.651	9.712	.8	92.242	12.331	82.833	11.073
82.0	88.898	11.884	72.896	9.745	**90.0**	92.325	12.342	83.092	11.108
.2	88.981	11.895	73.142	9.778	.2	92.414	12.354	83.357	11.143
.4	89.071	11.907	73.395	9.811	.4	92.504	12.366	83.624	11.179
.6	89.153	11.918	73.640	9.844	.6	92.594	12.378	83.890	11.214
.8	89.235	11.929	73.887	9.877	.8	92.676	12.389	84.150	11.249
83.0	89.317	11.940	74.133	9.910	**91.0**	92.766	12.401	84.417	11.285
.2	89.407	11.952	74.387	9.944	.2	92.856	12.413	84.685	11.321
.4	89.489	11.963	74.634	9.977	.4	92.945	12.425	84.952	11.356
.6	89.572	11.974	74.882	10.010	.6	93.035	12.437	85.220	11.392
.8	89.662	11.986	75.137	10.044	.8	93.118	12.448	85.482	11.427
84.0	89.744	11.997	75.385	10.078	**92.0**	93.207	12.460	85.750	11.463
.2	89.826	12.008	75.633	10.111	.2	93.297	12.472	86.020	11.499
.4	89.916	12.020	75.889	10.145	.4	93.387	12.484	86.289	11.535
.6	89.998	12.031	76.138	10.178	.6	93.477	12.496	86.560	11.571
.8	90.080	12.042	76.388	10.212	.8	93.559	12.507	86.823	11.607
85.0	90.170	12.054	76.645	10.246	**93.0**	93.649	12.519	87.094	11.643
.2	90.252	12.065	76.895	10.279	.2	93.738	12.531	87.364	11.679
.4	90.342	12.077	77.152	10.314	.4	93.828	12.543	87.635	11.715
.6	90.425	12.088	77.404	10.347	.6	93.918	12.555	87.907	11.750
.8	90.507	12.099	77.655	10.381	.8	94.008	12.567	88.180	11.788
86.0	90.597	12.111	77.913	10.415	**94.0**	94.097	12.579	88.451	11.824
.2	90.679	12.122	78.165	10.449	.2	94.187	12.591	88.724	11.861
.4	90.769	12.134	78.424	10.484	.4	94.277	12.603	88.997	11.895
.6	90.851	12.145	78.677	10.518	.6	94.367	12.615	89.271	11.934
.8	90.941	12.157	78.937	10.552	.8	94.457	12.627	89.545	11.971
87.0	91.023	12.168	79.190	10.586	**95.0**	94.546	12.639	89.819	12.007
.2	91.113	12.180	79.451	10.621					
.4	91.195	12.191	79.704	10.655					
.6	91.285	12.203	79.966	10.690					
.8	91.375	12.215	80.227	10.725					

TABLE 31

BRIX, SPECIFIC GRAVITY, AND WEIGHT PER UNIT VOLUME 27½°/27½° C.

Brix	Specific Gravity *	Weight (Pounds †)		Pounds Solids ‡ (Brix) per	
		1 Cu. Ft.	1 Gal.	1 Cu. Ft.	1 Gal.
0.0	1.00000	62.138	8.307	0.000	0.000
.2	1.00078	62.186	8.313	.124	.017
.4	1.00155	62.234	8.320	.249	.033
.6	1.00233	62.283	8.326	.374	.050
.8	1.00311	62.331	8.333	.499	.067
1.0	1.00389	62.380	8.339	.624	.083
.2	1.00466	62.428	8.346	.749	.100
.4	1.00544	62.476	8.352	.875	.117
.6	1.00622	62.524	8.359	1.000	.134
.8	1.00700	62.573	8.365	1.126	.151
2.0	1.00778	62.621	8.371	1.252	.167
.2	1.00856	62.670	8.378	1.379	.184
.4	1.00934	62.718	8.385	1.505	.201
.6	1.01013	62.767	8.391	1.632	.218
.8	1.01091	62.816	8.398	1.759	.235
3.0	1.01169	62.864	8.404	1.886	.252
.2	1.01248	62.913	8.411	2.013	.269
.4	1.01327	62.963	8.417	2.141	.286
.6	1.01406	63.012	8.424	2.268	.303
.8	1.01485	63.060	8.430	2.396	.320
4.0	1.01564	63.110	8.437	2.524	.338
.2	1.01643	63.159	8.443	2.653	.355
.4	1.01722	63.208	8.450	2.781	.372
.6	1.01802	63.258	8.457	2.910	.389
.8	1.01881	63.307	8.463	3.039	.406
5.0	1.01960	63.356	8.470	3.168	.424
.2	1.02040	63.406	8.476	3.297	.441
.4	1.02120	63.455	8.483	3.427	.458
.6	1.02200	63.505	8.490	3.556	.475
.8	1.02280	63.555	8.496	3.686	.493
6.0	1.02360	63.604	8.503	3.816	.510
.2	1.02440	63.654	8.510	3.947	.528
.4	1.02521	63.704	8.516	4.077	.545
.6	1.02601	63.754	8.523	4.208	.563
.8	1.02682	63.805	8.530	4.339	.580
7.0	1.02762	63.854	8.536	4.470	.598
.2	1.02843	63.905	8.543	4.601	.615
.4	1.02924	63.955	8.550	4.733	.633
.6	1.03005	64.005	8.557	4.864	.650
.8	1.03086	64.056	8.563	4.996	.668
8.0	1.03167	64.106	8.570	5.128	.686
.2	1.03248	64.156	8.577	5.261	.703
.4	1.03330	64.207	8.584	5.393	.721
.6	1.03411	64.258	8.590	5.529	.739
.8	1.03493	64.308	8.597	5.659	.757
9.0	1.03574	64.359	8.604	5.792	.774
.2	1.03656	64.410	8.611	5.926	.792
.4	1.03738	64.461	8.618	6.059	.810
.6	1.03820	64.512	8.624	6.193	.828
.8	1.03902	64.563	8.631	6.327	.846

* Calculated from the table of the Kaiserliche Normaleichungskommission to apparent degrees Brix and specific gravity of sugar solutions weighed in air with brass weights at 27½° C.

† Calculated (1943) from Brix and specific gravity figures for weights of water 62.138 lb. per cu. ft. and 8.307 lb. per gal.

‡ Calculated (1943) from Brix and weight per unit volume.

TABLE 31 (*Continued*)

Brix, Specific Gravity, and Weight per Unit Volume 27½°/27½° C.

Brix	Specific Gravity	Weight (Pounds)		Pounds Solids (Brix) per	
		1 Cu. Ft.	1 Gal.	1 Cu. Ft.	1 Gal.
10.0	1.03984	64.614	8.638	6.461	0.864
.2	1.04067	64.665	8.645	6.596	.882
.4	1.04149	64.716	8.652	6.730	.900
.6	1.04232	64.768	8.658	6.865	.918
.8	1.04314	64.819	8.665	7.000	.936
11.0	1.04397	64.870	8.672	7.136	.954
.2	1.04480	64.922	8.679	7.271	.972
.4	1.04564	64.974	8.686	7.407	.990
.6	1.04647	65.026	8.693	7.543	1.008
.8	1.04730	65.077	8.700	7.679	1.027
12.0	1.04813	65.129	8.706	7.815	1.045
.2	1.04897	65.181	8.713	7.952	1.063
.4	1.04981	65.233	8.720	8.089	1.081
.6	1.05065	65.285	8.728	8.226	1.100
.8	1.05148	65.337	8.735	8.363	1.118
13.0	1.05232	65.389	8.741	8.501	1.136
.2	1.05317	65.442	8.748	8.638	1.155
.4	1.05401	65.494	8.755	8.776	1.173
.6	1.05485	65.546	8.762	8.914	1.192
.8	1.05570	65.599	8.769	9.053	1.210
14.0	1.05654	65.651	8.776	9.191	1.229
.2	1.05739	65.704	8.783	9.330	1.247
.4	1.05824	65.757	8.790	9.469	1.266
.6	1.05909	65.810	8.797	9.608	1.284
.8	1.05994	65.863	8.805	9.748	1.303
15.0	1.06080	65.916	8.812	9.887	1.322
.2	1.06165	65.969	8.819	10.027	1.341
.4	1.06251	66.022	8.826	10.167	1.359
.6	1.06336	66.075	8.833	10.308	1.378
.8	1.06422	66.129	8.840	10.448	1.397
16.0	1.06508	66.182	8.847	10.589	1.416
.2	1.06594	66.235	8.854	10.730	1.434
.4	1.06680	66.289	8.861	10.871	1.453
.6	1.06767	66.343	8.869	11.013	1.472
.8	1.06853	66.396	8.876	11.155	1.491
17.0	1.06940	66.450	8.883	11.297	1.510
.2	1.07026	66.504	8.890	11.439	1.529
.4	1.07113	66.558	8.897	11.581	1.548
.6	1.07200	66.612	8.905	11.724	1.567
.8	1.07287	66.666	8.912	11.867	1.586
18.0	1.07374	66.720	8.919	12.010	1.604
.2	1.07461	66.774	8.926	12.153	1.625
.4	1.07549	66.829	8.933	12.297	1.644
.6	1.07636	66.883	8.941	12.440	1.663
.8	1.07724	66.938	8.949	12.583	1.682
19.0	1.07812	66.992	8.956	12.728	1.702
.2	1.07900	67.047	8.963	12.873	1.721
.4	1.07988	67.102	8.971	13.018	1.740
.6	1.08076	67.156	8.978	13.163	1.760
.8	1.08164	67.211	8.985	13.308	1.779

TABLE 31 (*Continued*)

Brix, Specific Gravity, and Weight per Unit Volume 27½°/27½° C.

Brix	Specific Gravity	Weight (Pounds)		Pounds Solids (Brix) per	
		1 Cu. Ft.	1 Gal.	1 Cu. Ft.	1 Gal.
20.0	1.08253	67.266	8.992	13.453	1.798
.2	1.08341	67.321	9.000	13.599	1.818
.4	1.08430	67.376	9.007	13.745	1.837
.6	1.08519	67.432	9.014	13.891	1.857
.8	1.08608	67.487	9.022	14.037	1.877
21.0	1.08697	67.542	9.029	14.184	1.896
.2	1.08786	67.597	9.037	14.331	1.916
.4	1.08876	87.653	9.044	14.478	1.935
.6	1.08965	67.709	9.052	14.625	1.955
.8	1.09055	67.765	9.059	14.773	1.975
22.0	1.09144	67.820	9.066	14.920	1.995
.2	1.09234	67.876	9.074	15.068	2.014
.4	1.09324	67.932	9.082	15.217	2.034
.6	1.09414	67.988	9.089	15.365	2.054
.8	1.09504	68.044	9.096	15.514	2.074
23.0	1.09595	68.100	9.104	15.663	2.094
.2	1.09685	68.156	9.112	15.812	2.114
.4	1.09776	68.213	9.119	15.962	2.134
.6	1.09867	68.269	9.127	16.111	2.154
.8	1.09957	68.325	9.134	16.261	2.174
24.0	1.10048	68.382	9.141	16.412	2.194
.2	1.10140	68.439	9.149	16.562	2.214
.4	1.10231	68.495	9.157	16.713	2.234
.6	1.10322	68.552	9.164	16.864	2.254
.8	1.10414	68.609	9.172	17.015	2.275
25.0	1.10551	68.666	9.179	17.167	2.295
.2	1.10597	68.723	9.187	17.318	2.315
.4	1.10689	68.780	9.195	17.470	2.336
.6	1.10781	68.837	9.203	17.622	2.356
.8	1.10873	68.894	9.210	17.775	2.376
26.0	1.10965	68.952	9.218	17.928	2.397
.2	1.11058	69.009	9.226	18.080	2.417
.4	1.11151	69.067	9.233	18.234	2.438
.6	1.11244	69.125	9.241	18.387	2.458
.8	1.11336	69.182	9.249	18.541	2.479
27.0	1.11429	69.239	9.256	18.695	2.499
.2	1.11522	69.298	9.264	18.849	2.520
.4	1.11616	69.356	9.272	19.004	2.541
.6	1.11709	69.414	9.280	19.158	2.561
.8	1.11802	69.472	9.287	19.313	2.582
28.0	1.11896	69.530	9.295	19.468	2.603
.2	1.11989	69.588	9.303	19.624	2.623
.4	1.12083	69.646	9.311	19.779	2.644
.6	1.12177	69.705	9.318	19.936	2.665
.8	1.12271	69.763	9.326	20.092	2.686
29.0	1.12365	69.821	9.334	20.248	2.707
.2	1.12460	69.880	9.342	20.405	2.728
.4	1.12555	69.939	9.350	20.562	2.749
.6	1.12649	69.998	9.358	20.719	2.770
.8	1.12744	70.057	9.366	20.877	2.791

TABLE 31 (*Continued*)

Brix, Specific Gravity, and Weight per Unit Volume 27½°/27½° C.

Brix	Specific Gravity	Weight (Pounds)		Pounds Solids (Brix) per	
		1 Cu. Ft.	1 Gal.	1 Cu. Ft.	1 Gal.
30.0	1.12839	70.116	9.373	21.035	2.812
.2	1.12934	70.175	9.381	21.193	2.833
.4	1.13030	70.235	9.389	21.351	2.854
.6	1.13125	70.294	9.397	21.510	2.876
.8	1.13221	70.353	9.405	21.669	2.897
31.0	1.13316	70.412	9.413	21.828	2.918
.2	1.13412	70.472	9.421	21.988	2.939
.4	1.13508	70.532	9.429	22.147	2.961
.6	1.13604	70.591	9.437	22.307	2.982
.8	1.13700	70.651	9.445	22.467	3.004
32.0	1.13797	70.711	9.453	22.628	3.025
.2	1.13893	70.771	9.461	22.788	3.046
.4	1.13990	70.831	9.469	22.949	3.068
.6	1.14087	70.891	9.477	23.110	3.090
.8	1.14184	70.952	9.485	23.272	3.111
33.0	1.14281	71.012	9.493	23.434	3.133
.2	1.14378	71.072	9.501	23.596	3.154
.4	1.14476	71.133	9.509	23.758	3.176
.6	1.14573	71.193	9.518	23.921	3.198
.8	1.14671	71.254	9.526	24.084	3.220
34.0	1.14768	71.315	9.534	24.247	3.242
.2	1.14866	71.375	9.542	24.410	3.263
.4	1.14965	71.437	9.550	24.574	3.285
.6	1.15063	71.498	9.558	24.738	3.307
.8	1.15161	71.559	9.566	24.903	3.329
35.0	1.15259	71.620	9.574	25.067	3.351
.2	1.15358	71.681	9.583	25.232	3.373
.4	1.15457	71.743	9.591	25.397	3.395
.6	1.15556	71.804	9.599	25.562	3.417
.8	1.15655	71.866	9.607	25.728	3.439
36.0	1.15753	71.927	9.616	25.894	3.462
.2	1.15853	71.989	9.624	26.060	3.484
.4	1.15952	72.050	9.632	26.226	3.506
.6	1.16052	72.112	9.640	26.393	3.528
.8	1.16152	72.174	9.649	26.560	3.551
37.0	1.16251	72.236	9.657	26.727	3.573
.2	1.16351	72.298	9.665	26.895	3.595
.4	1.16454	72.362	9.674	27.063	3.618
.6	1.16552	72.423	9.682	27.231	3.640
.8	1.16652	72.485	9.690	27.399	3.663
38.0	1.16753	72.548	9.699	27.568	3.686
.2	1.16854	72.611	9.707	27.737	3.708
.4	1.16955	72.673	9.716	27.906	3.731
.6	1.17056	72.736	9.724	28.076	3.754
.8	1.17157	72.799	9.732	28.246	3.776
39.0	1.17258	72.862	9.741	28.416	3.798
.2	1.17360	72.925	9.749	28.587	3.822
.4	1.17462	72.989	9.757	28.758	3.844
.6	1.17564	73.052	9.765	28.929	3.867
.8	1.17666	73.115	9.773	29.100	3.890

TABLE 31 (*Continued*)

Brix, Specific Gravity, and Weight per Unit Volume 27½°/27½° C.

Brix	Specific Gravity	Weight (Pounds)		Pounds Solids (Brix) per	
		1 Cu. Ft.	1 Gal.	1 Cu. Ft.	1 Gal.
40.0	1.17768	73.179	9.783	29.272	3.913
.2	1.17870	73.242	9.791	29.443	3.936
.4	1.17973	73.306	9.800	29.616	3.959
.6	1.18076	73.370	9.809	29.788	3.983
.8	1.18178	73.433	9.817	29.961	4.005
41.0	1.18281	73.497	9.826	30.134	4.029
.2	1.18384	73.561	9.834	30.307	4.052
.4	1.18488	73.626	9.843	30.481	4.075
.6	1.18592	73.691	9.851	30.655	4.098
.8	1.18695	73.755	9.860	30.830	4.122
42.0	1.18799	73.819	9.869	31.004	4.145
.2	1.18903	73.884	9.877	31.179	4.168
.4	1.19007	73.949	9.886	31.354	4.192
.6	1.19112	74.014	9.895	31.530	4.215
.8	1.19216	74.078	9.903	31.705	4.239
43.0	1.19320	74.143	9.912	31.881	4.262
.2	1.19425	74.208	9.921	32.058	4.286
.4	1.19530	74.274	9.929	32.235	4.309
.6	1.19635	74.339	9.938	32.412	4.333
.8	1.19740	74.404	9.947	32.589	4.357
44.0	1.19845	74.469	9.956	32.766	4.381
.2	1.19951	74.535	9.964	32.944	4.404
.4	1.20057	74.601	9.973	33.123	4.428
.6	1.20163	74.667	9.982	33.301	4.452
.8	1.20269	74.733	9.991	33.480	4.476
45.0	1.20375	74.799	10.000	33.660	4.500
.2	1.20481	74.864	10.008	33.839	4.524
.4	1.20588	74.931	10.017	34.018	4.548
.6	1.20695	74.997	10.026	34.199	4.572
.8	1.20801	75.063	10.035	34.379	4.596
46.0	1.20908	75.130	10.044	34.560	4.620
.2	1.21015	75.196	10.053	34.741	4.645
.4	1.21122	75.263	10.062	34.922	4.669
.6	1.21230	75.330	10.071	35.104	4.693
.8	1.21337	75.396	10.079	35.285	4.717
47.0	1.21445	75.463	10.088	35.468	4.741
.2	1.21553	75.531	10.097	35.651	4.766
.4	1.21661	75.598	10.106	35.833	4.790
.6	1.21769	75.665	10.115	36.017	4.816
.8	1.21877	75.732	10.124	36.200	4.839
48.0	1.21985	75.799	10.133	36.384	4.864
.2	1.22094	75.867	10.142	36.568	4.888
.4	1.22203	75.934	10.151	36.752	4.913
.6	1.22311	76.002	10.160	36.937	4.938
.8	1.22420	76.069	10.169	37.122	4.963
49.0	1.22529	76.137	10.178	37.307	4.987
.2	1.22639	76.205	10.188	37.493	5.013
.4	1.22748	76.273	10.197	37.679	5.037
.6	1.22858	76.342	10.206	37.866	5.062
.8	1.22968	76.410	10.215	38.052	5.087

TABLE 31 (*Continued*)

Brix, Specific Gravity, and Weight per Unit Volume 27½°/27½° C.

Brix	Specific Gravity	Weight (Pounds)		Pounds Solids (Brix) per	
		1 Cu. Ft.	1 Gal.	1 Cu. Ft.	1 Gal.
50.0	1.23077	76.478	10.224	38.239	5.112
.2	1.23187	76.546	10.233	38.426	5.137
.4	1.23298	76.615	10.242	38.614	5.162
.6	1.23408	76.683	10.252	38.802	5.188
.8	1.23518	76.752	10.261	38.990	5.213
51.0	1.23629	76.821	10.270	39.179	5.238
.2	1.23740	76.890	10.279	39.368	5.263
.4	1.23851	76.959	10.288	39.557	5.288
.6	1.23962	77.028	10.298	39.746	5.314
.8	1.24073	77.096	10.307	39.936	5.339
52.0	1.24184	77.165	10.316	40.126	5.364
.2	1.24295	77.234	10.325	40.316	5.390
.4	1.24407	77.304	10.334	40.507	5.415
.6	1.24519	77.374	10.344	40.699	5.441
.8	1.24631	77.444	10.353	40.890	5.466
53.0	1.24743	77.513	10.362	41.082	5.492
.2	1.24855	77.583	10.372	41.274	5.518
.4	1.24968	77.653	10.381	41.467	5.544
.6	1.25080	77.723	10.390	41.660	5.569
.8	1.25193	77.793	10.400	41.853	5.595
54.0	1.25305	77.863	10.409	42.046	5.621
.2	1.25419	77.933	10.419	42.240	5.647
.4	1.25532	78.004	10.428	42.434	5.673
.6	1.25645	78.074	10.437	42.628	5.699
.8	1.25758	78.144	10.447	42.823	5.725
55.0	1.25872	78.215	10.456	43.018	5.751
.2	1.25986	78.286	10.466	43.214	5.777
.4	1.26100	78.357	10.475	43.410	5.803
.6	1.26214	78.427	10.485	43.605	5.830
.8	1.26328	78.498	10.494	43.802	5.856
56.0	1.26443	78.570	10.504	44.000	5.882
.2	1.26557	78.641	10.513	44.196	5.908
.4	1.26672	78.712	10.523	44.394	5.935
.6	1.26787	78.784	10.532	44.592	5.961
.8	1.26902	78.855	10.542	44.790	5.988
57.0	1.27017	78.926	10.551	44.988	6.014
.2	1.27133	78.999	10.561	45.187	6.041
.4	1.27249	79.071	10.571	45.387	6.068
.6	1.27364	79.142	10.580	45.586	6.094
.8	1.27480	79.214	10.590	45.786	6.121
58.0	1.27596	79.286	10.599	45.986	6.147
.2	1.27712	79.358	10.609	46.186	6.174
.4	1.27829	79.431	10.619	46.388	6.202
.6	1.27945	79.503	10.628	46.589	6.228
.8	1.28061	79.575	10.638	46.790	6.255
59.0	1.28178	79.648	10.648	46.992	6.282
.2	1.28295	79.721	10.657	47.195	6.309
.4	1.28412	79.793	10.667	47.397	6.336
.6	1.28530	79.867	10.677	47.601	6.364
.8	1.28647	79.939	10.687	47.804	6.391

TABLE 31 (*Continued*)

Brix, Specific Gravity, and Weight per Unit Volume 27½°/27½° C.

Brix	Specific Gravity	Weight (Pounds)		Pounds Solids (Brix) per	
		1 Cu. Ft.	1 Gal.	1 Cu. Ft.	1 Gal.
60.0	1.27864	80.011	10.696	48.007	6.418
.2	1.28882	80.085	10.706	48.211	6.445
.4	1.29000	80.159	10.716	48.416	6.473
.6	1.29118	80.232	10.726	48.621	6.500
.8	1.29236	80.305	10.736	48.825	6.528
61.0	1.29354	80.379	10.745	49.031	6.554
.2	1.29473	80.453	10.755	49.237	6.582
.4	1.29592	80.526	10.765	49.443	6.610
.6	1.29710	80.600	10.775	49.650	6.637
.8	1.29829	80.674	10.785	49.857	6.665
62.0	1.29948	80.748	10.795	50.064	6.693
.2	1.30067	80.822	10.805	50.271	6.721
.4	1.30187	80.896	10.815	50.479	6.749
.6	1.30307	80.970	10.825	50.687	6.776
.8	1.30426	81.045	10.834	50.896	6.804
63.0	1.30546	81.119	10.844	51.105	6.832
.2	1.30666	81.194	10.854	51.315	6.860
.4	1.30786	81.268	10.864	51.524	6.888
.6	1.30907	81.344	10.874	51.735	6.916
.8	1.31027	81.418	10.884	51.945	6.944
64.0	1.31147	81.493	10.894	52.156	6.972
.2	1.31269	81.569	10.905	52.367	7.001
.4	1.31390	81.644	10.915	52.579	7.029
.6	1.31511	81.719	10.925	52.790	7.058
.8	1.31632	81.794	10.935	53.003	7.086
65.0	1.31753	81.869	10.945	53.215	7.114
.2	1.31875	81.945	10.955	53.428	7.143
.4	1.31997	82.021	10.965	53.642	7.171
.6	1.32119	82.097	10.975	53.856	7.200
.8	1.32240	82.172	10.985	54.069	7.228
66.0	1.32362	82.248	10.995	54.284	7.257
.2	1.32485	82.324	11.006	54.498	7.286
.4	1.32608	82.401	11.016	54.714	7.315
.6	1.32730	82.476	11.026	54.929	7.343
.8	1.32853	82.553	11.036	55.145	7.372
67.0	1.32975	82.629	11.046	55.361	7.401
.2	1.33099	82.706	11.057	55.578	7.430
.4	1.33222	82.782	11.067	55.795	7.459
.6	1.33346	82.859	11.077	56.013	7.488
.8	1.33469	82.936	11.087	56.231	7.517
68.0	1.33593	83.013	11.098	56.449	7.547
.2	1.33717	83.090	11.108	56.667	7.576
.4	1.33841	83.167	11.118	56.886	7.605
.6	1.33965	83.244	11.128	57.105	7.634
.8	1.34089	83.320	11.139	57.324	7.664
69.0	1.34213	83.398	11.149	57.545	7.693
.2	1.34338	83.476	11.159	57.765	7.722
.4	1.34463	83.553	11.170	57.986	7.752
.6	1.34588	83.631	11.180	58.207	7.781
.8	1.34713	83.709	11.191	58.429	7.811

TABLE 31 (*Continued*)

Brix, Specific Gravity, and Weight per Unit Volume 27½°/27½° C.

Brix	Specific Gravity	Weight (Pounds)		Pounds Solids (Brix) per	
		1 Cu. Ft.	1 Gal.	1 Cu. Ft.	1 Gal.
70.0	1.34838	83.786	11.201	58.650	7.841
.2	1.34964	83.865	11.211	58.873	7.870
.4	1.35090	83.943	11.222	59.096	7.900
.6	1.35216	84.021	11.232	59.319	7.930
.8	1.35341	84.099	11.243	59.542	7.960
71.0	1.35467	84.177	11.253	59.766	7.990
.2	1.35593	84.255	11.264	59.990	8.020
.4	1.35720	84.334	11.274	60.214	8.050
.6	1.35846	84.413	11.285	60.440	8.080
.8	1.35973	84.492	11.295	60.665	8.110
72.0	1.36099	84.570	11.306	60.890	8.140
.2	1.36226	84.649	11.316	61.112	8.170
.4	1.36354	84.728	11.327	61.343	8.201
.6	1.36481	84.807	11.337	61.570	8.231
.8	1.36608	84.886	11.348	61.797	8.261
73.0	1.36736	84.966	11.359	62.025	8.292
.2	1.36864	85.045	11.369	62.253	8.322
.4	1.36992	85.125	11.380	62.482	8.353
.6	1.37120	85.204	11.390	62.710	8.383
.8	1.37248	85.284	11.401	62.940	8.414
74.0	1.37376	85.363	11.412	63.169	8.445
.2	1.37504	85.443	11.422	63.399	8.475
.4	1.37633	85.523	11.433	63.629	8.506
.6	1.37762	85.603	11.444	63.860	8.537
.8	1.37890	85.683	11.455	64.091	8.568
75.0	1.38020	85.763	11.465	64.322	8.599
.2	1.38149	85.844	11.476	64.555	8.630
.4	1.38279	85.924	11.487	64.787	8.661
.6	1.38409	86.005	11.498	65.020	8.693
.8	1.38538	86.079	11.508	65.248	8.723
76.0	1.38668	86.166	11.519	65.486	8.754
.2	1.38798	86.247	11.530	65.720	8.786
.4	1.38928	86.328	11.541	65.955	8.817
.6	1.39058	86.408	11.552	66.189	8.849
.8	1.39188	86.489	11.562	66.424	8.880
77.0	1.39319	86.571	11.573	66.660	8.911
.2	1.39450	86.652	11.584	66.895	8.943
.4	1.39581	86.733	11.595	67.131	8.975
.6	1.39712	86.815	11.606	67.368	9.006
.8	1.39843	86.896	11.617	67.605	9.038
78.0	1.39974	86.978	11.628	67.843	9.070
.2	1.40106	87.060	11.639	68.081	9.102
.4	1.40238	87.142	11.650	68.319	9.134
.6	1.40370	87.224	11.661	68.558	9.166
.8	1.40501	87.305	11.671	68.796	9.197
79.0	1.40633	87.387	11.682	69.036	9.229
.2	1.40766	87.470	11.693	69.276	9.261
.4	1.40898	87.552	11.704	69.516	9.293
.6	1.41031	87.634	11.715	69.757	9.325
.8	1.41163	87.716	11.726	69.997	9.357

TABLE 31 (*Continued*)

Brix, Specific Gravity, and Weight per Unit Volume 27½°/27½° C.

Brix	Specific Gravity	Weight (Pounds)		Pounds Solids (Brix) per	
		1 Cu. Ft.	1 Gal.	1 Cu. Ft.	1 Gal.
80.0	1.41296	87.799	11.737	70.239	9.390
.2	1.41429	87.882	11.749	70.481	9.423
.4	1.41562	87.964	11.760	70.723	9.455
.6	1.41696	88.048	11.771	70.967	9.487
.8	1.41829	88.130	11.782	71.209	9.520
81.0	1.41962	88.213	11.793	71.453	9.552
.2	1.42096	88.296	11.804	71.696	9.585
.4	1.42231	88.380	11.815	71.941	9.617
.6	1.42365	88.463	11.826	72.186	9.650
.8	1.42499	88.547	11.837	72.431	9.683
82.0	1.42633	88.630	11.849	72.677	9.716
.2	1.42768	88.714	11.860	72.923	9.749
.4	1.42902	88.797	11.871	73.169	9.782
.6	1.43037	88.881	11.882	73.416	9.815
.8	1.43172	88.965	11.893	73.663	9.847
83.0	1.43307	89.049	11.905	73.911	9.881
.2	1.43442	89.133	11.916	74.159	9.914
.4	1.43578	89.217	11.927	74.407	9.947
.6	1.43713	89.301	11.938	74.656	9.980
.8	1.43849	89.386	11.950	74.905	10.014
84.0	1.43985	89.470	11.961	75.155	10.047
.2	1.44121	89.555	11.972	75.405	10.080
.4	1.44257	89.639	11.983	75.655	10.114
.6	1.44393	89.724	11.995	75.906	10.148
.8	1.44530	89.809	12.006	76.158	10.181
85.0	1.44666	89.893	12.017	76.409	10.214
.2	1.44803	89.978	12.029	76.661	10.249
.4	1.44940	90.063	12.040	76.914	10.282
.6	1.45077	90.149	12.052	77.168	10.317
.8	1.45214	90.234	12.063	77.421	10.350
86.0	1.45351	90.319	12.074	77.674	10.384
.2	1.45489	90.405	12.086	77.929	10.418
.4	1.45627	90.490	12.097	78.183	10.452
.6	1.45764	90.575	12.109	78.438	10.486
.8	1.45902	90.661	12.120	78.694	10.520
87.0	1.46040	90.747	12.132	78.950	10.555
.2	1.46178	90.833	12.143	79.206	10.589
.4	1.46317	90.919	12.155	79.463	10.624
.6	1.46455	91.005	12.166	79.720	10.657
.8	1.46594	91.091	12.178	79.978	10.692
88.0	1.46732	91.177	12.189	80.236	10.726
.2	1.46872	91.263	12.201	80.494	10.761
.4	1.47011	91.350	12.212	80.753	10.795
.6	1.47150	91.437	12.224	81.013	10.831
.8	1.47289	91.523	12.235	81.272	10.865
89.0	1.47428	91.609	12.247	81.532	10.900
.2	1.47568	91.696	12.258	81.793	10.934
.4	1.47708	91.783	12.270	82.054	10.969
.6	1.47848	91.870	12.282	82.322	11.005
.8	1.47988	91.957	12.293	82.577	11.039

TABLE 31 (*Continued*)

Brix, Specific Gravity, and Weight per Unit Volume 27½°/27½° C.

Brix	Specific Gravity	Weight (Pounds)		Pounds Solids (Brix) per	
		1 Cu. Ft.	1 Gal.	1 Cu. Ft.	1 Gal.
90.0	1.48128	92.044	12.305	82.840	11.075
.2	1.48269	92.132	12.317	83.103	11.110
.4	1.48409	92.219	12.328	83.366	11.145
.6	1.48550	92.307	12.340	83.630	11.180
.8	1.48691	92.394	12.352	83.894	11.216
91.0	1.48831	92.481	12.363	84.158	11.250
.2	1.48973	92.569	12.375	84.423	11.286
.4	1.49114	92.657	12.387	84.688	11.322
.6	1.49255	92.745	12.399	84.954	11.358
.8	1.49397	92.833	12.410	85.221	11.392
92.0	1.49538	92.921	12.422	85.487	11.428
.2	1.49680	93.009	12.434	85.754	11.464
.4	1.49822	93.097	12.446	86.022	11.500
.6	1.49964	93.185	12.458	86.289	11.536
.8	1.50107	93.274	12.469	86.558	11.571
93.0	1.50249	93.362	12.481	86.827	11.607
.2	1.50391	93.451	12.493	87.096	11.644
.4	1.50534	93.539	12.505	87.365	11.680
.6	1.50677	93.628	12.517	87.636	11.716
.8	1.50820	93.717	12.529	87.907	11.752
94.0	1.50962	93.805	12.540	88.177	11.788
.2	1.51106	93.895	12.552	88.449	11.824
.4	1.51249	93.984	12.564	88.721	11.860
.6	1.51393	94.073	12.576	88.993	11.897
.8	1.51536	94.162	12.588	89.266	11.933
95.0	1.51680	94.252	12.600	89.539	11.970
.2	1.51824	94.341	12.612	89.813	12.007
.4	1.51968	94.430	12.624	90.086	12.043
.6	1.52112	94.520	12.636	90.361	12.080
.8	1.52256	94.609	12.648	90.635	12.117
96.0	1.52401	94.700	12.660	90.923	12.154
.2	1.52546	94.790	12.672	91.188	12.191
.4	1.52690	94.879	12.684	91.463	12.227
.6	1.52835	94.969	12.696	91.740	12.264
.8	1.52980	95.059	12.708	92.017	12.301
97.0	1.53125	95.149	12.720	92.295	12.339
.2	1.53271	95.240	12.732	92.573	12.376
.4	1.53416	95.330	12.744	92.851	12.413
.6	1.53562	95.421	12.756	93.131	12.450
.8	1.53708	95.512	12.769	93.411	12.488
98.0	1.53853	95.602	12.781	93.690	12.525
.2	1.53999	95.693	12.793	93.971	12.563
.4	1.54146	95.784	12.805	94.251	12.600
.6	1.54292	95.875	12.817	94.533	12.638
.8	1.54438	95.965	12.829	94.813	12.675
99.0	1.54584	96.056	12.841	95.095	12.713
.2	1.54731	96.147	12.854	95.378	12.751
.4	1.54878	96.239	12.866	95.662	12.789
.6	1.55025	96.330	12.878	95.945	12.827
.8	1.55172	96.421	12.890	96.228	12.864
100.0	1.55319	96.513	12.902	96.513	12.902

TABLE 32

TEMPERATURE CORRECTIONS FOR BRIX READINGS

STANDARD 27½° C.

Temp., °C.	Observed Degrees Brix													
	0	5	10	15	20	25	30	35	40	45	50	55	60	70
	Subtract from the Readings													
10	0.75	0.82	0.89	0.96	1.02	1.08	1.14	1.20	1.24	1.28	1.31	1.33	1.35	1.37
11	0.74	0.79	0.86	0.92	0.98	1.03	1.09	1.14	1.18	1.21	1.24	1.26	1.28	1.30
12	0.72	0.76	0.82	0.88	0.93	0.98	1.04	1.08	1.12	1.14	1.17	1.19	1.20	1.22
13	0.69	0.73	0.78	0.84	0.88	0.93	0.98	1.02	1.06	1.07	1.10	1.12	1.13	1.15
14	0.66	0.70	0.74	0.79	0.83	0.88	0.92	0.96	0.99	1.00	1.03	1.05	1.06	1.07
15	0.63	0.66	0.70	0.74	0.78	0.82	0.86	0.89	0.92	0.93	0.95	0.97	0.98	0.99
16	0.60	0.62	0.66	0.69	0.73	0.76	0.80	0.83	0.86	0.86	0.88	0.90	0.91	0.92
17	0.56	0.58	0.61	0.64	0.68	0.70	0.74	0.77	0.79	0.79	0.81	0.83	0.83	0.84
18	0.52	0.54	0.56	0.59	0.62	0.64	0.68	0.70	0.72	0.72	0.74	0.75	0.75	0.76
19	0.48	0.49	0.51	0.54	0.56	0.58	0.61	0.63	0.65	0.65	0.67	0.68	0.68	0.68
20	0.43	0.44	0.46	0.48	0.50	0.52	0.54	0.56	0.58	0.58	0.59	0.60	0.60	0.60
21	0.38	0.39	0.41	0.42	0.44	0.46	0.47	0.49	0.51	0.51	0.51	0.52	0.52	0.52
22	0.33	0.34	0.35	0.36	0.38	0.40	0.40	0.42	0.43	0.43	0.43	0.44	0.44	0.44
23	0.28	0.28	0.29	0.30	0.32	0.33	0.33	0.35	0.36	0.36	0.36	0.36	0.36	0.36
24	0.22	0.22	0.23	0.24	0.25	0.26	0.26	0.27	0.28	0.28	0.28	0.28	0.28	0.28
25	0.16	0.16	0.17	0.18	0.18	0.19	0.19	0.20	0.20	0.20	0.20	0.20	0.20	0.20
26	0.10	0.10	0.11	0.11	0.11	0.12	0.12	0.12	0.12	0.12	0.12	0.12	0.12	0.12
27	0.03	0.03	0.04	0.04	0.04	0.04	0.04	0.04	0.04	0.04	0.04	0.04	0.04	0.04
	Add to the Readings													
28	0.04	0.04	0.04	0.04	0.04	0.04	0.04	0.04	0.04	0.04	0.04	0.04	0.04	0.04
29	0.11	0.11	0.11	0.11	0.11	0.11	0.12	0.12	0.12	0.12	0.12	0.12	0.12	0.12
30	0.18	0.18	0.18	0.18	0.18	0.19	0.19	0.20	0.20	0.20	0.20	0.20	0.20	0.20
31	0.25	0.25	0.25	0.26	0.26	0.27	0.27	0.28	0.28	0.28	0.28	0.28	0.28	0.29
32	0.32	0.33	0.33	0.34	0.34	0.35	0.35	0.36	0.36	0.36	0.36	0.36	0.36	0.37
33	0.40	0.41	0.41	0.42	0.42	0.43	0.43	0.44	0.44	0.44	0.44	0.44	0.44	0.45
34	0.48	0.49	0.49	0.50	0.50	0.51	0.52	0.52	0.52	0.53	0.53	0.53	0.53	0.53
35	0.56	0.57	0.57	0.58	0.59	0.60	0.61	0.61	0.61	0.62	0.62	0.62	0.62	0.62
36	0.64	0.65	0.65	0.67	0.68	0.69	0.70	0.70	0.70	0.71	0.70	0.70	0.70	0.70
37	0.72	0.74	0.74	0.76	0.77	0.78	0.79	0.79	0.79	0.80	0.79	0.79	0.79	0.79
38	0.81	0.83	0.83	0.85	0.86	0.87	0.88	0.88	0.88	0.89	0.88	0.88	0.88	0.88
39	0.90	0.92	0.92	0.94	0.95	0.96	0.97	0.97	0.97	0.98	0.97	0.97	0.97	0.97
40	0.99	1.01	1.01	1.03	1.04	1.05	1.06	1.06	1.06	1.07	1.06	1.05	1.05	1.05
45	1.48	1.50	1.50	1.52	1.53	1.53	1.53	1.53	1.52	1.52	1.51	1.50	1.50	1.48
50	2.03	2.04	2.04	2.05	2.06	2.05	2.04	2.03	2.01	2.00	1.99	1.97	1.96	1.92
55	2.62	2.63	2.63	2.64	2.62	2.60	2.58	2.55	2.52	2.50	2.48	2.45	2.43	2.37
60	3.26	3.28	3.27	3.25	3.22	3.18	3.13	3.09	3.04	3.02	2.98	2.94	2.90	2.83
65	3.95	3.98	3.95	3.90	3.86	3.79	3.71	3.66	3.58	3.56	3.50	3.44	3.38	3.30
70	4.70	4.74	4.70	4.60	4.54	4.44	4.32	4.24	4.14	4.12	4.03	3.96	3.87	3.78
75	5.50	5.56	4.48	5.34	5.26	5.12	4.95	4.86	4.72	4.70	4.57	4.50	4.37	4.27
80	6.35	6.41	6.32	6.12	6.02	5.84	5.62	5.51	5.32	5.30	5.14	5.04	4.88	4.77

TABLE 33 *

INTERNATIONAL SCALE (1936) OF REFRACTIVE INDICES OF SUCROSE SOLUTIONS

Per Cent Sucrose by Weight	n_D^{20}	n_D^{28}	Per Cent Sucrose by Weight	n_D^{20}	n_D^{28}	Per Cent Sucrose by Weight	n_D^{20}	n_D^{28}
0.0	1.33299	1.33219	4.0	1.33880	1.33795	8.0	1.34477	1.34387
0.1	1.33313	1.33233	4.1	1.33895	1.33810	8.1	1.34492	1.34402
0.2	1.33328	1.33248	4.2	1.33909	1.33824	8.2	1.34507	1.34417
0.3	1.33342	1.33262	4.3	1.33924	1.33839	8.3	1.34523	1.34432
0.4	1.33357	1.33276	4.4	1.33939	1.33853	8.4	1.34538	1.34447
0.5	1.33371	1.33291	4.5	1.33953	1.33868	8.5	1.34553	1.34463
0.6	1.33385	1.33305	4.6	1.33968	1.33883	8.6	1.34568	1.34478
0.7	1.33400	1.33319	4.7	1.33983	1.33897	8.7	1.34583	1.34493
0.8	1.33414	1.33333	4.8	1.33998	1.33912	8.8	1.34599	1.34508
0.9	1.33429	1.33348	4.9	1.34012	1.33926	8.9	1.34614	1.34523
1.0	1.33443	1.33362	5.0	1.34027	1.33941	9.0	1.34629	1.34538
1.1	1.33457	1.33376	5.1	1.34042	1.33956	9.1	1.34644	1.34553
1.2	1.33472	1.33391	5.2	1.34057	1.33971	9.2	1.34660	1.34569
1.3	1.33487	1.33405	5.3	1.34072	1.33985	9.3	1.34675	1.34584
1.4	1.33501	1.33420	5.4	1.34087	1.34000	9.4	1.34691	1.34599
1.5	1.33515	1.33434	5.5	1.34101	1.34015	9.5	1.34706	1.34615
1.6	1.33530	1.33448	5.6	1.34116	1.34030	9.6	1.34721	1.34630
1.7	1.33545	1.33463	5.7	1.34131	1.34045	9.7	1.34737	1.34645
1.8	1.33559	1.33477	5.8	1.34146	1.34059	9.8	1.34752	1.34660
1.9	1.33573	1.33492	5.9	1.34161	1.34074	9.9	1.34768	1.34676
2.0	1.33588	1.33506	6.0	1.34176	1.34089	10.0	1.34783	1.34691
2.1	1.33603	1.33520	6.1	1.34191	1.34104	10.1	1.34798	1.34706
2.2	1.33617	1.33535	6.2	1.34206	1.34119	10.2	1.34814	1.34722
2.3	1.33631	1.33549	6.3	1.34221	1.34134	10.3	1.34829	1.34737
2.4	1.33646	1.33563	6.4	1.34236	1.34149	10.4	1.34845	1.34752
2.5	1.33661	1.33577	6.5	1.34251	1.34163	10.5	1.34860	1.34767
2.6	1.33675	1.33592	6.6	1.34266	1.34178	10.6	1.34875	1.34783
2.7	1.33689	1.33606	6.7	1.34281	1.34193	10.7	1.34891	1.34798
2.8	1.33704	1.33620	6.8	1.34296	1.34208	10.8	1.34906	1.34813
2.9	1.33719	1.33635	6.9	1.34311	1.34223	10.9	1.34922	1.34829
3.0	1.33733	1.33649	7.0	1.34326	1.34238	11.0	1.34937	1.34844
3.1	1.33748	1.33664	7.1	1.34341	1.34253	11.1	1.34953	1.34859
3.2	1.33762	1.33678	7.2	1.34356	1.34268	11.2	1.34968	1.34875
3.3	1.33777	1.33693	7.3	1.34371	1.34283	11.3	1.34984	1.34891
3.4	1.33792	1.33707	7.4	1.34386	1.34298	11.4	1.34999	1.34906
3.5	1.33807	1.33722	7.5	1.34401	1.34313	11.5	1.35015	1.34921
3.6	1.33821	1.33737	7.6	1.34417	1.34327	11.6	1.35031	1.34937
3.7	1.33836	1.33751	7.7	1.34432	1.34342	11.7	1.35046	1.34953
3.8	1.33851	1.33766	7.8	1.34447	1.34357	11.8	1.35062	1.34968
3.9	1.33865	1.33780	7.9	1.34462	1.34372	11.9	1.35077	1.34983

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* See text, p. 445. Taken from *Proceedings* of the Ninth Session of the International Commission for Uniform Methods of Sugar Analysis, London, 1936. (*Intern. Sugar J.*, **39**, 23s). The refractive indices for tenths of per cent have been interpolated linearly by F. W. Zerban and checked against data of the National Bureau of Standards.

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TABLE 33 (*Continued*)

International Scale (1936) of Refractive Indices of Sucrose Solutions

Per Cent Sucrose by Weight	n_D^{20}	n_D^{28}	Per Cent Sucrose by Weight	n_D^{20}	n_D^{28}	Per Cent Sucrose by Weight	n_D^{20}	n_D^{28}
12.0	1.35093	1.34999	17.0	1.35890	1.35791	22.0	1.36719	1.36615
12.1	1.35109	1.35015	17.1	1.35906	1.35807	22.1	1.36736	1.36632
12.2	1.35124	1.35030	17.2	1.35923	1.35823	22.2	1.36753	1.36648
12.3	1.35140	1.35046	17.3	1.35939	1.35840	22.3	1.36770	1.36665
12.4	1.35156	1.35061	17.4	1.35955	1.35856	22.4	1.36787	1.36682
12.5	1.35171	1.35077	17.5	1.35971	1.35872	22.5	1.36803	1.36699
12.6	1.35187	1.35093	17.6	1.35988	1.35888	22.6	1.36820	1.36715
12.7	1.35203	1.35108	17.7	1.36004	1.35904	22.7	1.36837	1.36732
12.8	1.35219	1.35124	17.8	1.36020	1.35921	22.8	1.36854	1.36749
12.9	1.35234	1.35139	17.9	1.36037	1.35937	22.9	1.36871	1.36765
13.0	1.35250	1.35155	18.0	1.36053	1.35953	23.0	1.36888	1.36782
13.1	1.35266	1.35171	18.1	1.36069	1.35969	23.1	1.36905	1.36799
13.2	1.35282	1.35186	18.2	1.36086	1.35986	23.2	1.36922	1.36816
13.3	1.35297	1.35202	18.3	1.36103	1.36002	23.3	1.36939	1.36833
13.4	1.35313	1.35218	18.4	1.36119	1.36019	23.4	1.36956	1.36850
13.5	1.35329	1.35233	18.5	1.36135	1.36035	23.5	1.36973	1.36867
13.6	1.35345	1.35249	18.6	1.36152	1.36051	23.6	1.36991	1.36884
13.7	1.35361	1.35265	18.7	1.36169	1.36068	23.7	1.37008	1.36901
13.8	1.35376	1.35281	18.8	1.36185	1.36084	23.8	1.37025	1.36918
13.9	1.35392	1.35296	18.9	1.36201	1.36101	23.9	1.37042	1.36935
14.0	1.35408	1.35312	19.0	1.36218	1.36117	24.0	1.37059	1.36952
14.1	1.35424	1.35328	19.1	1.36235	1.36133	24.1	1.3708	1.3697
14.2	1.35440	1.35344	19.2	1.36251	1.36150	24.2	1.3709	1.3699
14.3	1.35456	1.35359	19.3	1.36268	1.36167	24.3	1.3711	1.3700
14.4	1.35472	1.35375	19.4	1.36284	1.36183	24.4	1.3713	1.3702
14.5	1.35487	1.35391	19.5	1.36301	1.36199	24.5	1.3715	1.3704
14.6	1.35503	1.35407	19.6	1.36318	1.36216	24.6	1.3716	1.3705
14.7	1.35519	1.35423	19.7	1.36334	1.36233	24.7	1.3718	1.3707
14.8	1.35535	1.35438	19.8	1.36351	1.36249	24.8	1.3720	1.3709
14.9	1.35551	1.35454	19.9	1.36367	1.36265	24.9	1.3721	1.3710
15.0	1.35567	1.35470	20.0	1.36384	1.36282	25.0	1.3723	1.3712
15.1	1.35583	1.35486	20.1	1.36401	1.36299	25.1	1.3725	1.3714
15.2	1.35599	1.35502	20.2	1.36417	1.36315	25.2	1.3726	1.3715
15.3	1.35615	1.35518	20.3	1.36434	1.36332	25.3	1.3728	1.3717
15.4	1.35631	1.35534	20.4	1.36451	1.36348	25.4	1.3730	1.3719
15.5	1.35647	1.35550	20.5	1.36467	1.36365	25.5	1.3731	1.3721
15.6	1.35664	1.35566	20.6	1.36484	1.36382	25.6	1.3733	1.3722
15.7	1.35680	1.35582	20.7	1.36501	1.36398	25.7	1.3735	1.3724
15.8	1.35696	1.35598	20.8	1.36518	1.36415	25.8	1.3737	1.3726
15.9	1.35712	1.35614	20.9	1.36534	1.36431	25.9	1.3738	1.3727
16.0	1.35728	1.35630	21.0	1.36551	1.36448	26.0	1.3740	1.3729
16.1	1.35744	1.35646	21.1	1.36568	1.36465	26.1	1.3742	1.3731
16.2	1.35760	1.35662	21.2	1.36585	1.36481	26.2	1.3744	1.3733
16.3	1.35777	1.35678	21.3	1.36601	1.36498	26.3	1.3745	1.3734
16.4	1.35793	1.35694	21.4	1.36618	1.36515	26.4	1.3747	1.3736
16.5	1.35809	1.35711	21.5	1.36635	1.36531	26.5	1.3749	1.3738
16.6	1.35825	1.35727	21.6	1.36652	1.36548	26.6	1.3751	1.3740
16.7	1.35841	1.35743	21.7	1.36669	1.36565	26.7	1.3753	1.3742
16.8	1.35858	1.35759	21.8	1.36685	1.36582	26.8	1.3754	1.3743
16.9	1.35874	1.35775	21.9	1.36702	1.36598	26.9	1.3756	1.3745

TABLE 33 (*Continued*)

INTERNATIONAL SCALE (1936) OF REFRACTIVE INDICES OF SUCROSE SOLUTIONS

Per Cent Sucrose by Weight	n_D^{20}	n_D^{28}	Per Cent Sucrose by Weight	n_D^{20}	n_D^{28}	Per Cent Sucrose by Weight	n_D^{20}	n_D^{28}
27.0	1.3758	1.3747	32.0	1.3847	1.3835	37.0	1.3939	1.3927
27.1	1.3760	1.3749	32.1	1.3849	1.3837	37.1	1.3941	1.3929
27.2	1.3761	1.3750	32.2	1.3851	1.3839	37.2	1.3943	1.3931
27.3	1.3763	1.3752	32.3	1.3852	1.3840	37.3	1.3945	1.3933
27.4	1.3765	1.3754	32.4	1.3854	1.3842	37.4	1.3947	1.3935
27.5	1.3767	1.3756	32.5	1.3856	1.3844	37.5	1.3949	1.3937
27.6	1.3768	1.3757	32.6	1.3858	1.3846	37.6	1.3950	1.3938
27.7	1.3770	1.3759	32.7	1.3860	1.3848	37.7	1.3952	1.3940
27.8	1.3772	1.3761	32.8	1.3861	1.3849	37.8	1.3954	1.3942
27.9	1.3773	1.3762	32.9	1.3863	1.3851	37.9	1.3956	1.3944
28.0	1.3775	1.3764	33.0	1.3865	1.3853	38.0	1.3958	1.3946
28.1	1.3777	1.3766	33.1	1.3867	1.3855	38.1	1.3960	1.3948
28.2	1.3779	1.3768	33.2	1.3869	1.3857	38.2	1.3962	1.3950
28.3	1.3780	1.3769	33.3	1.3870	1.3858	38.3	1.3964	1.3952
28.4	1.3782	1.3771	33.4	1.3872	1.3860	38.4	1.3966	1.3954
28.5	1.3784	1.3773	33.5	1.3874	1.3862	38.5	1.3968	1.3956
28.6	1.3786	1.3775	33.6	1.3876	1.3864	38.6	1.3970	1.3958
28.7	1.3788	1.3777	33.7	1.3878	1.3866	38.7	1.3972	1.3960
28.8	1.3789	1.3778	33.8	1.3879	1.3867	38.8	1.3974	1.3962
28.9	1.3791	1.3780	33.9	1.3881	1.3869	38.9	1.3976	1.3964
29.0	1.3793	1.3782	34.0	1.3883	1.3871	39.0	1.3978	1.3966
29.1	1.3795	1.3784	34.1	1.3885	1.3873	39.1	1.3980	1.3968
29.2	1.3797	1.3786	34.2	1.3887	1.3875	39.2	1.3982	1.3970
29.3	1.3798	1.3787	34.3	1.3889	1.3877	39.3	1.3984	1.3972
29.4	1.3800	1.3789	34.4	1.3891	1.3879	39.4	1.3986	1.3974
29.5	1.3802	1.3791	34.5	1.3893	1.3881	39.5	1.3987	1.3975
29.6	1.3804	1.3793	34.6	1.3894	1.3882	39.6	1.3989	1.3977
29.7	1.3806	1.3795	34.7	1.3896	1.3884	39.7	1.3991	1.3979
29.8	1.3807	1.3796	34.8	1.3898	1.3886	39.8	1.3993	1.3981
29.9	1.3809	1.3798	34.9	1.3900	1.3888	39.9	1.3995	1.3983
30.0	1.3811	1.3800	35.0	1.3902	1.3890	40.0	1.3997	1.3985
30.1	1.3813	1.3802	35.1	1.3904	1.3892	40.1	1.3999	1.3987
30.2	1.3815	1.3804	35.2	1.3906	1.3894	40.2	1.4001	1.3989
30.3	1.3816	1.3805	35.3	1.3907	1.3895	40.3	1.4003	1.3990
30.4	1.3818	1.3807	35.4	1.3909	1.3897	40.4	1.4005	1.3992
30.5	1.3820	1.3809	35.5	1.3911	1.3899	40.5	1.4007	1.3994
30.6	1.3822	1.3811	35.6	1.3913	1.3901	40.6	1.4008	1.3996
30.7	1.3824	1.3813	35.7	1.3915	1.3903	40.7	1.4010	1.3998
30.8	1.3825	1.3814	35.8	1.3916	1.3904	40.8	1.4012	1.3999
30.9	1.3827	1.3816	35.9	1.3918	1.3906	40.9	1.4014	1.4001
31.0	1.3829	1.3818	36.0	1.3920	1.3908	41.0	1.4016	1.4003
31.1	1.3831	1.3820	36.1	1.3922	1.3910	41.1	1.4018	1.4005
31.2	1.3833	1.3821	36.2	1.3924	1.3912	41.2	1.4020	1.4007
31.3	1.3834	1.3823	36.3	1.3926	1.3914	41.3	1.4022	1.4009
31.4	1.3836	1.3825	36.4	1.3928	1.3916	41.4	1.4024	1.4011
31.5	1.3838	1.3827	36.5	1.3929	1.3917	41.5	1.4026	1.4013
31.6	1.3840	1.3828	36.6	1.3931	1.3919	41.6	1.4028	1.4015
31.7	1.3842	1.3830	36.7	1.3933	1.3921	41.7	1.4030	1.4017
31.8	1.3843	1.3832	36.8	1.3935	1.3923	41.8	1.4032	1.4019
31.9	1.3845	1.3833	36.9	1.3937	1.3925	41.9	1.4034	1.4021

TABLE 33 (*Continued*)

INTERNATIONAL SCALE (1936) OF REFRACTIVE INDICES OF SUCROSE SOLUTIONS

Per Cent Sucrose by Weight	n_D^{20}	n_D^{28}	Per Cent Sucrose by Weight	n_D^{20}	n_D^{28}	Per Cent Sucrose by Weight	n_D^{20}	n_D^{28}
42.0	1.4036	1.4023	47.0	1.4137	1.4124	52.0	1.4242	1.4228
42.1	1.4038	1.4025	47.1	1.4139	1.4126	52.1	1.4244	1.4230
42.2	1.4040	1.4027	47.2	1.4141	1.4128	52.2	1.4246	1.4232
42.3	1.4042	1.4029	47.3	1.4143	1.4130	52.3	1.4249	1.4235
42.4	1.4044	1.4031	47.4	1.4145	1.4132	52.4	1.4251	1.4237
42.5	1.4046	1.4033	47.5	1.4147	1.4135	52.5	1.4253	1.4239
42.6	1.4048	1.4035	47.6	1.4150	1.4137	52.6	1.4255	1.4241
42.7	1.4050	1.4037	47.7	1.4152	1.4139	52.7	1.4257	1.4243
42.8	1.4052	1.4039	47.8	1.4154	1.4141	52.8	1.4260	1.4246
42.9	1.4054	1.4041	47.9	1.4156	1.4143	52.9	1.4262	1.4248
43.0	1.4056	1.4043	48.0	1.4158	1.4145	53.0	1.4264	1.4250
43.1	1.4058	1.4045	48.1	1.4160	1.4147	53.1	1.4266	1.4252
43.2	1.4060	1.4047	48.2	1.4162	1.4149	53.2	1.4268	1.4254
43.3	1.4062	1.4049	48.3	1.4164	1.4151	53.3	1.4270	1.4256
43.4	1.4064	1.4051	48.4	1.4166	1.4153	53.4	1.4272	1.4258
43.5	1.4066	1.4053	48.5	1.4169	1.4155	53.5	1.4275	1.4261
43.6	1.4068	1.4055	48.6	1.4171	1.4158	53.6	1.4277	1.4263
43.7	1.4070	1.4057	48.7	1.4173	1.4160	53.7	1.4279	1.4265
43.8	1.4072	1.4059	48.8	1.4175	1.4162	53.8	1.4281	1.4267
43.9	1.4074	1.4061	48.9	1.4177	1.4164	53.9	1.4283	1.4269
44.0	1.4076	1.4063	49.0	1.4179	1.4166	54.0	1.4285	1.4271
44.1	1.4078	1.4065	49.1	1.4181	1.4168	54.1	1.4287	1.4273
44.2	1.4080	1.4067	49.2	1.4183	1.4170	54.2	1.4289	1.4275
44.3	1.4082	1.4069	49.3	1.4185	1.4172	54.3	1.4292	1.4278
44.4	1.4084	1.4071	49.4	1.4187	1.4174	54.4	1.4294	1.4280
44.5	1.4086	1.4073	49.5	1.4189	1.4177	54.5	1.4296	1.4282
44.6	1.4088	1.4075	49.6	1.4192	1.4179	54.6	1.4298	1.4284
44.7	1.4090	1.4077	49.7	1.4194	1.4181	54.7	1.4300	1.4286
44.8	1.4092	1.4079	49.8	1.4196	1.4183	54.8	1.4303	1.4289
44.9	1.4094	1.4081	49.9	1.4198	1.4185	54.9	1.4305	1.4291
45.0	1.4096	1.4083	50.0	1.4200	1.4187	55.0	1.4307	1.4293
45.1	1.4098	1.4085	50.1	1.4202	1.4189	55.1	1.4309	1.4295
45.2	1.4100	1.4087	50.2	1.4204	1.4191	55.2	1.4311	1.4297
45.3	1.4102	1.4089	50.3	1.4206	1.4193	55.3	1.4313	1.4300
45.4	1.4104	1.4091	50.4	1.4208	1.4195	55.4	1.4316	1.4302
45.5	1.4107	1.4093	50.5	1.4211	1.4197	55.5	1.4318	1.4304
45.6	1.4109	1.4096	50.6	1.4213	1.4199	55.6	1.4320	1.4306
45.7	1.4111	1.4098	50.7	1.4215	1.4201	55.7	1.4322	1.4308
45.8	1.4113	1.4100	50.8	1.4217	1.4203	55.8	1.4325	1.4311
45.9	1.4115	1.4102	50.9	1.4219	1.4205	55.9	1.4327	1.4313
46.0	1.4117	1.4104	51.0	1.4221	1.4207	56.0	1.4329	1.4315
46.1	1.4119	1.4106	51.1	1.4223	1.4209	56.1	1.4331	1.4317
46.2	1.4121	1.4108	51.2	1.4225	1.4211	56.2	1.4333	1.4319
46.3	1.4123	1.4110	51.3	1.4227	1.4213	56.3	1.4336	1.4322
46.4	1.4125	1.4112	51.4	1.4229	1.4215	56.4	1.4338	1.4324
46.5	1.4127	1.4114	51.5	1.4231	1.4217	56.5	1.4340	1.4326
46.6	1.4129	1.4116	51.6	1.4234	1.4220	56.6	1.4342	1.4328
46.7	1.4131	1.4118	51.7	1.4236	1.4222	56.7	1.4344	1.4330
46.8	1.4133	1.4120	51.8	1.4238	1.4224	56.8	1.4347	1.4333
46.9	1.4135	1.4122	51.9	1.4240	1.4226	56.9	1.4349	1.4335

TABLE 33 (*Continued*)

INTERNATIONAL SCALE (1936) OF REFRACTIVE INDICES OF SUCROSE SOLUTIONS

Per Cent Sucrose by Weight	n_D^{20}	n_D^{28}	Per Cent Sucrose by Weight	n_D^{20}	n_D^{28}	Per Cent Sucrose by Weight	n_D^{20}	n_D^{28}
57.0	1.4351	1.4337	62.0	1.4464	1.4449	67.0	1.4579	1.4564
57.1	1.4353	1.4339	62.1	1.4466	1.4451	67.1	1.4581	1.4566
57.2	1.4355	1.4341	62.2	1.4468	1.4453	67.2	1.4584	1.4569
57.3	1.4358	1.4344	62.3	1.4471	1.4456	67.3	1.4586	1.4571
57.4	1.4360	1.4346	62.4	1.4473	1.4458	67.4	1.4589	1.4574
57.5	1.4362	1.4348	62.5	1.4475	1.4460	67.5	1.4591	1.4576
57.6	1.4364	1.4350	62.6	1.4477	1.4462	67.6	1.4593	1.4578
57.7	1.4366	1.4352	62.7	1.4479	1.4464	67.7	1.4596	1.4581
57.8	1.4369	1.4355	62.8	1.4482	1.4467	67.8	1.4598	1.4583
57.9	1.4371	1.4357	62.9	1.4484	1.4469	67.9	1.4601	1.4586
58.0	1.4373	1.4359	63.0	1.4486	1.4471	68.0	1.4603	1.4588
58.1	1.4375	1.4361	63.1	1.4488	1.4473	68.1	1.4605	1.4590
58.2	1.4378	1.4364	63.2	1.4491	1.4476	68.2	1.4608	1.4593
58.3	1.4380	1.4366	63.3	1.4493	1.4478	68.3	1.4610	1.4595
58.4	1.4382	1.4368	63.4	1.4495	1.4480	68.4	1.4613	1.4598
58.5	1.4385	1.4371	63.5	1.4497	1.4483	68.5	1.4615	1.4600
58.6	1.4387	1.4373	63.6	1.4500	1.4485	68.6	1.4617	1.4602
58.7	1.4389	1.4375	63.7	1.4502	1.4487	68.7	1.4620	1.4605
58.8	1.4391	1.4377	63.8	1.4504	1.4489	68.8	1.4622	1.4607
58.9	1.4394	1.4380	63.9	1.4507	1.4492	68.9	1.4625	1.4610
59.0	1.4396	1.4382	64.0	1.4509	1.4494	69.0	1.4627	1.4612
59.1	1.4398	1.4384	64.1	1.4511	1.4496	69.1	1.4629	1.4614
59.2	1.4400	1.4386	64.2	1.4514	1.4499	69.2	1.4632	1.4617
59.3	1.4403	1.4388	64.3	1.4516	1.4501	69.3	1.4634	1.4619
59.4	1.4405	1.4390	64.4	1.4518	1.4503	69.4	1.4637	1.4621
59.5	1.4407	1.4393	64.5	1.4521	1.4505	69.5	1.4639	1.4623
59.6	1.4409	1.4395	64.6	1.4523	1.4508	69.6	1.4641	1.4626
59.7	1.4411	1.4397	64.7	1.4525	1.4510	69.7	1.4644	1.4628
59.8	1.4414	1.4399	64.8	1.4527	1.4512	69.8	1.4646	1.4630
59.9	1.4416	1.4401	64.9	1.4530	1.4515	69.9	1.4649	1.4633
60.0	1.4418	1.4403	65.0	1.4532	1.4517	70.0	1.4651	1.4635
60.1	1.4420	1.4405	65.1	1.4534	1.4519	70.1	1.4653	1.4637
60.2	1.4423	1.4408	65.2	1.4537	1.4522	70.2	1.4656	1.4640
60.3	1.4425	1.4410	65.3	1.4539	1.4524	70.3	1.4658	1.4643
60.4	1.4427	1.4412	65.4	1.4541	1.4526	70.4	1.4661	1.4645
60.5	1.4429	1.4415	65.5	1.4544	1.4529	70.5	1.4663	1.4647
60.6	1.4432	1.4417	65.6	1.4546	1.4531	70.6	1.4666	1.4650
60.7	1.4434	1.4419	65.7	1.4548	1.4533	70.7	1.4668	1.4653
60.8	1.4436	1.4421	65.8	1.4550	1.4535	70.8	1.4671	1.4655
60.9	1.4439	1.4424	65.9	1.4553	1.4538	70.9	1.4673	1.4657
61.0	1.4441	1.4426	66.0	1.4555	1.4540	71.0	1.4676	1.4660
61.1	1.4443	1.4428	66.1	1.4557	1.4542	71.1	1.4678	1.4662
61.2	1.4446	1.4431	66.2	1.4560	1.4545	71.2	1.4681	1.4665
61.3	1.4448	1.4433	66.3	1.4562	1.4547	71.3	1.4683	1.4667
61.4	1.4450	1.4435	66.4	1.4565	1.4550	71.4	1.4685	1.4670
61.5	1.4453	1.4437	66.5	1.4567	1.4552	71.5	1.4688	1.4672
61.6	1.4455	1.4440	66.6	1.4569	1.4554	71.6	1.4690	1.4674
61.7	1.4457	1.4442	66.7	1.4572	1.4557	71.7	1.4693	1.4677
61.8	1.4459	1.4444	66.8	1.4574	1.4559	71.8	1.4695	1.4679
61.9	1.4462	1.4447	66.9	1.4577	1.4562	71.9	1.4698	1.4682

TABLE 33 (*Continued*)

INTERNATIONAL SCALE (1936) OF REFRACTIVE INDICES OF SUCROSE SOLUTIONS

Per Cent Sucrose by Weight	n_D^{20}	n_D^{28}	Per Cent Sucrose by Weight	n_D^{20}	n_D^{28}	Per Cent Sucrose by Weight	n_D^{20}	n_D^{28}
72.0	1.4700	1.4684	77.0	1.4825	1.4809	82.0	1.4954	1.4937
72.1	1.4703	1.4687	77.1	1.4827	1.4811	82.1	1.4956	1.4940
72.2	1.4705	1.4689	77.2	1.4830	1.4814	82.2	1.4959	1.4942
72.3	1.4708	1.4691	77.3	1.4832	1.4817	82.3	1.4962	1.4945
72.4	1.4710	1.4694	77.4	1.4835	1.4819	82.4	1.4964	1.4947
72.5	1.4713	1.4697	77.5	1.4838	1.4821	82.5	1.4967	1.4950
72.6	1.4715	1.4699	77.6	1.4840	1.4824	82.6	1.4970	1.4953
72.7	1.4717	1.4701	77.7	1.4843	1.4827	82.7	1.4972	1.4955
72.8	1.4720	1.4704	77.8	1.4845	1.4829	82.8	1.4975	1.4958
72.9	1.4722	1.4707	77.9	1.4848	1.4831	82.9	1.4978	1.4960
73.0	1.4725	1.4709	78.0	1.4850	1.4834	83.0	1.4980	1.4963
73.1	1.4727	1.4711	78.1	1.4853	1.4837	83.1	1.4983	1.4966
73.2	1.4730	1.4714	78.2	1.4855	1.4839	83.2	1.4985	1.4968
73.3	1.4732	1.4716	78.3	1.4858	1.4842	83.3	1.4988	1.4971
73.4	1.4735	1.4719	78.4	1.4860	1.4844	83.4	1.4991	1.4974
73.5	1.4737	1.4721	78.5	1.4863	1.4847	83.5	1.4993	1.4977
73.6	1.4740	1.4723	78.6	1.4865	1.4850	83.6	1.4996	1.4979
73.7	1.4742	1.4726	78.7	1.4868	1.4852	83.7	1.4999	1.4982
73.8	1.4744	1.4728	78.8	1.4871	1.4855	83.8	1.5001	1.4985
73.9	1.4747	1.4731	78.9	1.4873	1.4857	83.9	1.5004	1.4987
74.0	1.4749	1.4733	79.0	1.4876	1.4860	84.0	1.5007	1.4990
74.1	1.4752	1.4735	79.1	1.4878	1.4862	84.1	1.5009	1.4993
74.2	1.4754	1.4738	79.2	1.4881	1.4865	84.2	1.5012	1.4995
74.3	1.4757	1.4741	79.3	1.4883	1.4867	84.3	1.5015	1.4998
74.4	1.4759	1.4743	79.4	1.4886	1.4870	84.4	1.5017	1.5000
74.5	1.4762	1.4745	79.5	1.4888	1.4872	84.5	1.5020	1.5003
74.6	1.4764	1.4748	79.6	1.4891	1.4874	84.6	1.5022	1.5006
74.7	1.4767	1.4751	79.7	1.4893	1.4877	84.7	1.5025	1.5008
74.8	1.4769	1.4753	79.8	1.4896	1.4879	84.8	1.5028	1.5011
74.9	1.4772	1.4755	79.9	1.4898	1.4882	84.9	1.5030	1.5013
75.0	1.4774	1.4758	80.0	1.4901	1.4884	85.0	1.5033	1.5016
75.1	1.4777	1.4761	80.1	1.4904	1.4887			
75.2	1.4779	1.4763	80.2	1.4906	1.4889			
75.3	1.4782	1.4765	80.3	1.4909	1.4892			
75.4	1.4784	1.4768	80.4	1.4912	1.4894			
75.5	1.4787	1.4771	80.5	1.4914	1.4897			
75.6	1.4789	1.4773	80.6	1.4917	1.4900			
75.7	1.4792	1.4775	80.7	1.4919	1.4902			
75.8	1.4794	1.4778	80.8	1.4922	1.4905			
75.9	1.4797	1.4781	80.9	1.4925	1.4907			
76.0	1.4799	1.4783	81.0	1.4927	1.4910			
76.1	1.4802	1.4786	81.1	1.4930	1.4913			
76.2	1.4804	1.4788	81.2	1.4933	1.4915			
76.3	1.4807	1.4791	81.3	1.4935	1.4918			
76.4	1.4810	1.4793	81.4	1.4938	1.4921			
76.5	1.4812	1.4796	81.5	1.4941	1.4923			
76.6	1.4815	1.4799	81.6	1.4943	1.4926			
76.7	1.4817	1.4801	81.7	1.4946	1.4929			
76.8	1.4820	1.4804	81.8	1.4949	1.4932			
76.9	1.4822	1.4806	81.9	1.4951	1.4934			

TABLE 34

International Temperature Correction Table (1936) for the Normal Model of Refractometer, Above and Below 20° C.*

Temp. °C.	Per Cent Sucrose 0	5	10	15	20	25	30	35	40	45	50	55	60	65	70
	Subtract from the Per Cent Sucrose														
10	0.50	0.54	0.58	0.61	0.64	0.66	0.68	0.70	0.72	0.73	0.74	0.75	0.76	0.78	0.79
11	0.46	0.49	0.53	0.55	0.58	0.60	0.62	0.64	0.65	0.66	0.67	0.68	0.69	0.70	0.71
12	0.42	0.45	0.48	0.50	0.52	0.54	0.56	0.57	0.58	0.59	0.60	0.61	0.61	0.63	0.63
13	0.37	0.40	0.42	0.44	0.46	0.48	0.49	0.50	0.51	0.52	0.53	0.54	0.54	0.55	0.55
14	0.33	0.35	0.37	0.39	0.40	0.41	0.42	0.43	0.44	0.45	0.45	0.46	0.46	0.47	0.48
15	0.27	0.29	0.31	0.33	0.34	0.34	0.35	0.36	0.37	0.37	0.38	0.39	0.39	0.40	0.40
16	0.22	0.24	0.25	0.26	0.27	0.28	0.28	0.29	0.30	0.30	0.30	0.31	0.31	0.32	0.32
17	0.17	0.18	0.19	0.20	0.21	0.21	0.21	0.22	0.22	0.23	0.23	0.23	0.23	0.24	0.24
18	0.12	0.13	0.13	0.14	0.14	0.14	0.14	0.15	0.15	0.15	0.15	0.16	0.16	0.16	0.16
19	0.06	0.06	0.06	0.07	0.07	0.07	0.07	0.08	0.08	0.08	0.08	0.08	0.08	0.08	0.08
	Add to the Per Cent Sucrose														
21	0.06	0.07	0.07	0.07	0.07	0.08	0.08	0.08	0.08	0.08	0.08	0.08	0.08	0.08	0.08
22	0.13	0.13	0.14	0.14	0.15	0.15	0.15	0.15	0.15	0.16	0.16	0.16	0.16	0.16	0.16
23	0.19	0.20	0.21	0.22	0.22	0.23	0.23	0.23	0.23	0.24	0.24	0.24	0.24	0.24	0.24
24	0.26	0.27	0.28	0.29	0.30	0.30	0.31	0.31	0.31	0.31	0.31	0.32	0.32	0.32	0.32
25	0.33	0.35	0.36	0.37	0.38	0.38	0.39	0.40	0.40	0.40	0.40	0.40	0.40	0.40	0.40
26	0.40	0.42	0.43	0.44	0.45	0.46	0.47	0.48	0.48	0.48	0.48	0.48	0.48	0.48	0.48
27	0.48	0.50	0.52	0.53	0.54	0.55	0.55	0.56	0.56	0.56	0.56	0.56	0.56	0.56	0.56
28	0.56	0.57	0.60	0.61	0.62	0.63	0.63	0.64	0.64	0.64	0.64	0.64	0.64	0.64	0.64
29	0.64	0.66	0.68	0.69	0.71	0.72	0.72	0.73	0.73	0.73	0.73	0.73	0.73	0.73	0.73
30	0.72	0.74	0.77	0.78	0.79	0.80	0.80	0.81	0.81	0.81	0.81	0.81	0.81	0.81	0.81

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* See text, p. 442. Taken from *Proceedings* of the Ninth Session of the International Commission for Uniform Methods of Sugar Analysis, London, 1936 (*Intern. Sugar J.*, **39**, 24a).

TABLE 35

INTERNATIONAL TEMPERATURE CORRECTION TABLE (1936) FOR THE TROPICAL MODEL OF REFRACTOMETER, ABOVE AND BELOW 28° C.*

Temp. °C.	Per Cent Sucrose														
	0	5	10	15	20	25	30	35	40	45	50	55	60	65	70
	Subtract from the Per Cent Sucrose														
20	0.57	0.59	0.60	0.61	0.62	0.63	0.63	0.64	0.64	0.64	0.64	0.64	0.64	0.65	0.65
21	0.51	0.52	0.53	0.53	0.55	0.55	0.55	0.56	0.56	0.56	0.56	0.56	0.56	0.57	0.57
22	0.44	0.46	0.46	0.46	0.47	0.48	0.48	0.49	0.48	0.48	0.48	0.48	0.48	0.49	0.49
23	0.37	0.38	0.39	0.39	0.40	0.40	0.40	0.41	0.41	0.41	0.40	0.40	0.40	0.41	0.41
24	0.30	0.31	0.32	0.32	0.32	0.32	0.32	0.33	0.33	0.33	0.32	0.32	0.32	0.33	0.33
25	0.23	0.23	0.24	0.24	0.24	0.24	0.24	0.25	0.25	0.25	0.24	0.24	0.24	0.24	0.24
26	0.16	0.16	0.16	0.16	0.16	0.16	0.16	0.17	0.16	0.16	0.16	0.16	0.16	0.16	0.16
27	0.08	0.08	0.08	0.08	0.08	0.08	0.08	0.08	0.08	0.08	0.08	0.08	0.08	0.08	0.08
	Add to the Per Cent Sucrose														
29	0.09	0.09	0.09	0.09	0.09	0.09	0.09	0.08	0.08	0.08	0.08	0.08	0.08	0.08	0.08
30	0.17	0.17	0.17	0.17	0.17	0.17	0.17	0.17	0.17	0.17	0.17	0.17	0.17	0.17	0.17
31	0.26	0.26	0.26	0.26	0.26	0.26	0.26	0.25	0.25	0.25	0.25	0.25	0.25	0.25	0.25
32	0.35	0.35	0.35	0.35	0.35	0.34	0.34	0.34	0.34	0.34	0.34	0.33	0.33	0.33	0.33
33	0.44	0.44	0.44	0.44	0.44	0.43	0.43	0.43	0.43	0.42	0.42	0.42	0.42	0.42	0.42
34	0.54	0.54	0.53	0.53	0.53	0.52	0.52	0.52	0.52	0.51	0.51	0.50	0.50	0.50	0.50
35	0.63	0.63	0.63	0.62	0.62	0.62	0.61	0.61	0.61	0.60	0.60	0.59	0.59	0.59	0.5
36	0.73	0.73	0.73	0.72	0.72	0.72	0.71	0.70	0.70	0.69	0.69	0.68	0.67	0.67	0.6

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* See text, p. 443. Taken from *Proceedings* of the Ninth Session of the International Commissio for Uniform Methods of Sugar Analysis, London, 1936 (*Intern. Sugar J.*, **39**, 24s).

TABLE 36

SCHMITZ'S TABLE FOR SUCROSE (POL) FOR USE IN HORNE'S DRY LEAD METHOD WITH UNDILUTED SOLUTIONS *

Pol. Reading	Degrees Brix and Per Cents Sucrose																		Pol. Reading
	1.5	2.0	2.5	3.0	3.5	4.0	4.5	5.0	5.5	6.0	6.5	7.0	7.5	8.0	8.5	9.0	9.5	10.0	
1	0.26	0.26	0.26	0.26	0.26	0.26	0.26	0.26	0.26	0.25	0.25	0.25	0.25	0.25	0.25	0.25	0.25	0.25	1
2	0.52	0.52	0.52	0.52	0.52	0.51	0.51	0.51	0.51	0.51	0.51	0.51	0.51	0.51	0.50	0.50	0.50	0.50	2
3	0.78	0.78	0.77	0.77	0.77	0.77	0.77	0.77	0.77	0.76	0.76	0.76	0.76	0.76	0.76	0.75	0.75	0.75	3
4	1.04	1.03	1.03	1.03	1.03	1.03	1.02	1.02	1.02	1.02	1.02	1.01	1.01	1.01	1.01	1.01	1.00	1.00	4
5	1.29	1.29	1.29	1.29	1.29	1.28	1.28	1.28	1.28	1.27	1.27	1.27	1.27	1.26	1.26	1.26	1.25	1.25	5
6	.....	1.55	1.55	1.55	1.55	1.54	1.54	1.53	1.53	1.53	1.53	1.52	1.52	1.51	1.51	1.51	1.51	1.50	6
9	.....	1.81	1.81	1.80	1.80	1.80	1.80	1.79	1.79	1.79	1.78	1.77	1.77	1.77	1.76	1.76	1.76	1.75	7
8	.....	.....	2.06	2.06	2.06	2.05	2.05	2.04	2.04	2.04	2.03	2.03	2.02	2.02	2.02	2.01	2.01	2.00	8
9	.....	.....	.....	2.32	2.31	2.31	2.30	2.30	2.30	2.29	2.28	2.28	2.28	2.27	2.27	2.26	2.26	2.25	9
10	.....	.....	.....	**2.58**	**2.57**	**2.57**	**2.56**	**2.56**	**2.55**	**2.55**	**2.54**	**2.54**	**2.53**	**2.53**	**2.52**	**2.51**	**2.51**	**2.50**	**10**
11	.....	.....	.....	2.83	2.83	2.82	2.82	2.81	2.81	2.80	2.80	2.79	2.78	2.78	2.77	2.77	2.76	2.75	11
12	.....	.....	.....	.....	3.08	3.08	3.07	3.06	3.06	3.05	3.05	3.04	3.04	3.03	3.02	3.02	3.01	3.00	12
13	.....	.....	.....	.....	3.34	3.33	3.33	3.32	3.31	3.30	3.30	3.29	3.29	3.28	3.28	3.27	3.26	3.26	13
14	.....	.....	.....	.....	.....	3.59	3.58	3.58	3.57	3.56	3.56	3.55	3.54	3.53	3.52	3.52	3.51	3.51	14
15	.....	.....	.....	.....	.....	3.84	3.83	3.83	3.82	3.82	3.82	3.81	3.79	3.78	3.78	3.77	3.76	3.76	15
16	.....	.....	.....	.....	.....	.....	4.09	4.09	4.08	4.07	4.07	4.06	4.05	4.04	4.03	4.02	4.01	4.01	16
17	.....	.....	.....	.....	.....	.....	.....	4.34	4.33	4.33	4.32	4.31	4.30	4.29	4.28	4.28	4.27	4.26	17
18	.....	.....	.....	.....	.....	.....	.....	4.59	4.59	4.58	4.57	4.56	4.55	4.54	4.54	4.53	4.52	4.51	18
19	.....	.....	.....	.....	.....	.....	.....	.....	4.84	4.84	4.83	4.82	4.81	4.80	4.79	4.78	4.77	4.76	19
20	.....	.....	.....	.....	.....	.....	.....	.....	**5.10**	**5.09**	**5.08**	**5.07**	**5.06**	**5.05**	**5.04**	**5.03**	**5.02**	**5.01**	**20**
21	.....	.....	.....	.....	.....	.....	.....	.....	5.36	5.34	5.33	5.32	5.30	5.30	5.29	5.28	5.27	5.26	21
22	.....	.....	.....	.....	.....	.....	.....	.....	.....	5.60	5.59	5.58	5.57	5.55	5.54	5.53	5.52	5.51	22

* This table has been recalculated (1943) *for a normal weight of* 26.000 (see *p.* 372) and temperature of 20° C. by the formula

$$\text{Pol of juice} = \frac{\text{Pol reading} \times 26.000}{99.718 \times \text{specific gravity } 20°/20° \text{ C.}}$$

The recalculation made no significant change in the first half of the table as previously published for the old normal weight of 26.048 and 17½° C. The second half of the present table differs from the one previously published by 0.01 to 0.02 per cent sucrose (pol).

TABLE 36 (*Continued*)

SCHMITZ'S TABLE FOR SUCROSE (POL) FOR USE IN HORNE'S DRY LEAD METHOD WITH UNDILUTED SOLUTIONS

Degrees Brix from 0.5 to 12.0	
Tenths of the Polariscope Reading	Per Cent Sucrose
0.1	0.025
0.2	0.051
0.3	0.076
0.4	0.102
0.5	0.127
0.6	0.152
0.7	0.178
0.8	0.203
0.9	0.228

INSTRUCTIONS

Note the Brix of the solution. To an indefinite volume of the solution, contained in a narrow cylinder, add sufficient dry lead subacetate and a little dry sand; cover the mouth of the cylinder with the hand and shake thoroughly to mix the solution with the lead. Filter and polarize, using a 200-mm. tube. To the number in the table opposite the integral part of the polariscope reading and under the degree Brix nearest that observed, add the number in the small table corresponding to the tenths of the reading. The sum of these numbers is the polarization of the solution.

6.0	6.5	7.0	7.5	8.0	8.5	9.0	9.5	10.0	
5.85	5.84	5.83	5.82	5.81	5.80	5.79	5.77	5.76	23
.....	6.10	6.09	6.07	6.06	6.05	6.04	6.02	6.01	24
.....	6.35	6.34	6.33	6.31	6.30	6.29	6.27	6.26	25
.....	.....	6.59	6.58	6.57	6.55	6.54	6.52	6.51	26
.....	.....	6.84	6.83	6.82	6.80	6.79	6.77	6.76	27
.....	.....	.....	7.08	7.07	7.05	7.04	7.03	7.02	28
.....	.....	.....	7.34	7.33	7.31	7.29	7.28	7.27	29
.....	.....	.....	.....	**7.57**	**7.56**	**7.54**	**7.53**	**7.52**	**30**
.....	.....	.....	.....	7.82	7.81	7.79	7.78	7.76	31
.....	.....	.....	.....	.....	8.06	8.04	8.03	8.02	32
.....	.....	.....	.....	.....	8.31	8.30	8.28	8.27	33
.....	.....	.....	.....	.....	.....	8.55	8.53	8.52	34
.....	.....	.....	.....	.....	.....	.....	8.78	8.77	35
.....	.....	.....	.....	.....	.....	.....	9.03	9.02	36
.....	.....	.....	.....	.....	.....	.....	9.28	9.27	37
.....	.....	.....	.....	.....	.....	.....	.....	9.51	38
.....	.....	.....	.....	.....	.....	.....	.....	9.76	39

TABLE 36 (*Continued*)

SCHMITZ'S TABLE FOR SUCROSE (POL) FOR USE IN HORNE'S DRY LEAD METHOD WITH UNDILUTED SOLUTIONS

Pol. Reading	Degrees Brix and Per Cents Sucrose																				Pol. Reading
	10.5	11.0	11.5	12.0	12.5	13.0	13.5	14.0	14.5	15.0	15.5	16.0	16.5	17.0	17.5	18.0	18.5	19.0	19.5	20.0	
1	0.25	0.25	0.25	0.25	0.25	0.25	0.25	0.25	0.25	0.25	0.25	0.24	0.24	0.24	0.24	0.24	0.24	0.24	0.24	0.24	1
2	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.48	0.48	0.48	0.48	0.48	0.48	0.48	0.48	0.48	2
3	0.75	0.75	0.75	0.75	0.74	0.74	0.74	0.74	0.74	0.74	0.74	0.73	0.73	0.73	0.73	0.73	0.73	0.73	0.72	0.72	3
4	1.00	1.00	1.00	0.99	0.99	0.99	0.99	0.99	0.99	0.98	0.98	0.98	0.98	0.97	0.97	0.97	0.97	0.97	0.96	0.96	4
5	1.25	1.25	1.25	1.24	1.24	1.24	1.24	1.24	1.23	1.23	1.23	1.22	1.22	1.22	1.22	1.21	1.21	1.21	1.21	1.20	5
6	1.50	1.50	1.50	1.49	1.49	1.49	1.48	1.48	1.48	1.48	1.47	1.47	1.46	1.46	1.46	1.45	1.45	1.45	1.45	1.44	6
7	1.75	1.75	1.75	1.74	1.74	1.74	1.73	1.73	1.72	1.72	1.71	1.71	1.70	1.70	1.69	1.69	1.69	1.69	1.69	1.68	7
8	2.00	2.00	1.99	1.98	1.98	1.98	1.98	1.97	1.97	1.97	1.96	1.96	1.95	1.95	1.94	1.94	1.94	1.93	1.93	1.92	8
9	2.25	2.24	2.24	2.24	2.24	2.24	2.23	2.22	2.22	2.21	2.21	2.21	2.20	2.20	2.19	2.19	2.18	2.18	2.17	2.16	9
10	**2.50**	**2.49**	**2.49**	**2.48**	**2.48**	**2.48**	**2.47**	**2.47**	**2.46**	**2.46**	**2.45**	**2.45**	**2.44**	**2.44**	**2.43**	**2.43**	**2.42**	**2.42**	**2.41**	**2.41**	10
11	2.75	2.74	2.73	2.73	2.72	2.72	2.71	2.71	2.70	2.70	2.69	2.68	2.68	2.67	2.67	2.67	2.66	2.66	2.65	2.64	11
12	3.00	2.99	2.98	2.98	2.97	2.97	2.96	2.96	2.95	2.95	2.94	2.93	2.93	2.92	2.92	2.91	2.91	2.90	2.89	2.89	12
13	3.25	3.24	3.23	3.23	3.22	3.21	3.20	3.20	3.19	3.19	3.18	3.17	3.17	3.16	3.16	3.15	3.14	3.14	3.13	3.13	13
14	3.50	3.50	3.49	3.48	3.47	3.46	3.45	3.45	3.44	3.43	3.42	3.42	3.41	3.41	3.41	3.40	3.39	3.38	3.37	3.37	14
15	3.75	3.74	3.73	3.72	3.71	3.71	3.70	3.69	3.69	3.68	3.67	3.66	3.66	3.65	3.65	3.64	3.63	3.62	3.62	3.61	15
16	4.00	3.99	3.98	3.97	3.96	3.96	3.95	3.94	3.94	3.93	3.92	3.91	3.90	3.88	3.88	3.88	3.87	3.86	3.85	3.85	16
17	4.25	4.25	4.23	4.22	4.21	4.20	4.19	4.19	4.18	4.17	4.16	4.15	4.14	4.12	4.12	4.12	4.11	4.11	4.10	4.09	17
18	4.50	4.49	4.48	4.47	4.46	4.45	4.44	4.44	4.43	4.42	4.41	4.40	4.39	4.37	4.37	4.37	4.35	4.35	4.34	4.33	18
19	4.75	4.74	4.73	4.72	4.71	4.70	4.69	4.68	4.67	4.66	4.65	4.64	4.63	4.61	4.61	4.61	4.60	4.59	4.58	4.57	19
20	**5.00**	**4.99**	**4.98**	**4.97**	**4.96**	**4.95**	**4.94**	**4.93**	**4.92**	**4.91**	**4.90**	**4.89**	**4.88**	**4.86**	**4.86**	**4.85**	**4.84**	**4.83**	**4.82**	**4.81**	20
21	5.25	5.23	5.22	5.21	5.20	5.19	5.18	5.17	5.16	5.15	5.14	5.13	5.12	5.10	5.10	5.09	5.08	5.07	5.06	5.05	21
22	5.50	5.48	5.47	5.46	5.45	5.44	5.43	5.42	5.41	5.40	5.39	5.38	5.37	5.35	5.35	5.34	5.32	5.31	5.30	5.29	22
23	5.75	5.73	5.72	5.71	5.70	5.69	5.68	5.67	5.66	5.65	5.64	5.62	5.61	5.59	5.58	5.58	5.56	5.55	5.54	5.53	23
24	6.00	5.98	5.97	5.96	5.95	5.94	5.93	5.92	5.91	5.90	5.89	5.87	5.86	5.84	5.83	5.82	5.81	5.80	5.79	5.77	24
25	6.25	6.23	6.22	6.21	6.20	6.18	6.17	6.16	6.15	6.14	6.13	6.11	6.10	6.08	6.07	6.06	6.05	6.04	6.03	6.01	25

TABLE 36 (*Continued*)

SCHMITZ'S TABLE FOR SUCROSE (POL) FOR USE IN HORNE'S DRY LEAD METHOD WITH UNDILUTED SOLUTIONS

Pol. Reading	Degrees Brix and Per Cents Sucrose																				Pol. Reading
	10.5	11.0	11.5	12.0	12.5	13.0	13.5	14.0	14.5	15.0	15.5	16.0	16.5	17.0	17.5	18.0	18.5	19.0	19.5	20.0	
26	6.50	6.49	6.48	6.46	6.44	6.43	6.42	6.41	6.40	6.39	6.38	6.36	6.34	6.33	6.32	6.30	6.29	6.28	6.27	6.26	**26**
27	6.75	6.74	6.72	6.70	6.69	6.68	6.67	6.65	6.64	6.63	6.62	6.60	6.59	6.57	6.56	6.55	6.54	6.52	6.51	6.50	27
28	7.00	6.98	6.97	6.95	6.94	6.93	6.93	6.90	6.89	6.87	6.86	6.85	6.84	6.82	6.81	6.79	6.78	6.77	6.75	6.74	28
29	7.25	7.23	7.22	7.20	7.19	7.17	7.16	7.15	7.14	7.12	7.11	7.09	7.08	7.06	7.05	7.03	7.02	7.01	6.99	6.98	29
30	**7.50**	**7.48**	**7.47**	**7.45**	**7.44**	**7.42**	**7.41**	**7.40**	**7.39**	**7.37**	**7.35**	**7.34**	**7.33**	**7.31**	**7.29**	**7.27**	**7.26**	**7.25**	**7.23**	**7.22**	**30**
31	7.75	7.73	7.71	7.70	7.68	7.66	7.65	7.64	7.63	7.61	7.60	7.58	7.57	7.55	7.53	7.51	7.50	7.49	7.47	7.46	31
32	8.00	7.98	7.96	7.95	7.93	7.91	7.90	7.89	7.88	7.86	7.84	7.82	7.81	7.79	7.78	7.76	7.75	7.73	7.71	7.70	32
33	8.25	8.23	8.21	8.20	8.18	8.16	8.15	8.13	8.12	8.10	8.09	8.07	8.05	8.03	8.01	8.00	7.99	7.97	7.95	7.94	33
34	8.50	8.48	8.47	8.45	8.43	8.41	8.40	8.38	8.37	8.35	8.34	8.32	8.30	8.28	8.26	8.24	8.23	8.22	8.20	8.18	34
35	8.75	8.73	8.71	8.69	8.68	8.66	8.65	8.63	8.61	8.59	8.58	8.56	8.54	8.52	8.50	8.48	8.47	8.46	8.44	8.42	35
36	9.00	8.98	8.96	8.94	8.92	8.91	8.90	8.88	8.86	8.84	8.82	8.80	8.78	8.76	8.75	8.73	8.71	8.70	8.68	8.66	36
37	9.25	9.23	9.21	9.19	9.17	9.15	9.14	9.12	9.10	9.08	9.07	9.05	9.03	9.01	8.99	8.97	8.95	8.94	8.92	8.90	37
38	9.50	9.48	9.46	9.44	9.42	9.40	9.39	9.37	9.35	9.33	9.31	9.29	9.27	9.26	9.24	9.21	9.20	9.18	9.16	9.14	38
39	9.75	9.73	9.71	9.69	9.67	9.65	9.64	9.62	9.60	9.58	9.55	9.53	9.51	9.50	9.48	9.46	9.44	9.43	9.40	9.38	39

Degrees Brix from 0.5 to 20.0	
Tenths of the Polariscope Reading	Per Cents Sucrose (Pol)
0.1	0.02
0.2	0.05
0.3	0.07
0.4	0.10
0.5	0.12
0.6	0.15
0.7	0.17
0.8	0.19
0.9	0.22

TABLE 36 (*Continued*)

SCHMITZ'S TABLE FOR SUCROSE (POL) FOR USE IN HORNE'S DRY LEAD METHOD WITH UNDILUTED SOLUTIONS

Pol Reading	Degrees Brix and Per Cents Sucrose																	Pol Reading
	11.5	12.0	12.5	13.0	13.5	14.0	14.5	15.0	15.5	16.0	16.5	17.0	17.5	18.0	18.5	19.0	19.5	
40	**9.97**	**9.95**	**9.93**	**9.91**	**9.89**	**9.87**	**9.85**	**9.83**	**9.81**	**9.79**	**9.77**	**9.75**	**9.73**	**9.71**	**9.69**	**9.67**	**9.65**	**40**
41	10.21	10.19	10.18	10.16	10.13	10.11	10.09	10.07	10.05	10.03	10.01	9.99	9.97	9.95	9.93	9.91	9.89	41
42	10.46	10.44	10.43	10.41	10.38	10.36	10.34	10.32	10.30	10.27	10.24	10.23	10.21	10.19	10.17	10.15	10.13	42
43	10.71	10.69	10.68	10.66	10.63	10.61	10.59	10.57	10.54	10.52	10.50	10.48	10.45	10.43	10.42	10.39	10.37	43
44	10.96	10.94	10.92	10.90	10.87	10.86	10.84	10.82	10.79	10.77	10.75	10.73	10.70	10.68	10.66	10.64	10.61	44
45	11.21	11.19	11.17	11.15	11.12	11.10	11.08	11.06	11.03	11.01	10.99	10.97	10.94	10.92	10.90	10.88	10.86	45
46	......	11.44	11.42	11.40	11.37	11.35	11.33	11.30	11.28	11.26	11.24	11.21	11.19	11.17	11.15	11.12	11.09	46
47	......	11.68	11.67	11.64	11.62	11.60	11.57	11.55	11.52	11.50	11.48	11.45	11.43	11.41	11.38	11.36	11.34	47
48	......	......	11.91	11.90	11.87	11.85	11.82	11.80	11.77	11.75	11.73	11.70	11.67	11.65	11.63	11.60	11.57	48
49	......	......	12.16	12.14	12.11	12.09	12.06	12.04	12.01	11.99	11.97	11.94	11.91	11.89	11.87	11.84	11.81	49
50	......	......	......	**12.39**	**12.36**	**12.34**	**12.31**	**12.29**	**12.26**	**12.24**	**12.21**	**12.19**	**12.16**	**12.14**	**12.11**	**12.09**	**12.06**	**50**
51	......	......	......	12.64	12.61	12.58	12.55	12.53	12.50	12.48	12.45	12.43	12.40	12.38	12.35	12.33	12.30	51
52	......	......	......	......	12.86	12.83	12.80	12.78	12.75	12.72	12.70	12.68	12.65	12.63	12.59	12.57	12.54	52
53	......	......	......	......	13.10	13.08	13.05	13.02	13.00	12.97	12.94	12.92	12.89	12.87	12.84	12.81	12.78	53
54	......	......	......	......	......	13.33	13.30	13.27	13.25	13.21	13.19	13.16	13.13	13.12	13.08	13.05	13.01	54
55	......	......	......	......	......	13.57	13.54	13.51	13.49	13.45	13.43	13.40	13.37	13.35	13.32	13.29	13.26	55
56	......	......	......	......	......	......	13.79	13.76	13.74	13.70	13.67	13.65	13.62	13.59	13.56	13.53	13.51	56
57							14.03	14.00	13.98	13.94	13.91	13.89	13.86	13.83	13.80	13.77	13.75	57
58							......	14.25	14.22	14.19	14.16	14.13	14.10	14.08	14.05	14.02	13.99	58
59							......	14.49	14.46	14.43	14.40	14.37	14.34	14.32	14.29	14.26	14.23	59
60							......	......	**14.71**	**14.68**	**14.65**	**14.62**	**14.59**	**14.56**	**14.53**	**14.50**	**14.47**	**60**
61							......	......	14.96	14.92	14.89	14.86	14.83	14.80	14.77	14.74	14.71	61
62										15.17	15.14	15.11	15.08	15.05	15.01	14.98	14.95	62
63										15.41	15.38	15.35	15.32	15.29	15.25	15.22	15.19	63
64										15.66	15.63	15.60	15.57	15.54	15.50	15.47	15.44	64
65										......	15.87	15.84	15.81	15.78	15.74	15.71	15.68	65
66										......	16.11	16.08	16.05	16.02	15.98	15.95	15.92	66
67										......	......	16.32	16.29	16.26	16.22	16.19	16.16	67
68										......	......	......	16.53	16.50	16.47	16.44	16.40	68
69										......	......	......	16.78	16.74	16.71	16.68	16.64	69
70										......	......	......	**17.02**	**16.99**	**16.95**	**16.92**	**16.88**	**70**
71										......	......	......	......	17.23	17.19	17.16	17.12	71

Degrees Brix from 11.5 to 22.5	
Tenths of the Polariscope Reading	Per Cents Sucrose (Pol)
0.1	0.02
0.2	0.05
0.3	0.07
0.4	0.10
0.5	0.12
0.6	0.15
0.7	0.17
0.8	0.19
0.9	0.22

Pol. R.	Brix, etc.	
	19.0	19.5
72	17.39	17.35
73	17.63	17.59
74	17.87	17.83
75	18.11	18.08
76	18.35	18.32

TABLE 36 (*Continued*)

SCHMITZ'S TABLE FOR SUCROSE (POL) FOR USE IN HORNE'S DRY LEAD METHOD WITH UNDILUTED SOLUTIONS

Pol Reading	Degrees Brix and Per Cents Sucrose									Pol Reading
	20.0	20.5	21.0	21.5	22.0	22.5	23.0	23.5	24.0	
40	**9.63**	**9.61**	**9.59**	**9.57**	**9.55**	**9.53**	**9.51**	**9.49**	**9.47**	**40**
41	9.87	9.85	9.83	9.81	9.79	9.77	9.75	9.73	9.71	41
42	10.11	10.09	10.07	10.05	10.03	10.01	9.99	9.97	9.95	42
43	10.35	10.33	10.31	10.29	10.27	10.25	10.23	10.21	10.19	43
44	10.59	10.57	10.55	10.53	10.50	10.48	10.47	10.45	10.42	44
45	10.83	10.81	10.79	10.77	10.74	10.72	10.70	10.68	10.65	45
46	11.07	11.05	11.03	11.01	10.98	10.96	10.94	10.91	10.89	46
47	11.31	11.29	11.27	11.25	11.21	11.20	11.18	11.15	11.13	47
48	11.55	11.53	11.51	11.49	11.46	11.44	11.42	11.39	11.37	48
49	11.79	11.77	11.75	11.73	11.70	11.68	11.66	11.63	11.61	49
50	**12.04**	**12.01**	**11.99**	**11.96**	**11.94**	**11.91**	**11.89**	**11.86**	**11.84**	**50**
51	12.28	12.25	12.23	12.20	12.18	12.15	12.13	12.10	12.08	51
52	12.52	12.49	12.47	12.44	12.42	12.39	12.36	12.34	12.31	52
53	12.76	12.73	12.71	12.68	12.66	12.63	12.60	12.58	12.54	53
54	12.99	12.97	12.95	12.92	12.90	12.87	12.84	12.82	12.78	54
55	13.23	13.21	13.18	13.16	13.13	13.10	13.07	13.05	13.02	55
56	13.48	13.45	13.42	13.40	13.37	13.34	13.31	13.29	13.25	56
57	13.72	13.69	13.66	13.64	13.71	13.58	13.55	13.53	13.49	57
58	13.96	13.93	13.90	13.88	13.85	13.82	13.78	13.76	13.73	58
59	14.20	14.17	14.14	14.12	14.09	14.06	14.02	13.99	13.97	59
60	**14.45**	**14.42**	**14.39**	**14.36**	**14.33**	**14.30**	**14.27**	**14.24**	**14.21**	**60**
61	14.69	14.66	14.63	14.60	14.57	14.54	14.51	14.48	14.44	61
62	14.93	14.90	14.87	14.84	14.81	14.78	14.75	14.71	14.68	62
63	15.17	15.14	15.11	15.08	15.05	15.01	14.98	14.95	14.92	63
64	15.41	15.38	15.35	15.32	15.29	15.26	15.22	15.19	15.16	64
65	15.65	15.62	15.59	15.56	15.53	15.50	15.46	15.43	15.40	65
66	15.89	15.86	15.83	15.80	15.76	15.73	15.70	15.66	15.63	66
67	16.13	16.10	16.07	16.04	16.00	15.97	15.94	15.90	15.87	67
68	16.37	16.34	16.31	16.28	16.24	16.21	16.18	16.14	16.11	68
69	16.61	16.58	16.55	16.52	16.48	16.45	16.41	16.37	16.35	69
70	**16.86**	**16.82**	**16.79**	**16.75**	**16.72**	**16.68**	**16.65**	**16.61**	**16.58**	**70**
71	17.10	17.06	17.02	16.99	16.95	16.91	16.88	16.85	16.81	71
72	17.34	17.30	17.26	17.23	17.19	17.15	17.12	17.09	17.05	72
73	17.58	17.54	17.50	17.47	17.43	17.39	17.36	17.33	17.29	73
74	17.82	17.78	17.74	17.71	17.67	17.63	17.60	17.57	17.52	74
75	18.06	18.02	17.98	17.95	17.91	17.87	17.83	17.80	17.76	75
76	18.30	18.26	18.22	18.19	18.15	18.11	18.07	18.04	18.00	76
77	18.54	18.50	18.46	18.43	18.39	18.35	18.31	18.28	18.25	77
78	18.78	18.74	18.70	18.67	18.63	18.59	18.55	18.51	18.47	78
79	19.02	18.98	18.94	18.91	18.87	18.83	18.79	18.75	18.71	79
80	**19.26**	**19.22**	**19.18**	**19.14**	**19.10**	**19.06**	**19.02**	**18.98**	**18.94**	**80**
81	......	19.46	19.42	19.38	19.34	19.30	19.26	19.22	19.18	81
82	......	19.70	19.66	19.62	19.58	19.54	19.50	19.46	19.42	82
83	......	......	19.90	19.86	19.82	19.78	19.74	19.70	19.66	83
84	......	......	20.14	20.10	20.06	20.01	19.97	19.94	19.90	84
85	......	......	......	20.34	20.30	20.25	20.21	20.17	20.12	85

Degrees Brix from 23 to 24

Tenths of the Polariscope Reading	Per Cents Sucrose (Pol)	Tenths of the Polariscope Reading	Per Cents Sucrose (Pol)
0.1	0.02	0.5	0.12
0.2	0.05	0.6	0.14
0.3	0.07	0.7	0.17
0.4	0.09	0.8	0.19
		0.9	0.21

TABLE 37

AVAILABLE SUCROSE TABLE *

Values of the factor $1.4 - \frac{40}{\text{Coefficient purity}} \times 100$, for coefficients of purity ranging from 77 to 93, advancing by tenths. See p. 614 for suggestions relative to the use of this table.

	SUCROSE (pol)									
Purity Coefficient	0.0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
77	88.05	88.12	88.19	88.25	88.32	88.39	88.45	88.52	88.59	88.65
78	88.72	88.78	88.85	88.92	88.98	89.04	89.11	89.18	89.24	89.31
79	89.37	89.43	89.50	89.56	89.63	89.69	89.75	89.81	89.88	89.94
80	90.00	90.07	90.13	90.19	90.25	90.32	90.38	90.44	90.50	90.56
81	90.62	90.68	90.74	90.80	90.86	90.92	90.98	91.04	91.10	91.16
82	91.22	91.28	91.34	91.39	91.45	91.51	91.57	91.63	91.69	91.75
83	91.81	91.87	91.93	91.99	92.04	92.10	92.15	92.21	92.27	92.32
84	92.38	92.44	92.50	92.55	92.61	92.67	92.72	92.78	92.84	92.89
85	92.94	93.00	93.06	93.11	93.16	93.22	93.28	93.33	93.38	93.43
86	93.49	93.54	93.59	93.64	93.70	93.75	93.80	93.85	93.91	93.97
87	94.02	94.07	94.12	94.18	94.23	94.28	94.34	94.39	94.44	94.49
88	94.55	94.60	94.65	94.70	94.76	94.81	94.86	94.91	94.95	95.01
89	95.06	95.11	95.16	95.21	95.26	95.31	95.36	95.41	95.46	95.51
90	95.56	95.61	95.65	95.70	95.75	95.80	95.85	95.90	95.95	96.00
91	96.04	96.09	96.14	96.18	96.23	96.28	96.33	96.37	96.42	96.47
92	96.52	96.57	96.62	96.67	96.71	96.75	96.80	96.85	96.90	96.95
93	96.99	97.04	97.08	97.13	97.17	97.22	97.27	97.31	97.35	97.40

* Methods of Chemical Control, H. C. Prinsen Geerligs.

TABLE 37*a*

AVAILABLE 96° SUGAR (97.0 PURITY, 1.03 MOISTURE) FOR JUICE OF VARIOUS PURITIES

(Calculated by E. M. Copp (1943), using *SJM* formula and 28.57 purity molasses and then dividing resulting retention by 0.96. See p. 616 for method of application. Replaces Table 37*b* of previous edition.)

Purity Coefficient	0.0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
77	92.88	92.95	93.01	93.08	93.16	93.22	93.29	93.36	93.44	93.51
78	93.58	93.65	93.71	93.78	93.85	93.92	93.99	94.06	94.13	94.19
79	94.26	94.33	94.40	94.46	94.53	94.60	94.66	94.73	94.79	94.86
80	94.93	94.99	95.06	95.12	95.19	95.25	95.32	95.38	95.45	95.51
81	95.58	95.64	95.71	95.77	95.84	95.90	95.96	96.02	96.09	96.15
82	96.21	96.27	96.34	96.40	96.46	96.52	96.59	96.65	96.71	96.77
83	96.84	96.89	96.95	97.01	97.08	97.14	97.20	97.26	97.32	97.38
84	97.44	97.50	97.56	97.62	97.68	97.73	97.79	97.85	97.91	97.96
85	98.02	98.08	98.15	98.20	98.26	98.32	98.38	98.43	98.49	98.55
86	98.61	98.66	98.72	98.78	98.84	98.89	98.95	99.00	99.06	99.11
87	99.17	99.22	99.28	99.33	99.39	99.44	99.50	99.55	99.61	99.66
88	99.72	99.77	99.83	99.88	99.94	99.99	100.04	100.09	100.15	100.20
89	100.26	100.31	100.37	100.42	100.47	100.52	100.58	100.63	100.68	100.73
90	100.78	100.83	100.89	100.94	100.99	101.04	101.10	101.15	101.20	101.25
91	101.31	101.35	101.40	101.45	101.50	101.55	101.61	101.66	101.71	101.76
92	101.82	101.86	101.91	101.95	102.00	102.05	102.10	102.15	102.20	102.24
93	102.29	102.34	102.38	102.43	102.47	102.52	102.57	102.62	102.67	102.72

TABLE 38

TABLE OF BOILING HOUSE RECOVERIES (RETENTIONS) FOR 100° PURITY SUGAR AND DIFFERENT MOLASSES AND JUICE PURITIES

(Calculated (1943) by *SJM* formula of Noël Deerr. See p. 613)

Purity of Juice	Purity of Molasses																
	25	26	27	28	28.57	29	30	31	32	33	34	35	36	37	38	39	40
72	87.04	86.34	85.55	84.88	84.44	84.12	83.33	82.53	81.70	80.85	79.97	79.06	78.13	77.16	76.16	75 14	74.07
73	87.67	87.00	86.32	85.62	85.21	84.89	84.15	83.38	82.60	81.78	80.95	80.08	79.20	78.28	77.33	76.35	75.34
74	88.29	87.66	87.00	86.34	85.95	85.65	84.94	84.21	83.47	82.69	81.90	81.08	80.24	79.37	78.47	77.54	76.58
75	88.89	88.29	87.67	87.04	86.67	86.38	85.71	85.02	84.31	83.58	82.83	82.05	81.25	80.42	79.57	78.69	77.78
76	89.47	88.90	88.32	87.72	87.39	87.10	86.47	85.81	85.14	84.45	83.73	83.00	82 24	81.45	80.65	79.81	78.95
77	90.04	89.51	88.95	88.38	88.05	87.80	87.20	86.58	85.94	85.29	84.61	83.92	83.20	82.44	81.69	80.90	80.09
78	90.60	90.09	89.57	89.03	88.72	88.48	87.91	87.33	86.73	86.11	85.47	84.81	84.13	83.44	82.71	81.97	81.20
79	91.14	90.66	90.17	89.66	89.37	89.14	88.61	88.06	87.49	86.91	86.30	85.69	85.05	84.39	83.71	83.00	82.28
80	91.66	91.22	90.75	90.28	90.00	89.79	89.29	88.77	88.23	87.69	87.12	86.54	85.94	85.32	84.68	84.02	83.33
81	92.18	91.76	91.32	90.88	90.62	90.42	89.95	89.49	88.96	88.45	87.92	87.37	86.81	86.22	85.62	85.00	84.36
82	92.68	92.29	91.88	91.46	91.22	91.03	90.59	90.14	89.67	89.19	88.69	88.18	87.65	87.11	86.51	85.97	85.37
83	93.17	92.80	92.42	92.04	91.81	91.63	91.22	90.80	90.36	89.91	89.45	88.97	88.48	87.97	87.45	86.90	86.35
84	93.65	93.31	92.95	92.59	92.38	92.22	91.84	91.44	91.04	90.62	90.19	89.74	89.29	88.81	88.33	87.82	87.30
85	94.12	93.80	93.47	93.14	92.94	92.79	92.44	92.07	91.70	91.31	90.91	90.50	90.07	89.64	89.18	88.72	88.24
86	94.57	94.28	93.98	93.67	93.49	93.35	93.02	92.69	92.34	91.98	91.61	91.23	90.84	90.44	90.02	89.59	89.15
87	95.02	94.75	94.47	94.19	94.02	93.90	93.60	93.29	92.97	92.64	92.30	91.95	91.59	91.22	90.84	90.45	90.04
88	95.45	95.21	94.96	94.70	94.55	94.43	94.16	93.87	93.58	93.28	92.98	92.66	92.33	91.99	91.64	91.28	90.91
89	95.88	95.66	95.43	95.19	95.06	94.95	94.70	94.45	94.18	93.91	93.63	93.34	93.05	92.74	92.42	92.10	91.76
90	96.30	96.10	95.89	95.68	95.56	95.46	95.24	95.01	94.77	94.53	94.28	94.02	93.25	93.47	93.19	92.90	92.59
91	96.70	96.53	96.34	96.15	96.04	95.96	95.76	95.56	95.35	95.13	94.90	94.67	94.44	94.19	93.94	93.69	93.41
92	97.10	96.94	96.78	96.62	96.52	96.45	96.27	96.09	95.91	95.72	95.52	95.32	95.11	94.89	94.67	94.44	94.20

TABLE 39

CONVERSION OF SUCROSE (POL) IN SUGAR OF ANY GIVEN PURITY TO "96° TEST"

(97° Purity = 96° Pol and 1.03 H_2O)

(E. M. Copp)

Purity of Raw Sugar	Purities of Final Molasses									
	24	26	28	28.57	30	32	34	36	38	40
96.0	1.0380	1.0377	1.0373	1.0373	1.0368	1.0363	1.0358	1.0353	1.0348	1.0341
.1	1.0385	1.0381	1.0377	1.0376	1.0373	1.0369	1.0364	1.0360	1.0354	1.0348
.2	1.0388	1.0385	1.0381	1.0380	1.0378	1.0374	1.0370	1.0365	1.0361	1.0356
.3	1.0392	1.0389	1.0386	1.0386	1.0383	1.0379	1.0376	1.0372	1.0368	1.0364
.4	1.0395	1.0393	1.0390	1.0390	1.0388	1.0385	1.0382	1.0378	1.0375	1.0371
.5	1.0399	1.0397	1.0395	1.0395	1.0392	1.0390	1.0388	1.0385	1.0382	1.0379
.6	1.0403	1.0401	1.0399	1.0399	1.0397	1.0396	1.0393	1.0391	1.0389	1.0386
.7	1.0406	1.0405	1.0404	1.0404	1.0402	1.0401	1.0399	1.0398	1.0396	1.0394
.8	1.0410	1.0409	1.0408	1.0408	1.0407	1.0406	1.0405	1.0404	1.0403	1.0402
.9	1.0413	1.0413	1.0412	1.0412	1.0412	1.0411	1.0411	1.0410	1.0410	1.0409
97.0	1.0417	1.0417	1.0417	1.0417	1.0417	1.0417	1.0417	1.0417	1.0417	1.0417
.1	1.0420	1.0421	1.0421	1.0421	1.0421	1.0422	1.0422	1.0423	1.0424	1.0424
.2	1.0424	1.0424	1.0425	1.0426	1.0426	1.0427	1.0428	1.0429	1.0430	1.0432
.3	1.0427	1.0428	1.0430	1.0431	1.0431	1.0433	1.0434	1.0436	1.0437	1.0439
.4	1.0431	1.0432	1.0434	1.0435	1.0436	1.0438	1.0440	1.0442	1.0444	1.0447
.5	1.0434	1.0436	1.0438	1.0439	1.0440	1.0443	1.0445	1.0448	1.0451	1.0454
.6	1.0437	1.0440	1.0443	1.0444	1.0445	1.0448	1.0452	1.0454	1.0458	1.0462
.7	1.0441	1.0444	1.0447	1.0448	1.0450	1.0453	1.0457	1.0461	1.0465	1.0469
.8	1.0445	1.0448	1.0451	1.0453	1.0455	1.0459	1.0463	1.0467	1.0472	1.0476
.9	1.0448	1.0452	1.0456	1.0457	1.0460	1.0464	1.0468	1.0473	1.0478	1.0484
98.0	1.0452	1.0456	1.0460	1.0462	1.0464	1.0469	1.0474	1.0479	1.0485	1.0491
.1	1.0455	1.0459	1.0464	1.0466	1.0469	1.0474	1.0480	1.0486	1.0492	1.0499
.2	1.0458	1.0463	1.0468	1.0470	1.0474	1.0479	1.0485	1.0492	1.0499	1.0506
.3	1.0462	1.0467	1.0473	1.0475	1.0478	1.0485	1.0491	1.0498	1.0505	1.0513
.4	1.0465	1.0471	1.0477	1.0479	1.0483	1.0490	1.0497	1.0504	1.0512	1.0521
.5	1.0469	1.0475	1.0481	1.0483	1.0488	1.0495	1.0502	1.0510	1.0519	1.0528
.6	1.0472	1.0479	1.0485	1.0488	1.0492	1.0500	1.0508	1.0516	1.0526	1.0535
.7	1.0476	1.0482	1.0489	1.0492	1.0497	1.0505	1.0513	1.0523	1.0532	1.0543
.8	1.0479	1.0486	1.0494	1.0496	1.0502	1.0510	1.0519	1.0529	1.0539	1.0550
.9	1.0482	1.0490	1.0498	1.0501	1.0506	1.0515	1.0525	1.0535	1.0546	1.0557
99.0	1.0486	1.0494	1.0502	1.0505	1.0511	1.0520	1.0530	1.0541	1.0552	1.0564

Note that table is for converting sucrose (pol) in sugar per cent cane (not the sugar itself per cent cane) to 96° test. Purity of sugar may be taken as pol plus moisture; molasses purity to nearest whole degree.

Example. Sucrose (pol) in sugar % cane = 12.74
Purity of sugar = 97.8 (97.2 pol + 0.6 H_2O)
Purity of molasses = 35.1
For 97.8 and 35.1 find 1.0465 in the table.

Then 1.0465 × 12.74 = 13.34, the equivalent yield in 96° sugar.

Another application of this table is explained on p. 618 in finding the retention in equivalent 96° test sugar.

TABLE 40

REDUCED BOILING HOUSE RECOVERY ESG (R_{85}) CORRESPONDING TO VIRTUAL MOLASSES OF VARIOUS PURITIES

Calculated (1943) from formula (see p. 621)

$$R_{85} = 100 \times \frac{100(85 - \text{Virt. Mol.})}{85(100 - \text{Virt. Mol.})}$$

	0.0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
26	93.80	93.77	93.73	93.70	93.67	93.64	93.60	93.57	93.54	93.51
27	93.47	93.44	93.40	93.36	93.33	93.30	93.26	93.23	93.20	93.17
28	93.14	93.11	93.08	93.05	93.01	92.98	92.94	92.80	92.87	92.83
29	92.79	92.76	92.72	92.69	92.65	92.62	92.58	92.54	92.51	92.47
30	92.44	92.40	92.36	92.33	92.29	92.26	92.22	92.18	92.15	92.11
31	92.07	92.04	92.00	91.96	91.92	91.88	91.85	91.81	91.77	91.73
32	91.70	91.66	91.62	91.58	91.54	91.50	91.46	91.43	91.39	91.35
33	91.31	91.27	91.23	91.19	91.15	91.11	91.07	91.03	90.99	90.95
34	90.91	90.87	90.83	90.79	90.75	90.70	90.66	90.62	90.58	90.54
35	90.50	90.46	90.41	90.37	90.33	90.29	90.24	90.20	90.16	90.12
36	90.07	90.03	89.99	89.94	89.90	89.86	89.81	89.77	89.72	89.68
37	89.64	89.59	89.55	89.50	89.46	89.41	89.37	89.32	89.28	89.23
38	89.18	89.14	89.09	89.05	89.00	88.95	88.91	88.86	88.81	88.76
39	88.72	88.67	88.62	88.57	88.53	88.48	88.43	88.38	88.33	88.28
40	88.24	88.19	88.14	88.09	88.04	87.99	87.94	87.89	87.84	87.79

TABLE 41

Expanded Horne's Table for the Calculation of Coefficients of purity

(Explanatory)

The fifth edition of this book (1915) included a table by Dr. W. D. Horne for use in calculating coefficients of purity with his dry lead defecation. The range of the table was from 15° to 16° Brix, which limited its usefulness. This table has been expanded to include all degrees Brix from 12 to 20. The table will prove useful in vacuum pan and crystallizer control work and in refinery routine control. (See page 532 for method of making purities.) In the table on the pages following, opposite the degree Brix corrected to 20° C. and under the direct polariscope reading of the solution (after clarification with Horne's dry lead) will be found the coefficient of purity.

The table has been calculated using E. W. Rice's modification of Casamajor's formula for "water purities." The Rice formula is for densities at 20° C., true cubic centimeters (ml), and a normal weight of 26.0 grams, whereas Casamajor's was for the older units at 17½° C. and 26.048 normal weights. The formula according to Rice is

$$\text{Factor} = \frac{26 \times 100}{99.718 \times \text{sp. gr.} \times \text{Brix}}$$

The direct polariscope reading of the solution times the factor corresponding to the Brix gives the coefficient of purity as shown in the expanded Horne's table. The factors according to Rice's formula follow:

Purity Factors for Use with Dry Lead Defecation.—(E. W. Rice) *

°Bx.	0.0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
0	.....	260.6336	130.2660	86.8102	65.0829	52.0461	43.3549	37.1469	32.4909	28.8695
1	25.9725	23.6022	21.6269	19.9556	18.5230	17.2814	16.1951	15.2365	14.3845	13.6221
2	12.9360	12.3152	11.7508	11.2355	10.7632	10.3286	9.9275	9.5561	9.2113	8.8901
3	8.5905	8.3102	8.0473	7.8004	7.5680	7.3490	7.1420	6.9463	6.7608	6.5849
4	6.4178	6.2588	6.1074	5.9630	5.8253	5.6936	5.5676	5.4470	5.3314	5.2206
5	5.1142	5.0120	4.9136	4.8191	4.7280	4.6402	4.5555	4.4738	4.3950	4.3188
6	4.2451	4.1739	4.1050	4.0382	3.9755	3.9109	3.8501	3.7912	3.7339	3.6784
7	3.6244	3.5720	3.5209	3.4713	3.4231	3.3761	3.3304	3.2858	3.2424	3.2001
8	3.1589	3.1186	3.0794	3.0411	3.0037	2.9671	2.9315	2.8966	2.8626	2.8293
9	2.7968	2.7649	2.7338	2.7033	2.6735	2.6443	2.6157	2.5877	2.5603	2.5334
10	2.5071	2.4813	2.4560	2.4312	2.4068	2.3830	2.3596	2.3366	2.3140	2.2919
11	2.2701	2.2488	2.2278	2.2072	2.1870	2.1671	2.1475	2.1283	2.1095	2.0909
12	2.0727	2.0547	2.0370	2.0197	2.0026	1.9858	1.9692	1.9529	1.9369	1.9211
13	1.9056	1.8903	1.8752	1.8603	1.8457	1.8313	1.8171	1.8031	1.7893	1.7757
14	1.7623	1.7491	1.7361	1.7233	1.7106	1.6981	1.6858	1.6737	1.6617	1.6499
15	1.6382	1.6267	1.6154	1.6042	1.5931	1.5822	1.5714	1.5608	1.5503	1.5399
16	1.5296	1.5195	1.5095	1.4997	1.4899	1.4803	1.4708	1.4614	1.4521	1.4429
17	1.4338	1.4249	1.4160	1.4072	1.3986	1.3900	1.3816	1.3732	1.3649	1.3567
18	1.3487	1.3407	1.3328	1.3249	1.3172	1.3095	1.3020	1.2945	1.2871	1.2797
19	1.2725	1.2653	1.2582	1.2511	1.2442	1.2373	1.2305	1.2237	1.2171	1.2104
20	1.2039	1.1974	1.1910	1.1846	1.1784	1.1721	1.1660	1.1598	1.1538	1.1478
21	1.1419	1.1360	1.1302	1.1244	1.1187	1.1130	1.1074	1.1018	1.0963	1.0909
22	1.0855	1.0801	1.0748	1.0696	1.0643	1.0592	1.0540	1.0490	1.0439	1.0389
23	1.0340	1.0291	1.0242	1.0194	1.0146	1.0099	1.0052	1.0005	.9959	.9913
24	.9868	.9823	.9778	.9734	.9690	.9647	.9603	.9560	.9518	.9476
25	.9434									

* "Facts about Sugar," October 29, 1927.

TABLE 41 (*Continued*)

EXPANDED HORNE'S TABLE OF COEFFICIENTS OF PURITY

Pol. =	**15.0**	**15.2**	**15.4**	**15.6**	**15.8**	**16.0**	**16.2**	**16.4**	**16.6**	**16.8**	**17.0**	**17.2**	**17.4**	**17.6**	**17.8**	**18.0**	**18.2**	**18.4**	**18.6**	**18.8**	**19.0**	**19.2**	**19.4**	**19.6**	**19.8**
12.0	31.1	31.5	31.9	32.3	32.7	33.2	33.6	34.0	34.4	34.8	35.2	35.7	36.1	36.5	36.9	37.3	37.7	38.1	38.6	39.0	39.4	39.8	40.2	40.6	41.0
.1	30.8	31.2	31.6	32.1	32.5	32.9	33.3	33.7	34.1	34.5	34.9	35.3	35.8	36.2	36.6	37.0	37.4	37.8	38.2	38.6	39.0	39.5	39.9	40.3	40.7
.2	30.6	31.0	31.4	31.8	32.2	32.6	33.0	33.4	33.8	34.2	34.6	35.0	35.4	35.9	36.3	36.7	37.1	37.5	37.9	38.3	38.7	39.1	39.5	39.9	40.3
.3	30.3	30.7	31.1	31.5	31.9	32.3	32.7	33.1	33.5	33.9	34.3	34.7	35.1	35.5	36.0	36.4	36.8	37.2	37.6	38.0	38.4	38.8	39.2	39.6	40.0
.4	30.0	30.4	30.8	31.2	31.6	32.0	32.4	32.8	33.2	33.6	34.0	34.4	34.8	35.2	35.6	36.0	36.4	36.8	37.2	37.6	38.0	38.5	38.9	39.3	39.7
.5	29.8	30.2	30.6	31.0	31.4	31.8	32.2	32.6	33.0	33.4	33.8	34.2	34.6	35.0	35.3	35.7	36.1	36.5	36.9	37.3	37.7	38.1	38.5	38.9	39.3
.6	29.5	29.9	30.3	30.7	31.1	31.5	31.9	32.3	32.7	33.1	33.5	33.9	34.3	34.7	35.1	35.4	35.8	36.2	36.6	37.0	37.4	37.8	38.2	38.6	39.0
.7	29.3	29.7	30.1	30.5	30.9	31.2	31.6	32.0	32.4	32.8	33.2	33.6	34.0	34.4	34.8	35.2	35.5	35.9	36.2	36.7	37.1	37.5	37.9	38.3	38.7
.8	29.1	29.4	29.8	30.2	30.6	31.0	31.4	31.8	32.2	32.5	32.9	33.3	33.7	34.1	34.5	34.9	35.3	35.6	36.0	36.4	36.8	37.2	37.6	38.0	38.4
.9	28.8	29.2	29.6	30.0	30.4	30.7	31.1	31.5	31.9	32.3	32.7	33.0	33.4	33.8	34.2	34.6	35.0	35.3	35.7	36.1	36.5	36.9	37.3	37.7	38.0

Pol. =	**20.0**	**20.2**	**20.4**	**20.6**	**20.8**	**21.0**	**21.2**	**21.4**	**21.6**	**21.8**	**22.0**	**22.2**	**22.4**	**22.6**	**22.8**	**23.0**	**23.2**	**23.4**	**23.6**	**23.8**	**24.0**	**24.2**	**24.4**	**24.6**	**24.8**
12.0	41.5	41.9	42.3	42.7	43.1	43.5	44.0	44.4	44.8	45.2	45.6	46.0	46.4	46.8	47.3	47.7	48.1	48.5	48.9	49.3	49.7	50.2	50.6	51.0	51.4
.1	41.1	41.5	41.9	42.3	42.7	43.1	43.6	44.0	44.4	44.8	45.2	45.6	46.0	46.4	46.8	47.3	47.7	48.1	48.5	48.9	49.3	49.7	50.1	50.5	51.0
.2	40.7	41.1	41.6	42.0	42.4	42.8	43.2	43.6	44.0	44.4	44.8	45.2	45.6	46.0	46.4	46.9	47.3	47.7	48.1	48.5	48.9	49.3	49.7	50.1	50.5
.3	40.4	40.8	41.2	41.6	42.0	42.4	42.8	43.2	43.6	44.0	44.4	44.8	45.2	45.6	46.0	46.5	46.9	47.3	47.7	48.1	48.5	48.9	49.3	49.7	50.1
.4	40.1	40.5	40.9	41.3	41.7	42.1	42.5	42.9	43.3	43.7	44.1	44.5	44.9	45.3	45.7	46.1	46.5	46.9	47.3	47.7	48.1	48.5	48.9	49.3	49.7
.5	39.7	40.1	40.5	40.9	41.3	41.7	42.1	42.5	42.9	43.3	43.7	44.1	44.5	44.9	45.3	45.7	46.1	46.5	46.9	47.3	47.7	48.1	48.5	48.9	49.2
.6	39.4	39.8	40.2	40.6	41.0	41.4	41.7	42.1	42.5	42.9	43.3	43.7	44.1	44.5	44.9	45.3	45.7	46.1	46.5	46.9	47.3	47.7	48.0	48.4	48.8
.7	39.1	39.4	39.8	40.2	40.6	41.0	41.4	41.8	42.2	42.6	43.0	43.4	43.7	44.1	44.5	44.9	45.3	45.6	46.1	46.5	46.9	47.3	47.7	48.0	48.4
.8	38.7	39.1	39.5	39.9	40.3	40.7	41.1	41.4	41.8	42.2	42.6	43.0	43.4	43.8	44.2	44.5	44.9	45.3	45.7	46.1	46.5	46.9	47.3	47.6	48.0
.9	38.4	38.8	39.2	39.6	40.0	40.3	40.7	41.1	41.5	41.9	42.3	42.6	43.0	43.4	43.8	44.2	44.6	45.0	45.3	45.7	46.1	46.5	46.9	47.3	47.6

Pol. =	**25.0**	**25.2**	**25.4**	**25.6**	**25.8**	**26.0**	**26.2**	**26.4**	**26.6**	**26.8**	**27.0**	**27.2**	**27.4**	**27.6**	**27.8**	**28.0**	**28.2**	**28.4**	**28.6**	**28.8**	**29.0**	**29.2**	**29.4**	**29.6**	**29.8**
12.0	51.8	52.2	52.6	53.1	53.5	53.9	54.3	54.7	55.1	55.5	56.0	56.4	56.8	57.2	57.6	58.0	58.5	58.9	59.3	59.7	60.1	60.5	60.9	61.4	61.8
.1	51.4	51.8	52.2	52.6	53.0	53.4	53.8	54.2	54.7	55.1	55.5	55.9	56.3	56.7	57.1	57.5	57.9	58.4	58.8	59.2	59.6	60.0	60.4	60.8	61.2
.2	50.9	51.3	51.7	52.1	52.6	53.0	53.4	53.8	54.2	54.6	55.0	55.4	55.8	56.2	56.6	57.0	57.4	57.9	58.3	58.7	59.1	59.5	59.9	60.3	60.7
.3	50.5	50.9	51.3	51.7	52.1	52.5	52.9	53.3	53.7	54.1	54.5	54.9	55.3	55.7	56.1	56.6	57.0	57.4	57.8	58.2	58.6	59.0	59.4	59.8	60.2
.4	50.1	50.5	50.9	51.3	51.7	52.1	52.5	52.9	53.3	53.7	54.1	54.5	54.9	55.3	55.7	56.1	56.5	56.9	57.3	57.7	58.1	58.5	58.9	59.3	59.7
.5	49.6	50.0	50.4	50.8	51.2	51.6	52.0	52.4	52.8	53.2	53.6	54.0	54.4	54.8	55.2	55.6	56.0	56.4	56.8	57.2	57.6	58.0	58.4	58.8	59.2
.6	49.2	49.6	50.0	50.4	50.8	51.2	51.6	52.0	52.4	52.8	53.1	53.6	54.0	54.3	54.7	55.1	55.5	55.9	56.3	56.7	57.1	57.5	57.9	58.3	58.7
.7	48.8	49.2	49.6	50.0	50.4	50.8	51.2	51.6	51.9	52.3	52.7	53.1	53.5	53.9	54.3	54.7	55.1	55.5	55.9	56.2	56.6	57.0	57.4	57.8	58.2
.8	48.4	48.8	49.2	49.6	50.0	50.4	50.7	51.1	51.5	51.9	52.3	52.7	53.1	53.5	53.9	54.2	54.6	55.0	55.4	55.8	56.2	56.6	56.9	57.3	57.7
.9	48.0	48.4	48.8	49.2	49.6	50.0	50.3	50.7	51.1	51.5	51.9	52.3	52.6	53.0	53.4	53.8	54.2	54.6	54.9	55.3	55.7	56.1	56.5	56.9	57.3

TABLE 41 (*Continued*)

EXPANDED HORNE'S TABLE OF COEFFICIENTS OF PURITY

Pol. =	**30.0**	**30.2**	**30.4**	**30.6**	**30.8**	**31.0**	**31.2**	**31.4**	**31.6**	**31.8**	**32.0**	**32.2**	**32.4**	**32.6**	**32.8**	**33.0**	**33.2**	**33.4**	**33.6**	**33.8**	**34.0**	**34.2**	**34.4**	**34.6**	**34.8**
12.0	62.2	62.6	63.0	63.4	63.8	64.3	64.7	65.1	65.5	65.9	66.3	66.7	67.2	67.6	68.0	68.4	68.8	69.2	69.6	70.1	70.5	70.9	71.3	71.7	72.1
.1	61.6	62.1	62.5	62.9	63.3	63.7	64.1	64.5	64.9	65.3	65.8	66.2	66.6	67.0	67.4	67.8	68.2	68.6	69.0	69.4	69.9	70.3	70.7	71.1	71.5
.2	61.1	61.5	61.9	62.3	62.7	63.1	63.6	64.0	64.4	64.8	65.2	65.6	66.0	66.4	66.8	67.2	67.6	68.0	68.4	68.9	69.3	69.7	70.1	70.5	70.9
.3	60.6	61.0	61.4	61.8	62.2	62.6	63.0	63.4	63.8	64.2	64.6	65.0	65.4	65.8	66.2	66.7	67.1	67.5	67.9	68.3	68.7	69.1	69.5	69.9	70.3
.4	60.1	60.5	60.9	61.3	61.7	62.1	62.5	62.9	63.3	63.7	64.1	64.5	64.9	65.3	65.7	66.1	66.5	66.9	67.3	67.7	68.1	68.5	68.9	69.3	69.7
.5	59.6	60.0	60.4	60.8	61.2	61.6	62.0	62.4	62.8	63.1	63.5	63.9	64.3	64.7	65.1	65.5	65.9	66.3	66.7	67.1	67.5	67.9	68.3	68.7	69.1
.5	59.1	59.5	59.9	60.3	60.7	61.0	61.4	61.8	62.2	62.6	63.0	63.4	63.8	64.2	64.6	65.0	65.4	65.8	66.2	66.6	67.0	67.3	67.7	68.1	68.5
.7	58.6	59.0	59.4	59.8	60.1	60.5	60.9	61.3	61.7	62.1	62.5	62.9	63.3	63.7	64.1	64.4	64.8	65.2	65.6	66.0	66.4	66.8	67.2	67.6	68.0
.8	58.1	58.5	58.9	59.3	59.7	60.0	60.4	60.8	61.2	61.6	62.0	62.4	62.8	63.1	63.5	63.9	64.3	64.7	65.1	65.5	65.9	66.2	66.6	67.0	67.4
.9	57.6	58.0	58.4	58.8	59.2	59.6	60.0	60.3	60.7	61.1	61.5	61.9	62.2	62.6	63.0	63.4	63.8	64.2	64.5	64.9	65.3	65.7	66.1	66.5	66.9

Pol. =	**35.0**	**35.2**	**35.4**	**35.6**	**35.8**	**36.0**	**36.2**	**36.4**	**36.6**	**36.8**	**37.0**	**37.2**	**37.4**	**37.6**	**37.8**	**38.0**	**38.2**	**38.4**	**38.6**	**38.8**	**39.0**	**39.2**	**39.4**	**39.6**	**39.8**
12.0	72.5	73.0	73.4	73.8	74.2	74.6	75.0	75.4	75.9	76.3	76.7	77.1	77.5	77.9	78.3	78.8	79.2	79.6	80.0	80.4	80.8	81.2	81.7	82.1	82.5
.1	71.9	72.3	72.7	73.1	73.6	74.0	74.4	74.8	75.2	75.6	76.0	76.4	76.8	77.3	77.7	78.1	78.5	78.9	79.3	79.7	80.1	80.5	81.0	81.4	81.8
.2	71.3	71.7	72.1	72.5	72.9	73.3	73.7	74.1	74.6	75.0	75.4	75.8	76.2	76.6	77.0	77.4	77.8	78.2	78.6	79.0	79.4	79.9	80.3	80.7	81.1
.3	70.7	71.1	71.5	71.9	72.3	72.7	73.1	73.5	73.9	74.3	74.7	75.1	75.5	75.9	76.3	76.7	77.2	77.6	78.0	78.4	78.8	79.2	79.6	80.0	80.4
.4	70.1	70.5	70.9	71.3	71.7	72.1	72.5	72.9	73.3	73.7	74.1	74.5	74.9	75.3	75.7	76.1	76.5	76.9	77.3	77.7	78.1	78.5	78.9	79.3	79.7
.5	69.5	69.9	70.3	70.7	71.1	71.5	71.9	72.3	72.7	73.1	73.5	73.9	74.3	74.7	75.1	75.5	75.9	76.3	76.7	77.0	77.4	77.8	78.2	78.6	79.0
.6	68.9	69.3	69.7	70.1	70.5	70.9	71.3	71.7	72.1	72.5	72.9	73.3	73.6	74.0	74.4	74.8	75.2	75.6	76.0	76.4	76.8	77.2	77.6	78.0	78.4
.7	68.4	68.7	69.1	69.5	69.9	70.3	70.7	71.1	71.5	71.9	72.3	72.6	73.0	73.4	73.8	74.2	74.6	75.0	75.4	75.8	76.2	76.6	76.9	77.3	77.7
.8	67.8	68.2	68.6	69.0	69.3	69.7	70.1	70.5	70.9	71.3	71.7	72.1	72.4	72.8	73.2	73.6	74.0	74.4	74.8	75.2	75.5	75.9	76.3	76.7	77.1
.9	67.2	67.6	68.0	68.4	68.8	69.2	69.5	69.9	70.3	70.7	71.1	71.5	71.8	72.2	72.6	73.0	73.4	73.8	74.2	74.5	74.9	75.3	75.7	76.1	76.5

Pol. =	**40.0**	**40.2**	**40.4**	**40.6**	**40.8**	**41.0**	**41.2**	**41.4**	**41.6**	**41.8**	**42.0**	**42.2**	**42.4**	**42.6**	**42.8**	**43.0**	**43.2**	**43.4**	**43.6**	**43.8**	**44.0**	**44.2**	**44.4**	**44.6**	**44.8**
12.0	82.9	83.3	83.7	84.2	84.6	85.0	85.4	85.8	86.2	86.6	87.1	87.5	87.9	88.3	88.7	89.1	89.5	90.0	90.4	90.8	91.2	91.6	92.0	92.4	92.9
.1	82.2	82.6	83.0	83.4	83.8	84.2	84.7	85.1	85.5	85.9	86.3	86.7	87.1	87.5	87.9	88.4	88.8	89.2	89.6	90.0	90.4	90.8	91.2	91.6	92.1
.2	81.5	81.9	82.3	82.7	83.1	83.5	83.9	84.3	84.7	85.1	85.6	86.0	86.4	86.8	87.2	87.6	88.0	88.4	88.8	89.2	89.6	90.0	90.4	90.9	91.3
.3	80.8	81.2	81.6	82.0	82.4	82.8	83.2	83.6	84.0	84.4	84.8	85.2	85.6	86.0	86.4	86.8	87.3	87.7	88.1	88.5	88.9	89.3	89.7	90.1	90.5
.4	80.1	80.5	80.9	81.3	81.7	82.1	82.5	82.9	83.3	83.7	84.1	84.5	84.9	85.3	85.7	86.1	86.5	86.9	87.3	87.7	88.1	88.5	88.9	89.3	89.7
.5	79.4	79.8	80.2	80.6	81.0	81.4	81.8	82.2	82.6	83.0	83.4	83.8	84.2	84.6	85.0	85.4	85.8	86.2	86.6	87.0	87.4	87.8	88.2	88.6	89.0
.6	78.8	79.2	79.6	79.9	80.3	80.7	81.1	81.5	81.9	82.3	82.7	83.1	83.5	83.9	84.3	84.7	85.1	85.5	85.9	86.3	86.6	87.0	87.4	87.8	88.2
.7	78.1	78.5	78.9	79.3	79.7	80.1	80.5	80.9	81.2	81.6	82.0	82.4	82.8	83.2	83.6	84.0	84.4	84.8	85.1	85.5	85.9	86.3	86.7	87.1	87.5
.8	77.5	77.9	78.3	78.6	79.0	79.4	79.8	80.2	80.6	81.0	81.3	81.7	82.1	82.5	82.9	83.3	83.7	84.1	84.4	84.8	85.2	85.6	86.0	86.4	86.6
.9	76.8	77.2	77.6	78.0	78.4	78.8	79.1	79.5	79.9	80.3	80.7	81.1	81.5	81.8	82.2	82.6	83.0	83.4	83.8	84.1	84.5	84.9	85.3	85.7	86.1

TABLE 41 (*Continued*)

EXPANDED HORNE'S TABLE OF COEFFICIENTS OF PURITY

Pol. =	**15.0**	**15.2**	**15.4**	**15.6**	**15.8**	**16.0**	**16.2**	**16.4**	**16.6**	**16.8**	**17.0**	**17.2**	**17.4**	**17.6**	**17.8**	**18.0**	**18.2**	**18.4**	**18.6**	**18.8**	**19.0**	**19.2**	**19.4**	**19.6**	**19.8**
13.0	28.6	29.0	29.3	29.7	30.1	30.5	30.9	31.3	31.6	32.0	32.4	32.8	33.2	33.5	33.9	34.3	34.7	35.1	35.4	35.8	36.2	36.6	37.0	37.3	37.7
.1	28.4	28.7	29.1	29.5	29.9	30.2	30.6	31.0	31.4	31.8	32.1	32.5	32.9	33.3	33.6	34.0	34.4	34.8	35.2	35.5	35.9	36.3	36.7	37.0	37.4
.2	28.1	28.5	28.9	29.3	29.6	30.0	30.4	30.8	31.1	31.5	31.9	32.3	32.6	33.0	33.4	33.8	34.1	34.5	34.9	35.3	35.6	36.0	36.4	36.8	37.1
.3	27.9	28.3	28.6	29.0	29.4	29.8	30.1	30.5	30.9	31.3	31.6	32.0	32.4	32.7	33.1	33.5	33.9	34.2	34.6	35.0	35.3	35.7	36.1	36.5	36.8
.4	27.7	28.1	28.4	28.8	20.2	29.5	29.9	30.3	30.6	31.0	31.4	31.7	32.1	32.5	32.9	33.2	33.6	34.0	34.3	34.7	35.1	35.4	35.8	36.2	36.5
.5	27.5	27.8	28.2	28.6	28.9	29.3	29.7	30.0	30.4	30.8	31.1	31.5	31.9	32.2	32.6	33.0	33.3	33.7	34.1	34.4	34.8	35.2	35.5	35.9	36.3
.6	27.3	27.6	28.0	28.3	28.7	29.1	29.4	29.8	30.2	30.5	30.9	31.3	31.6	32.0	32.3	32.7	33.1	33.4	33.8	34.2	34.5	34.9	35.3	35.6	36.0
.7	27.0	27.4	27.8	28.1	28.5	28.8	29.2	29.6	29.9	30.3	30.7	31.0	31.4	31.7	32.1	32.5	32.8	33.2	33.5	33.9	34.3	34.6	35.0	35.3	35.7
.8	26.8	27.2	27.6	27.9	28.3	28.6	29.0	29.3	29.7	30.1	30.4	30.8	31.1	31.5	31.8	32.2	32.6	32.9	33.3	33.6	34.0	34.4	34.7	35.1	35.4
.9	26.6	27.0	27.3	27.7	28.1	28.4	28.8	29.1	29.5	29.8	30.2	30.5	30.9	31.3	31.6	32.0	32.3	32.7	33.0	33.4	33.7	34.1	34.4	34.8	35.2

Pol. =	**20.0**	**20.2**	**20.4**	**20.6**	**20.8**	**21.0**	**21.2**	**21.4**	**21.6**	**21.8**	**22.0**	**22.2**	**22.4**	**22.6**	**22.8**	**23.0**	**23.2**	**23.4**	**23.6**	**23.8**	**24.0**	**24.2**	**24.4**	**24.6**	**24.8**
13.0	38.1	38.5	38.9	39.3	39.6	40.0	40.4	40.8	41.2	41.5	41.9	42.3	42.7	43.1	43.4	43.8	44.2	44.6	45.0	45.4	45.7	46.1	46.5	46.9	47.3
.1	37.8	38.2	38.6	38.9	39.3	39.7	40.1	40.5	40.8	41.2	41.6	42.0	42.3	42.7	43.1	43.5	43.9	44.2	44.6	45.0	45.4	45.7	46.1	46.5	46.9
.2	37.5	37.9	38.3	38.6	39.0	39.4	39.8	40.1	40.5	40.9	41.3	41.6	42.0	42.4	42.8	43.1	43.5	43.9	44.3	44.6	45.0	45.4	45.8	46.1	46.5
.3	37.2	37.6	38.0	38.3	38.7	39.1	39.4	39.8	40.2	40.6	40.9	41.3	41.7	42.0	42.4	42.8	43.2	43.5	43.9	44.3	44.6	45.0	45.4	45.8	46.1
.4	36.9	37.3	37.7	38.0	38.4	38.8	39.1	39.5	39.9	40.2	40.6	41.0	41.3	41.7	42.1	42.5	42.8	43.2	43.6	43.9	44.3	44.7	45.0	45.4	45.8
.5	36.6	37.0	37.4	37.7	38.1	38.5	38.8	39.2	39.6	39.9	40.3	40.7	41.0	41.4	41.8	42.1	42.5	42.9	43.2	43.6	44.0	44.3	44.7	45.0	45.4
.6	36.3	36.7	37.1	37.4	37.8	38.2	38.5	38.9	39.2	39.6	40.0	40.3	40.7	41.1	41.4	41.8	42.2	42.5	42.9	43.2	43.6	44.0	44.3	44.7	45.1
.7	36.1	36.4	36.8	37.1	37.5	37.9	38.2	38.6	38.9	39.3	39.7	40.0	40.4	40.8	41.0	41.5	41.8	42.2	42.6	42.9	43.3	43.6	44.0	44.4	44.7
.8	35.8	36.1	36.5	36.9	37.2	37.6	37.9	38.3	38.6	39.0	39.3	39.7	40.1	40.4	40.8	41.1	41.5	41.8	42.1	42.5	42.9	43.3	43.6	44.0	44.3
.9	35.5	35.9	36.2	36.6	36.9	37.3	37.6	38.0	38.4	38.7	39.1	39.4	39.8	40.1	40.5	40.8	41.2	41.6	41.9	42.3	42.6	43.0	43.3	43.7	44.0

Pol. =	**25.0**	**25.2**	**25.4**	**25.6**	**25.8**	**26.0**	**26.2**	**26.4**	**26.6**	**26.8**	**27.0**	**27.2**	**27.4**	**27.6**	**27.8**	**28.0**	**28.2**	**28.4**	**28.6**	**28.8**	**29.0**	**29.2**	**29.4**	**29.6**	**29.8**
13.0	47.6	48.0	48.4	48.8	49.2	49.5	49.9	50.3	50.7	51.1	51.5	51.8	52.2	52.6	53.0	53.4	53.7	54.1	54.5	54.9	55.3	55.6	56.0	56.4	56.8
.1	47.3	47.6	48.0	48.4	48.8	49.1	49.5	49.9	50.3	50.7	51.0	51.4	51.7	52.2	52.6	52.9	53.3	53.7	54.1	54.4	54.8	55.2	55.6	56.0	56.3
.2	46.9	47.3	47.6	48.0	48.4	48.8	49.1	49.5	49.8	50.3	50.6	51.0	51.4	51.8	52.1	52.5	52.9	53.3	53.6	54.0	54.4	54.8	55.1	55.5	55.9
.3	46.5	46.9	47.3	47.6	47.9	48.4	48.7	49.1	49.5	49.8	50.2	50.6	51.0	51.3	51.7	52.0	52.5	52.8	53.2	53.6	53.9	54.3	54.7	55.1	55.4
.4	46.1	46.5	46.9	47.2	47.6	48.0	48.4	48.7	49.0	49.5	49.8	50.2	50.6	50.9	51.3	51.7	52.0	52.4	52.8	53.2	53.5	53.9	54.3	54.6	55.0
.5	45.8	46.1	46.5	46.9	47.2	47.6	48.0	48.3	48.7	49.1	49.4	49.8	50.2	50.5	50.9	51.3	51.6	52.0	52.4	52.7	53.1	53.5	53.8	54.2	54.6
.6	45.4	45.8	46.2	46.5	46.9	47.2	47.6	48.0	48.3	48.7	49.1	49.4	49.8	50.2	50.5	50.9	51.2	51.6	52.0	52.3	52.7	53.1	53.4	53.8	54.1
.7	45.1	45.3	45.8	46.2	46.5	46.9	47.2	47.6	48.0	48.3	48.7	49.0	49.4	49.8	50.1	50.5	50.8	51.2	51.6	51.9	52.3	52.7	53.0	53.4	53.7
.8	44.7	45.0	45.4	45.7	46.1	46.5	46.8	47.2	47.5	47.9	48.2	48.6	48.9	49.3	49.7	50.0	50.4	50.7	51.1	51.4	51.8	52.1	52.5	52.8	53.2
.9	44.4	44.7	45.1	45.5	45.8	46.2	46.5	46.9	47.2	47.6	47.9	48.3	48.7	49.0	49.4	49.7	50.1	50.4	50.8	51.1	51.5	51.9	52.2	52.6	52.9

TABLE 41 (*Continued*)

EXPANDED HORNE'S TABLE OF COEFFICIENTS OF PURITY

Pol. =	30.0	30.2	30.4	30.6	30.8	31.0	31.2	31.4	31.6	31.8	32.0	32.2	32.4	32.6	32.8	33.0	33.2	33.4	33.6	33.8	34.0	34.2	34.4	34.6	34.8
13.0	57.2	57.5	57.9	58.3	58.6	59.1	59.5	59.8	60.2	60.6	61.0	61.4	61.7	62.1	62.5	62.9	63.3	63.6	64.0	64.4	64.8	65.2	65.6	65.9	66.3
.1	56.7	57.1	57.5	57.8	58.2	58.6	59.0	59.4	59.7	60.1	60.5	60.8	61.2	61.6	62.0	62.4	62.7	63.1	63.5	63.9	64.2	64.6	65.0	65.4	65.8
.2	56.3	56.6	57.0	57.4	57.8	58.1	58.5	58.9	59.3	59.6	60.0	60.4	60.8	61.1	61.5	61.9	62.3	62.6	63.0	63.4	63.8	64.1	64.5	64.9	65.3
.3	55.8	56.2	56.6	56.9	57.3	57.7	58.0	58.4	58.8	59.2	59.5	59.9	60.3	60.6	61.0	61.4	61.8	62.1	62.5	62.9	63.3	63.6	64.0	64.4	64.7
.4	55.3	55.7	56.1	56.5	56.8	57.2	57.6	58.0	58.3	58.7	59.1	59.4	59.8	60.2	60.5	60.9	61.3	61.6	62.0	62.4	62.8	63.1	63.5	63.9	64.2
.5	54.9	55.3	55.7	56.0	56.4	56.8	57.1	57.5	57.9	58.2	58.6	59.0	59.3	59.7	60.1	60.4	60.8	61.2	61.5	61.9	62.3	62.7	63.0	63.4	63.7
.6	54.5	54.9	55.2	55.6	56.0	56.3	56.7	57.1	57.4	57.8	58.1	58.5	58.9	59.2	59.6	60.0	60.3	60.7	61.1	61.4	61.8	62.1	62.5	62.9	63.2
.7	54.1	54.5	54.8	55.2	55.5	55.9	56.3	56.6	57.0	57.3	57.7	58.1	58.4	58.8	59.1	59.5	59.9	60.2	60.6	60.9	61.3	61.7	62.0	62.4	62.7
.8	53.7	54.0	54.4	54.8	55.1	55.5	55.8	56.2	56.5	56.9	57.3	57.6	58.0	58.3	58.7	59.0	59.4	59.8	60.1	60.5	60.8	61.2	61.6	61.9	62.3
.9	53.3	53.6	54.0	54.3	54.7	55.0	55.4	55.8	56.1	56.5	56.8	57.2	57.5	57.9	58.2	58.6	59.0	59.3	59.7	60.0	60.4	60.7	61.1	61.4	61.8

Pol. =	35.0	35.2	35.4	35.6	35.8	36.0	36.2	36.4	36.6	36.8	37.0	37.2	37.4	37.6	37.8	38.0	38.2	38.4	38.6	38.8	39.0	39.2	39.4	39.6	39.8
13.0	66.7	67.1	67.5	67.8	68.2	68.6	69.0	69.3	69.7	70.1	70.5	70.9	71.3	71.7	72.0	72.4	72.8	73.2	73.6	74.0	74.3	74.7	75.1	75.5	75.8
.1	66.2	66.5	66.9	67.3	67.7	68.1	68.4	68.8	69.2	69.6	69.9	70.3	70.7	71.1	71.5	71.8	72.2	72.6	73.0	73.3	73.7	74.1	74.5	74.9	75.2
.2	65.6	66.0	66.4	66.8	67.1	67.5	67.9	68.3	68.6	69.0	69.4	69.8	70.1	70.5	70.9	71.3	71.6	72.0	72.4	72.8	73.1	73.5	73.9	74.3	74.6
.3	65.1	65.5	65.9	66.2	66.6	67.0	67.3	67.7	68.1	68.5	68.8	69.2	69.6	69.9	70.3	70.7	71.1	71.4	71.8	72.2	72.6	72.9	73.3	73.7	74.0
.4	64.6	65.0	65.3	65.7	66.1	66.4	66.8	67.2	67.6	67.9	68.3	68.7	69.0	69.4	69.8	70.1	70.5	70.9	71.2	71.6	72.0	72.4	72.7	73.1	73.5
.5	64.1	64.5	64.8	65.2	65.6	65.9	66.3	66.7	67.0	67.4	67.8	68.1	68.5	68.9	69.2	69.6	70.0	70.3	70.7	71.1	71.4	71.8	72.2	72.5	72.9
.6	63.6	64.0	64.3	64.7	65.1	65.4	65.8	66.1	66.5	66.9	67.2	67.6	68.0	68.3	68.7	69.0	69.4	69.8	70.1	70.5	70.9	71.2	71.6	72.0	72.3
.7	63.1	63.5	63.8	64.2	64.6	64.9	65.3	65.6	66.0	66.4	66.7	67.1	67.4	67.8	68.2	68.5	68.9	69.2	69.6	70.0	70.3	70.7	71.0	71.4	71.8
.8	62.6	63.0	63.3	63.7	64.1	64.4	64.8	65.1	65.5	65.8	66.2	66.6	66.9	67.3	67.6	68.0	68.4	68.7	69.1	69.4	69.8	70.1	70.5	70.9	71.2
.9	62.1	62.5	62.9	63.2	63.6	63.9	64.3	64.6	65.0	65.3	65.7	66.1	66.4	66.8	67.1	67.5	67.8	68.2	68.5	68.9	69.3	69.6	70.0	70.3	70.7

Pol. =	40.0	40.2	40.4	40.6	40.8	41.0	41.2	41.4	41.6	41.8	42.0	42.2	42.4	42.6	42.8	43.0	43.2	43.4	43.6	43.8	44.0	44.2	44.4	44.6	44.8
13.0	76.2	76.6	77.0	77.4	77.7	78.1	78.5	78.9	79.3	79.7	80.0	80.4	80.8	81.2	81.6	81.9	82.3	82.7	83.1	83.5	83.8	84.2	84.6	85.0	85.4
.1	75.6	76.0	76.4	76.7	77.1	77.5	77.9	78.3	78.6	79.0	79.4	79.8	80.1	80.5	80.9	81.3	81.7	82.0	82.4	82.8	83.2	83.6	83.9	84.3	84.7
.2	75.0	75.4	75.8	76.1	76.5	76.9	77.3	77.6	78.0	78.4	78.8	79.1	79.5	79.9	80.3	80.6	81.0	81.4	81.8	82.1	82.5	82.9	83.3	83.6	84.0
.3	74.4	74.8	75.2	75.5	75.9	76.3	76.6	77.0	77.4	77.8	78.1	78.5	78.9	79.2	79.6	80.0	80.4	80.7	81.1	81.5	81.9	82.2	82.6	83.0	83.3
.4	73.8	74.2	74.6	74.9	75.3	75.7	76.0	76.4	76.8	77.2	77.5	77.9	78.3	78.6	79.0	79.4	79.7	80.0	80.5	80.8	81.2	81.6	81.9	82.3	82.7
.5	73.3	73.6	74.0	74.4	74.7	75.1	75.4	75.8	76.2	76.5	76.9	77.3	77.6	78.0	78.4	78.7	79.1	79.5	79.8	80.2	80.6	80.9	81.3	81.7	82.0
.6	72.7	73.0	73.4	73.8	74.1	74.5	74.9	75.2	75.6	76.0	76.3	76.7	77.0	77.4	77.8	78.1	78.5	78.9	79.2	79.6	80.0	80.3	80.7	81.0	81.4
.7	72.1	72.5	72.8	73.2	73.6	73.9	74.3	74.6	75.0	75.4	75.8	76.1	76.5	76.8	77.2	77.5	77.9	78.3	78.6	79.0	79.3	79.7	80.1	80.4	80.8
.8	71.6	71.9	72.3	72.6	73.0	73.4	73.7	74.1	74.4	74.8	75.2	75.5	75.9	76.2	76.6	76.9	77.3	77.7	78.0	78.4	78.7	79.1	79.4	79.8	80.2
.9	71.0	71.4	71.8	72.1	72.4	72.8	73.2	73.5	73.9	74.2	74.6	74.9	75.3	75.6	76.0	76.4	76.7	77.1	77.4	77.8	78.1	78.5	78.8	79.2	79.6

TABLE 41 (*Continued*)

EXPANDED HORNE'S TABLE OF COEFFICIENTS OF PURITY

Pol. =	**20.0**	**20.2**	**20.4**	**20.6**	**20.8**	**21.0**	**21.2**	**21.4**	**21.6**	**21.8**	**22.0**	**22.2**	**22.4**	**22.6**	**22.8**	**23.0**	**23.2**	**23.4**	**23.6**	**23.8**	**24.0**	**24.2**	**24.4**	**24.6**	**24.8**
14.0	35.2	35.6	36.0	36.3	36.7	37.0	37.4	37.7	38.1	38.4	38.8	39.1	39.5	39.8	40.2	40.5	40.9	41.2	41.6	41.9	42.3	42.6	43.0	43.4	43.7
.1	35.0	35.3	35.7	36.0	36.4	36.7	37.1	37.4	37.8	38.1	38.5	38.8	39.2	39.5	39.9	40.2	40.6	40.9	41.3	41.6	42.0	42.3	42.7	43.0	43.4
.2	34.7	35.1	35.4	35.8	36.1	36.5	36.8	37.2	37.5	37.8	38.2	38.5	38.9	39.2	39.6	39.9	40.3	40.6	41.0	41.3	41.7	42.0	42.4	42.7	43.1
.3	34.5	34.8	35.2	35.5	35.8	36.2	36.5	36.9	37.2	37.6	37.9	38.3	38.6	38.9	39.3	39.6	40.0	40.3	40.7	41.0	41.4	41.7	42.0	42.4	42.7
.4	34.2	34.6	34.9	35.2	35.6	35.9	36.3	36.6	36.9	37.3	37.6	38.0	38.3	38.7	39.0	39.3	39.7	40.0	40.4	40.7	41.1	41.4	41.7	42.1	42.4
.5	34.0	34.3	34.6	35.0	35.3	35.7	36.0	36.3	36.7	37.0	37.4	37.7	38.0	38.4	38.7	39.1	39.4	39.7	40.1	40.4	40.8	41.1	41.4	41.8	42.1
.6	33.7	34.1	34.4	34.7	35.1	35.4	35.7	36.1	36.4	36.8	37.1	37.4	37.8	38.1	38.4	38.8	39.1	39.4	39.8	40.1	40.5	40.8	41.1	41.5	41.8
.7	33.5	33.8	34.1	34.5	34.8	35.1	35.5	35.8	36.2	36.5	36.8	37.2	37.5	37.8	38.2	38.5	38.8	39.2	39.5	39.8	40.2	40.5	40.8	41.2	41.5
.8	33.2	33.6	33.9	34.2	34.6	34.9	35.2	35.6	35.9	36.2	36.6	36.9	37.2	37.6	37.9	38.2	38.6	38.9	39.2	39.5	39.9	40.2	40.5	40.9	41.2
.9	33.0	33.3	33.6	34.0	34.3	34.6	35.0	35.3	35.6	36.0	36.3	36.6	37.0	37.3	37.6	37.9	38.3	38.6	38.9	39.3	39.6	39.9	40.3	40.6	40.9

Pol. =	**25.0**	**25.2**	**25.4**	**25.6**	**25.8**	**26.0**	**26.2**	**26.4**	**26.6**	**26.8**	**27.0**	**27.2**	**27.4**	**27.6**	**27.8**	**28.0**	**28.2**	**28.4**	**28.6**	**28.8**	**29.0**	**29.2**	**29.4**	**29.6**	**29.8**
14.0	44.1	44.4	44.8	45.1	45.5	45.8	46.2	46.5	46.9	47.2	47.6	47.9	48.3	48.6	49.0	49.3	49.7	50.0	50.4	50.8	51.1	51.5	51.8	52.2	52.5
.1	43.7	44.1	44.4	44.8	45.1	45.5	45.8	46.2	46.5	46.9	47.2	47.6	47.9	48.3	48.6	49.0	49.3	49.7	50.0	50.4	50.7	51.1	51.4	51.8	52.1
.2	43.4	43.7	44.1	44.4	44.8	45.1	45.5	45.8	46.2	46.5	46.9	47.2	47.6	47.9	48.3	48.6	49.0	49.3	49.7	50.0	50.3	50.7	51.0	51.4	51.7
.3	43.1	43.4	43.8	44.1	44.5	44.8	45.2	45.5	45.8	46.2	46.5	46.9	47.2	47.6	47.9	48.3	48.6	48.9	49.3	49.6	50.0	50.3	50.7	51.0	51.4
.4	42.8	43.1	43.4	43.8	44.1	44.5	44.8	45.2	45.5	45.8	46.2	46.5	46.9	47.2	47.6	47.9	48.2	48.6	48.9	49.3	49.6	49.9	50.3	50.6	51.0
.5	42.5	42.8	43.1	43.5	43.8	44.2	44.5	44.8	45.2	45.5	45.8	46.2	46.5	46.9	47.2	47.5	47.9	48.2	48.6	48.9	49.2	49.6	49.9	50.3	50.6
.6	42.1	42.5	42.8	43.2	43.5	43.8	44.2	44.5	44.8	45.2	45.5	45.9	46.2	46.5	46.9	47.2	47.5	47.9	48.2	48.6	48.9	49.2	49.6	49.9	50.2
.7	41.8	42.2	42.5	42.8	43.2	43.5	43.9	44.2	44.5	44.9	45.2	45.5	45.9	46.2	46.5	46.9	47.2	47.5	47.9	48.2	48.5	48.9	49.2	49.5	49.9
.8	41.5	41.9	42.2	42.5	42.9	43.2	43.5	43.9	44.2	44.5	44.9	45.2	45.5	45.9	46.2	46.5	46.9	47.2	47.5	47.9	48.2	48.5	48.9	49.2	49.5
.9	41.2	41.6	41.9	42.2	42.6	42.9	43.2	43.6	43.9	44.2	44.5	44.9	45.2	45.5	45.9	46.2	46.5	46.9	47.2	47.5	47.8	48.2	48.5	48.8	49.2

Pol. =	**30.0**	**30.2**	**30.4**	**30.6**	**30.8**	**31.0**	**31.2**	**31.4**	**31.6**	**31.8**	**32.0**	**32.2**	**32.4**	**32.6**	**32.8**	**33.0**	**33.2**	**33.4**	**33.6**	**33.8**	**34.0**	**34.2**	**34.4**	**34.6**	**34.8**
14.0	52.9	53.2	53.6	53.9	54.3	54.6	55.0	55.3	55.7	56.0	56.4	56.7	57.1	57.5	57.8	58.2	58.5	58.9	59.2	59.6	59.9	60.3	60.6	61.0	61.3
.1	52.5	52.8	53.2	53.5	53.9	54.2	54.6	54.9	55.3	55.6	56.0	56.2	56.7	57.0	57.4	57.7	58.1	58.4	58.8	59.1	59.5	59.8	60.2	60.5	60.9
.2	52.1	52.4	52.8	53.1	53.5	53.8	54.2	54.5	54.9	55.2	55.6	55.9	56.2	56.6	56.9	57.3	57.6	58.0	58.3	58.7	59.0	59.4	59.7	60.1	60.4
.3	51.7	52.0	52.4	52.7	53.1	53.4	53.8	54.1	54.5	54.8	55.1	55.5	55.8	56.2	56.5	56.9	57.2	57.6	57.9	58.2	58.6	58.9	59.3	59.6	60.0
.4	51.3	51.7	52.0	52.3	52.7	53.0	53.4	53.7	54.1	54.4	54.7	55.1	55.4	55.8	56.1	56.4	56.8	57.1	57.5	57.8	58.2	58.5	58.8	59.2	59.5
.5	50.9	51.3	51.6	52.0	52.3	52.6	53.0	53.3	53.7	54.0	54.3	54.7	55.0	55.4	55.7	56.0	56.4	56.7	57.1	57.4	57.7	58.1	58.4	58.8	59.1
.6	50.6	50.9	51.2	51.6	51.9	52.3	52.6	52.9	53.3	53.6	53.9	54.3	54.6	55.0	55.3	55.6	56.0	56.3	56.7	57.0	57.3	57.7	58.0	58.3	58.7
.7	50.2	50.5	50.9	51.2	51.5	51.9	52.2	52.6	52.9	53.2	53.6	53.9	54.2	54.6	54.9	55.2	55.6	55.9	56.2	56.6	56.9	57.2	57.6	57.9	58.2
.8	49.9	50.2	50.5	50.8	51.2	51.5	51.8	52.2	52.5	52.8	53.2	53.5	53.8	54.2	54.5	54.8	55.2	55.5	55.8	56.2	56.5	56.8	57.2	57.5	57.8
.9	49.5	49.8	50.2	50.5	50.8	51.1	51.5	51.8	52.1	52.5	52.8	53.1	53.5	53.8	54.1	54.4	54.8	55.1	55.4	55.8	56.1	56.4	56.8	57.1	57.4

TABLE 41 (*Continued*)

EXPANDED HORNE'S TABLE OF COEFFICIENTS OF PURITY

Pol. =	**35.0**	**35.2**	**35.4**	**35.6**	**35.8**	**36.0**	**36.2**	**36.4**	**36.6**	**36.8**	**37.0**	**37.2**	**37.4**	**37.6**	**37.8**	**38.0**	**38.2**	**38.4**	**38.6**	**38.8**	**39.0**	**39.2**	**39.4**	**39.6**	**39.8**
14.0	61.7	62.0	62.4	62.7	63.1	63.4	63.8	64.1	64.5	64.9	65.2	65.6	65.9	66.3	66.6	67.0	67.3	67.7	68.0	68.4	68.7	69.1	69.4	69.8	70.1
.1	61.2	61.5	61.9	62.2	62.6	62.9	63.3	63.6	64.0	64.3	64.7	65.0	65.4	65.7	66.1	66.4	66.8	67.1	67.5	67.8	68.2	68.5	68.9	69.2	69.6
.2	60.8	61.1	61.5	61.8	62.2	62.5	62.8	63.2	63.5	63.9	64.2	64.6	64.9	65.3	65.6	66.0	66.3	66.7	67.0	67.4	67.7	68.1	68.4	68.7	69.1
.3	60.3	60.7	61.0	61.3	61.7	62.0	62.4	62.7	63.1	63.4	63.8	64.1	64.5	64.8	65.1	65.5	65.8	66.2	66.5	66.9	67.2	67.6	67.9	68.2	68.6
.4	59.9	60.2	60.6	60.9	61.2	61.6	61.9	62.3	62.6	63.0	63.3	63.6	64.0	64.3	64.7	65.0	65.3	65.7	66.0	66.4	66.7	67.1	67.4	67.7	68.1
.5	59.4	59.8	60.1	60.5	60.8	61.1	61.5	61.8	62.2	62.5	62.8	63.2	63.5	63.8	64.2	64.5	54.9	65.2	65.5	65.9	66.2	66.6	66.9	67.2	67.6
.6	59.0	59.3	59.7	60.0	60.4	60.7	61.0	61.4	61.7	62.0	62.4	62.7	63.0	63.4	63.7	64.1	64.4	64.7	65.1	65.4	65.7	66.1	66.4	66.8	67.1
.7	58.6	58.9	59.2	59.6	59.9	60.3	60.6	60.9	61.3	61.6	61.9	62.3	62.6	62.9	63.3	63.6	63.9	64.3	64.6	64.9	65.3	65.6	65.9	66.3	66.6
.8	58.2	58.5	58.8	59.2	59.5	59.8	60.2	60.5	60.8	61.2	61.5	61.8	62.1	62.5	62.8	63.1	63.5	63.8	64.1	64.5	64.8	65.1	65.5	65.8	66.1
.9	57.7	58.1	58.4	58.7	59.1	59.4	59.7	60.1	60.4	60.7	61.0	61.4	61.7	62.0	62.4	62.7	63.0	63.4	63.7	64.0	64.3	64.7	65.0	65.3	65.7

Pol. =	**40.0**	**40.2**	**40.4**	**40.6**	**40.8**	**41.0**	**41.2**	**41.4**	**41.6**	**41.8**	**42.0**	**42.2**	**42.4**	**42.6**	**42.8**	**43.0**	**43.2**	**43.4**	**43.6**	**43.8**	**44.0**	**44.2**	**44.4**	**44.6**	**44.8**
14.0	70.5	70.8	71.2	71.5	71.9	72.3	72.6	73.0	73.3	73.7	74.0	74.4	74.7	75.1	75.4	75.8	76.1	76.5	76.8	77.2	77.5	77.9	78.2	78.6	79.0
.1	69.9	70.3	70.6	71.0	71.3	71.7	72.0	72.4	72.7	73.1	73.4	73.8	74.1	74.5	74.8	75.2	75.5	75.9	76.2	76.6	76.9	77.3	77.6	78.0	78.3
.2	69.4	69.8	70.1	70.5	70.8	71.2	71.5	71.9	72.2	72.6	72.9	73.3	73.6	74.0	74.3	74.7	75.0	75.3	75.7	76.0	76.4	76.7	77.1	77.4	77.8
.3	68.9	69.3	69.6	70.0	70.3	70.7	71.0	71.3	71.7	72.0	72.3	72.7	73.1	73.4	73.8	74.1	74.4	74.8	75.1	75.5	75.8	76.2	76.5	76.9	77.2
.4	68.4	68.8	69.1	69.5	69.8	70.1	70.5	70.8	71.2	71.5	71.8	72.2	72.5	72.9	73.2	73.6	73.9	74.2	74.6	74.9	75.3	75.6	76.0	76.3	76.6
.5	67.9	68.3	68.6	68.9	69.3	69.6	70.0	70.3	70.6	71.0	71.3	71.7	72.0	72.3	72.7	73.0	73.4	73.7	74.0	74.4	74.7	75.1	75.4	75.7	76.1
.6	67.4	67.8	68.1	68.4	68.8	69.1	69.5	69.8	70.1	70.5	70.8	71.1	71.5	71.8	72.2	72.5	72.8	73.2	73.5	73.8	74.2	74.5	74.8	75.2	75.5
.7	66.9	67.3	67.6	68.0	68.3	68.6	69.0	69.3	69.6	70.0	70.3	70.6	71.0	71.3	71.6	72.0	72.3	72.6	73.0	73.3	73.6	74.0	74.3	74.6	75.0
.8	66.5	66.8	67.1	67.5	67.8	68.1	68.5	68.8	69.1	69.5	69.8	70.1	70.5	70.8	71.1	71.5	71.8	72.1	72.5	72.8	73.1	73.4	73.8	74.1	74.4
.9	66.0	66.3	66.7	67.0	67.3	67.6	68.0	68.3	68.6	69.0	69.3	69.6	70.0	70.3	70.6	70.9	71.3	71.6	71.9	72.3	72.6	72.9	73.3	73.6	73.9

Pol. =	**45.0**	**45.2**	**45.4**	**45.6**	**45.8**	**46.0**	**46.2**	**46.4**	**46.6**	**46.8**	**47.0**	**47.2**	**47.4**	**47.6**	**47.8**	**48.0**	**48.2**	**48.4**	**48.6**	**48.8**	**49.0**	**49.2**	**49.4**	**49.6**	**49.8**
14.0	79.3	79.7	80.0	80.4	80.7	81.1	81.4	81.8	82.1	82.5	82.8	83.2	83.5	83.9	84.2	84.6	84.9	85.3	85.6	86.0	86.4	86.7	87.1	87.4	87.8
.1	78.7	79.0	79.4	79.7	80.1	80.4	80.8	81.1	81.5	81.8	82.2	82.5	82.9	83.2	83.6	83.9	84.3	84.6	85.0	85.3	85.7	86.0	86.4	86.7	87.1
.2	78.1	78.5	78.8	79.2	79.5	79.9	80.2	80.6	80.9	81.2	81.6	81.9	82.3	82.6	83.0	83.3	83.7	84.0	84.4	84.7	85.1	85.4	85.8	86.1	86.5
.3	77.5	77.8	78.2	78.6	78.9	79.3	79.6	80.0	80.3	80.7	81.0	81.3	81.7	82.0	82.4	82.7	83.1	83.4	83.8	84.1	84.4	84.8	85.1	85.5	85.8
.4	77.0	77.3	77.7	78.0	78.3	78.7	79.0	79.4	79.7	80.1	80.4	80.7	81.1	81.4	81.8	82.1	82.5	82.8	83.1	83.5	83.8	84.2	84.5	84.8	85.2
.5	76.4	76.8	77.1	77.4	77.8	78.1	78.5	78.8	79.1	79.5	79.8	80.2	80.5	80.8	81.2	81.5	81.8	82.2	82.5	82.9	83.2	83.5	83.9	84.2	84.6
.6	75.9	76.2	76.5	76.9	77.2	77.5	77.9	78.2	78.6	78.9	79.2	79.6	79.9	80.2	80.6	80.9	81.3	81.6	81.9	82.3	82.6	82.9	83.3	83.6	84.0
.7	75.3	75.7	76.0	76.3	76.7	77.0	77.3	77.7	78.0	78.3	78.7	79.0	79.3	79.7	80.0	80.3	80.6	81.0	81.3	81.7	82.0	82.3	82.7	83.0	83.4
.8	74.8	75.1	75.4	75.8	76.1	76.4	76.8	77.1	77.4	77.8	78.1	78.4	78.8	79.1	79.4	79.8	80.1	80.4	80.8	81.1	81.4	81.8	82.1	82.4	82.8
.9	74.2	74.6	74.9	75.2	75.6	75.9	76.2	76.6	76.9	77.2	77.5	77.9	78.2	78.5	78.9	79.2	79.5	79.9	80.2	80.5	80.8	81.2	81.5	81.8	82.2

TABLE 41 (*Continued*)

EXPANDED HORNE'S TABLE OF COEFFICIENTS OF PURITY

Pol. =	**30.0**	**30.2**	**30.4**	**30.6**	**30.8**	**31.0**	**31.2**	**31.4**	**31.6**	**31.8**	**32.0**	**32.2**	**32.4**	**32.6**	**32.8**	**33.0**	**33.2**	**33.4**	**33.6**	**33.8**	**34.0**	**34.2**	**34.4**	**34.6**	**34.8**
15.0	49.1	49.5	49.8	50.1	50.5	50.8	51.1	51.4	51.8	52.1	52.4	52.8	53.1	53.4	53.7	54.1	54.4	54.7	55.0	55.4	55.7	56.0	56.4	56.7	57.0
.1	48.8	49.1	49.5	49.8	50.0	50.4	50.8	51.1	51.4	51.7	52.1	52.4	52.7	53.0	53.4	53.7	54.0	54.3	54.7	55.0	55.3	55.6	56.0	56.3	56.6
.2	48.5	48.8	49.1	49.4	49.8	50.1	50.4	50.7	51.0	51.4	51.7	52.0	52.3	52.7	53.0	53.3	53.6	54.0	54.3	54.6	54.9	55.2	55.6	55.9	56.2
.3	48.1	48.4	48.8	49.1	49.4	49.7	50.1	50.4	50.7	51.0	51.3	51.7	52.0	52.3	52.6	52.9	53.3	53.6	53.9	54.2	54.5	54.9	55.2	55.5	55.8
.4	47.8	48.1	48.4	48.7	49.1	49.4	49.7	50.0	50.2	50.7	51.0	51.3	51.6	51.9	52.3	52.6	52.9	53.2	53.5	53.8	54.2	54.5	54.8	55.1	55.4
.5	47.5	47.8	48.1	48.4	48.7	49.0	49.4	49.7	50.0	50.3	50.6	50.9	51.2	51.6	51.9	52.2	52.5	52.8	53.2	53.5	53.8	54.1	54.4	54.7	55.1
.6	47.1	47.5	47.8	48.1	48.4	48.7	49.0	49.3	49.7	50.0	50.3	50.6	50.9	51.2	51.5	51.9	52.2	52.5	52.8	53.1	53.4	53.7	54.1	54.4	54.7
.7	46.8	47.1	47.4	47.8	48.1	48.4	48.7	49.0	49.3	49.6	49.9	50.3	50.6	50.9	51.2	51.5	51.8	52.1	52.4	52.8	53.1	53.4	53.7	54.0	54.3
.8	46.5	46.8	47.1	47.4	47.7	48.1	48.4	48.7	49.0	49.3	49.6	49.9	50.2	50.5	50.8	51.2	51.5	51.8	52.1	52.4	52.7	53.0	53.3	53.6	54.0
.9	46.2	46.5	46.8	47.1	47.4	47.7	48.0	48.4	48.7	49.0	49.3	49.6	49.9	50.2	50.5	50.8	51.1	51.4	51.7	52.0	52.4	52.7	53.0	53.3	53.6

Pol. =	**35.0**	**35.2**	**35.4**	**35.6**	**35.8**	**36.0**	**36.2**	**36.4**	**36.6**	**36.8**	**37.0**	**37.2**	**37.4**	**37.6**	**37.8**	**38.0**	**38.2**	**38.4**	**38.6**	**38.8**	**39.0**	**39.2**	**39.4**	**39.6**	**39.8**
15.0	57.3	57.7	58.0	58.3	58.6	59.0	59.3	59.6	60.0	60.3	60.6	60.9	61.3	61.6	61.9	62.3	62.6	62.9	63.2	63.6	63.9	64.2	64.5	64.9	65.2
.1	56.9	57.3	57.6	57.9	58.2	58.6	58.9	59.2	59.5	59.9	60.2	60.5	60.8	61.2	61.5	61.8	62.1	62.5	62.8	63.1	63.4	63.8	64.1	64.4	64.7
.2	56.5	56.9	57.2	57.5	57.8	58.2	58.5	58.8	59.1	59.4	59.8	60.1	60.4	60.7	61.1	61.4	61.[illegible]	62.0	62.4	62.7	63.0	63.3	63.6	64.0	64.3
.3	56.1	56.5	56.8	57.1	57.4	57.8	58.1	58.4	58.7	59.0	59.4	59.7	60.0	60.3	60.6	61.0	61.3	61.6	61.9	62.2	62.6	62.9	63.2	63.5	63.8
.4	55.8	56.1	56.4	56.7	57.0	57.4	57.7	58.0	58.3	58.6	58.9	59.3	59.6	59.9	60.2	60.5	60.9	61.2	61.5	61.8	62.1	62.4	62.8	63.1	63.4
.5	55.4	55.7	56.0	56.3	56.6	57.0	57.3	57.6	57.9	58.2	58.5	58.9	59.2	59.5	59.8	60.1	60.4	60.8	61.1	61.4	61.7	62.0	62.3	62.7	63.0
.6	55.0	55.3	55.6	55.9	56.3	56.6	56.9	57.2	57.5	57.8	58.1	58.5	58.8	59.1	59.4	59.7	60.0	60.3	60.7	61.0	61.3	61.6	61.9	62.2	62.5
.7	54.6	54.9	55.3	55.6	55.9	56.2	56.5	56.8	57.1	57.4	57.7	58.1	58.4	58.7	59.0	59.3	59.6	59.9	60.2	60.6	60.9	61.3	61.5	61.8	62.1
.8	54.3	54.6	54.9	55.2	55.5	55.8	56.1	56.4	56.7	57.1	57.4	57.7	58.0	58.3	58.6	58.9	59.2	59.5	59.8	60.2	60.5	60.8	61.1	61.4	61.7
.9	53.9	54.2	54.5	54.8	55.1	55.4	55.7	56.1	56.4	56.7	57.0	57.3	57.6	57.9	58.2	58.5	58.8	59.1	59.4	59.7	60.1	60.4	60.7	61.0	61.3

Pol. =	**40.0**	**40.2**	**40.4**	**40.6**	**40.8**	**41.0**	**41.2**	**41.4**	**41.6**	**41.8**	**42.0**	**42.2**	**42.4**	**42.6**	**42.8**	**43.0**	**43.2**	**43.4**	**43.6**	**43.8**	**44.0**	**44.2**	**44.4**	**44.6**	**44.8**
15.0	65.5	65.9	66.2	66.5	66.8	67.2	67.5	67.8	68.1	68.5	68.8	69.1	69.5	69.8	70.1	70.4	70.8	71.1	71.4	71.8	72.1	72.4	72.7	73.1	73.4
.1	65.1	65.4	65.7	66.0	66.4	66.7	67.0	67.3	67.7	68.0	68.3	68.6	69.0	69.3	69.6	69.9	70.3	70.6	70.9	71.2	71.6	71.9	72.2	72.6	72.9
.2	64.6	64.9	65.3	65.6	65.9	66.2	66.6	66.9	67.2	67.5	67.8	68.2	68.5	68.8	69.1	69.5	69.8	70.1	70.4	70.8	71.1	71.4	71.7	72.0	72.4
.3	64.2	64.5	64.8	65.1	65.5	65.8	66.1	66.4	66.7	67.1	67.4	67.7	68.0	68.3	68.7	69.0	69.3	69.6	69.9	70.2	70.6	70.9	71.2	71.5	71.9
.4	63.9	64.0	64.4	64.7	65.0	65.3	65.6	66.0	66.3	66.6	66.9	67.2	67.5	67.9	68.2	68.5	68.8	69.1	69.5	69.8	70.1	70.4	70.7	71.1	71.4
.5	63.3	63.6	63.9	64.2	64.6	64.9	65.2	65.5	65.8	66.1	66.5	66.8	67.1	67.4	67.7	68.0	68.4	68.7	69.0	69.3	69.6	69.9	70.2	70.6	70.9
.6	62.9	63.2	63.5	63.8	64.1	64.4	64.7	65.1	65.4	65.7	66.0	66.3	66.6	66.9	67.3	67.6	67.9	68.2	68.5	68.8	69.1	69.6	69.8	70.1	70.4
.7	62.4	62.7	63.1	63.4	63.7	64.0	64.3	64.6	64.9	65.2	65.6	65.9	66.2	66.5	66.8	67.1	67.4	67.7	68.1	68.4	68.7	69.0	69.3	69.6	69.9
.8	62.0	62.3	62.6	62.9	63.3	63.6	63.9	64.2	64.5	64.8	65.1	65.4	65.7	66.0	66.4	66.7	67.0	67.3	67.6	67.9	68.2	68.5	68.8	69.1	69.5
.9	61.6	61.9	62.2	62.5	62.8	63.1	63.4	63.8	64.1	64.4	64.7	65.0	65.3	65.6	65.9	66.2	66.5	66.8	67.1	67.4	67.8	68.1	68.4	68.7	69.0

TABLE 41 (*Continued*)

EXPANDED HORNE'S TABLE OF COEFFICIENTS OF PURITY

Pol. =	**45.0**	**45.2**	**45.4**	**45.6**	**45.8**	**46.0**	**46.2**	**46.4**	**46.6**	**46.8**	**47.0**	**47.2**	**47.4**	**47.6**	**47.8**	**48.0**	**48.2**	**48.4**	**48.6**	**48.8**	**49.0**	**49.2**	**49.4**	**49.6**	**49.8**
15.0	73.7	74.0	74.4	74.7	75.0	75.4	75.7	76.0	76.3	76.7	77.0	77.3	77.7	78.0	78.3	78.6	79.0	79.3	79.6	79.9	80.3	80.6	80.9	81.3	81.6
.1	73.2	73.5	73.9	74.2	74.5	74.8	75.2	75.5	75.8	76.1	76.5	76.8	77.1	77.4	77.8	78.1	78.4	78.7	79.1	79.4	79.7	80.0	80.4	80.7	81.0
.2	72.7	73.0	73.3	73.7	74.0	74.3	74.6	75.0	75.3	75.6	75.9	76.2	76.6	76.9	77.2	77.5	77.9	78.2	78.5	78.8	79.2	79.5	79.8	80.1	80.4
.3	72.2	72.5	72.8	73.2	73.5	73.8	74.1	74.4	74.8	75.1	75.4	75.7	76.0	76.4	76.7	77.0	77.3	77.6	78.0	78.3	78.6	78.9	79.2	79.6	79.9
.4	71.7	72.0	72.3	72.6	73.0	73.3	73.6	73.9	74.2	74.6	74.9	75.2	75.5	75.8	76.2	76.5	76.8	77.1	77.4	77.7	78.1	78.4	78.7	79.0	79.3
.5	71.2	71.5	71.8	72.1	72.5	72.8	73.1	73.4	73.7	74.0	74.4	74.7	75.0	75.3	75.6	75.9	76.3	76.6	76.9	77.2	77.5	77.8	78.2	78.5	78.8
.6	70.7	71.0	71.2	71.6	72.0	72.3	72.6	72.9	73.2	73.5	73.9	74.2	74.5	74.8	75.1	75.4	75.8	76.1	76.4	76.7	77.0	77.3	77.6	77.9	78.3
.7	70.2	70.5	70.9	71.2	71.5	71.8	72.1	72.4	72.7	73.0	73.4	73.7	74.0	74.3	74.6	74.9	75.2	75.5	75.9	76.2	76.5	76.8	77.1	77.4	77.7
.8	69.8	70.1	70.4	70.7	71.0	71.3	71.6	71.9	72.2	72.6	72.9	73.2	73.5	73.8	74.1	74.4	74.7	75.0	75.3	75.7	76.0	76.3	76.6	76.9	77.2
.9	69.3	69.6	69.9	70.2	70.5	70.8	71.1	71.5	71.8	72.1	72.4	72.7	73.0	73.3	73.6	73.9	74.2	74.5	74.8	75.1	75.5	75.8	76.1	76.4	76.7

Pol. =	**50.0**	**50.2**	**50.4**	**50.6**	**50.8**	**51.0**	**51.2**	**51.4**	**51.6**	**51.8**	**52.0**	**52.2**	**52.4**	**52.6**	**52.8**	**53.0**	**53.2**	**53.4**	**53.6**	**53.8**	**54.0**	**54.2**	**54.4**	**54.6**	**54.8**
15.0	81.9	82.2	82.6	82.9	83.2	83.5	83.9	84.2	84.5	84.9	85.2	85.5	85.8	86.2	86.5	86.8	87.2	87.5	87.8	88.1	88.5	88.8	89.1	89.4	89.8
.1	81.3	81.7	82.0	82.3	82.6	83.0	83.3	83.7	83.9	84.3	84.6	84.9	85.2	85.6	85.9	86.2	86.5	86.9	87.2	87.5	87.8	88.2	88.5	88.8	89.1
.2	80.8	81.1	81.4	81.7	82.1	82.4	82.7	83.0	83.4	83.7	84.0	84.3	84.6	85.0	85.3	85.6	85.9	86.3	86.6	86.9	87.2	87.6	87.9	88.2	88.5
.3	80.2	80.5	80.9	81.2	81.5	81.8	82.1	82.5	82.8	83.1	83.4	83.7	84.1	84.4	84.7	85.0	85.3	85.7	86.0	86.3	86.6	86.9	87.3	87.6	87.9
.4	79.7	80.0	80.3	80.6	80.9	81.2	81.6	81.9	82.2	82.5	82.8	83.2	83.5	83.8	84.1	84.4	84.8	85.1	85.4	85.7	86.0	86.3	86.7	87.0	87.3
.5	79.1	79.4	79.7	80.1	80.4	80.7	81.0	81.3	81.6	82.0	82.2	82.6	82.9	83.2	83.5	83.9	84.2	84.5	84.8	85.1	85.4	85.8	86.1	86.4	86.7
.6	78.6	78.9	79.2	79.5	79.8	80.1	80.5	80.8	81.1	81.4	81.7	82.0	82.3	82.7	83.0	83.3	83.6	83.9	84.2	84.5	84.9	85.2	85.5	85.8	86.1
.7	78.0	78.4	78.7	79.0	79.3	79.6	79.9	80.2	80.5	80.8	81.2	81.5	81.8	82.1	82.4	82.7	83.0	83.3	83.6	84.0	84.3	84.6	84.9	85.2	85.5
.8	77.5	77.8	78.1	78.4	78.8	79.1	79.4	79.7	80.0	80.3	80.6	80.9	81.2	81.5	81.9	82.2	82.5	82.8	83.1	83.4	83.7	84.0	84.3	84.6	85.0
.9	77.0	77.3	77.6	77.9	78.2	78.5	78.8	79.2	79.5	79.8	80.1	80.4	80.7	81.0	81.3	81.6	81.9	82.2	82.5	82.8	83.2	83.5	83.8	84.1	84.4

Pol. =	**55.0**	**55.2**	**55.4**	**55.6**	**55.8**	**56.0**	**56.2**	**56.4**	**56.6**	**56.8**	**57.0**	**57.2**	**57.4**	**57.6**	**57.8**	**58.0**	**58.2**	**58.4**	**58.6**	**58.8**	**59.0**	**59.2**	**59.4**	**59.6**	**59.8**
15.0	90.1	90.4	90.8	91.1	91.4	91.7	92.1	92.4	92.7	93.0	93.4	93.7	94.0	94.4	94.7	95.0	95.3	95.7	96.0	96.3	96.7	97.0	97.3	97.6	98.0
.1	89.5	89.8	90.1	90.4	90.8	91.1	91.4	91.7	92.1	92.4	92.7	93.0	93.4	93.7	94.0	94.3	94.7	95.0	95.3	95.6	96.0	96.3	96.6	97.0	97.3
.2	88.8	89.2	89.5	89.8	90.1	90.5	90.8	91.1	91.4	91.8	92.1	92.4	92.7	93.0	93.4	93.7	94.0	94.3	94.7	95.0	95.3	95.6	96.0	96.3	96.6
.3	88.2	88.6	88.9	89.2	89.5	89.8	90.2	90.5	90.8	91.1	91.4	91.8	92.1	92.4	92.7	93.0	93.4	93.7	94.0	94.3	94.6	95.0	95.3	95.6	95.9
.4	87.6	87.9	88.3	88.6	88.9	89.2	89.5	89.9	90.2	90.5	90.8	91.1	91.4	91.8	92.1	92.4	92.7	93.0	93.4	93.7	94.0	94.3	94.6	94.9	95.3
.5	87.0	87.3	87.7	88.0	88.3	88.6	88.9	89.2	89.6	89.9	90.2	90.5	90.8	91.1	91.5	91.8	92.1	92.4	92.7	93.0	93.3	93.7	94.0	94.3	94.6
.6	86.4	86.7	87.1	87.4	87.7	88.0	88.3	88.6	88.9	89.3	89.6	89.9	90.2	90.5	90.8	91.1	91.5	91.8	92.1	92.4	92.7	93.0	93.3	93.7	94.0
.7	85.8	86.2	86.5	86.8	87.1	87.4	87.7	88.0	88.3	88.7	89.0	89.3	89.6	89.9	90.2	90.5	90.8	91.2	91.5	91.8	92.1	92.4	92.7	93.0	93.3
.8	85.3	85.6	85.9	86.2	86.5	86.8	87.1	87.4	87.7	88.1	88.4	88.7	89.0	89.3	89.6	89.9	90.2	90.5	90.8	91.2	91.5	91.8	92.1	92.4	92.7
.9	84.7	85.0	85.3	85.6	85.9	86.2	86.5	86.9	87.2	87.5	87.8	88.1	88.4	88.7	89.0	89.3	89.6	89.9	90.2	90.5	90.9	91.2	91.5	91.8	92.1

TABLE 41 (*Continued*)

EXPANDED HORNE'S TABLE OF COEFFICIENTS OF PURITY

Pol. =	**35.0**	**35.2**	**35.4**	**35.6**	**35.8**	**36.0**	**36.2**	**36.4**	**36.6**	**36.8**	**37.0**	**37.2**	**37.4**	**37.6**	**37.8**	**38.0**	**38.2**	**38.4**	**38.6**	**38.8**	**39.0**	**39.2**	**39.4**	**39.6**	**39.8**
16.0	53.5	53.8	54.1	54.5	54.8	55.1	55.4	55.7	56.0	56.3	56.6	56.9	57.2	57.5	57.8	58.1	58.4	58.7	59.0	59.3	59.7	60.0	60.3	60.6	60.9
.1	53.2	53.5	53.8	54.1	54.4	54.7	55.0	55.3	55.6	55.9	56.2	56.5	56.8	57.1	57.4	57.7	58.0	58.3	58.7	59.0	59.3	59.6	59.9	60.2	60.5
.2	52.8	53.1	53.4	53.7	54.0	54.3	54.6	54.9	55.2	55.5	55.9	56.2	56.5	56.8	57.1	57.4	57.7	58.0	58.3	58.6	58.9	59.2	59.5	59.8	60.1
.3	52.5	52.8	53.1	53.4	53.7	54.0	54.3	54.6	54.9	55.2	55.5	55.8	56.1	56.4	56.6	57.0	57.3	57.6	57.9	58.2	58.5	58.8	59.1	59.4	59.7
.4	52.1	52.4	52.7	53.0	53.3	53.6	53.9	54.2	54.5	54.8	55.1	55.4	55.7	56.0	56.3	56.6	56.9	57.2	57.5	57.8	58.1	58.4	58.7	59.0	59.3
.5	51.8	52.1	52.4	52.7	53.0	53.3	53.6	53.9	54.2	54.5	54.8	55.1	55.4	55.7	56.0	56.3	56.5	56.8	57.1	57.4	57.7	58.0	58.3	58.6	58.9
.6	51.5	51.8	52.1	52.4	52.7	52.9	53.2	53.5	53.8	54.1	54.4	54.7	55.0	55.3	55.6	55.9	56.2	56.5	56.8	57.1	57.4	57.7	57.9	58.2	58.5
.7	51.1	51.4	51.7	52.0	52.3	52.6	52.9	53.2	53.5	53.8	54.1	54.4	54.7	54.9	55.2	55.5	55.8	56.1	56.4	56.7	57.0	57.3	57.6	57.9	58.2
.8	50.8	51.1	51.4	51.7	52.0	52.3	52.6	52.9	53.1	53.4	53.7	54.0	54.3	54.6	54.9	55.2	55.5	55.8	56.1	56.3	56.6	56.9	57.2	57.5	57.8
.9	50.5	50.8	51.1	51.4	51.7	51.9	52.2	52.5	52.8	53.1	53.4	53.7	54.0	54.3	54.5	54.8	55.1	55.4	55.7	56.0	56.3	56.6	56.9	57.1	57.4

Pol. =	**40.0**	**40.2**	**40.4**	**40.6**	**40.8**	**41.0**	**41.2**	**41.4**	**41.6**	**41.8**	**42.0**	**42.2**	**42.4**	**42.6**	**42.8**	**43.0**	**43.2**	**43.4**	**43.6**	**43.8**	**44.0**	**44.2**	**44.4**	**44.6**	**44.8**
16.0	61.2	61.5	61.8	62.1	62.4	62.7	63.0	63.3	63.6	63.9	64.2	64.5	64.9	65.2	65.5	65.8	66.1	66.4	66.7	67.0	67.3	67.6	67.9	68.2	68.5
.1	60.8	61.1	61.4	61.7	62.0	62.3	62.6	62.9	63.2	63.5	63.8	64.1	64.4	64.7	65.0	65.3	65.6	65.9	66.3	66.6	66.9	67.2	67.5	67.8	68.1
.2	60.4	60.7	61.0	61.3	61.6	61.9	62.2	62.5	62.8	63.1	63.4	63.7	64.0	64.3	64.6	64.9	65.2	65.5	65.8	66.1	66.4	66.7	67.0	67.3	67.6
.3	60.0	60.3	60.6	60.9	61.2	61.5	61.8	62.1	62.4	62.7	63.0	63.3	63.6	63.9	64.2	64.5	64.8	65.1	65.4	65.7	66.0	66.3	66.6	66.9	67.2
.4	59.6	59.9	60.2	60.5	60.8	61.1	61.4	61.7	62.0	62.3	62.6	62.9	63.2	63.5	63.8	64.1	64.4	64.7	65.0	65.3	65.6	65.9	66.2	66.4	66.7
.5	59.2	59.5	59.8	60.1	60.4	60.7	61.0	61.3	61.6	61.9	62.2	62.5	62.8	63.1	63.4	63.7	63.9	64.2	64.5	64.8	65.1	65.5	65.7	66.0	66.3
.6	58.8	59.1	59.4	59.7	60.0	60.3	60.6	60.9	61.2	61.5	61.8	62.1	62.4	62.7	63.0	63.2	63.5	63.8	64.1	64.4	64.7	65.0	65.3	65.6	65.9
.7	58.5	58.7	59.0	59.3	59.6	59.9	60.2	60.5	60.8	61.1	61.4	61.7	62.0	62.3	62.5	62.8	63.1	63.4	63.7	64.0	64.3	64.6	64.9	65.2	65.5
.8	58.1	58.4	58.7	59.0	59.2	59.5	59.8	60.1	60.4	60.7	61.0	61.3	61.6	61.9	62.1	62.4	62.7	63.0	63.3	63.6	63.9	64.2	64.5	64.8	65.1
.9	57.7	58.0	58.3	58.6	58.9	59.2	59.4	59.7	60.0	60.3	60.6	60.9	61.2	61.5	61.8	62.0	62.3	62.6	62.9	63.2	63.5	63.8	64.1	64.4	64.6

Pol. =	**45.0**	**45.2**	**45.4**	**45.6**	**45.8**	**46.0**	**46.2**	**46.4**	**46.6**	**46.8**	**47.0**	**47.2**	**47.4**	**47.6**	**47.8**	**48.0**	**48.2**	**48.4**	**48.6**	**48.8**	**49.0**	**49.2**	**49.4**	**49.6**	**49.8**
16.0	68.8	69.1	69.4	69.7	70.1	70.4	70.7	71.0	71.3	71.6	71.9	72.2	72.5	72.8	73.1	73.4	73.7	74.0	74.3	74.6	75.0	75.3	75.6	75.9	76.2
.1	68.4	68.7	69.0	69.3	69.6	69.9	70.2	70.5	70.8	71.1	71.4	71.7	72.0	72.3	72.6	72.9	73.2	73.5	73.8	74.2	74.5	74.8	75.1	75.4	75.7
.2	67.9	68.2	68.5	68.8	69.1	68.4	69.7	70.0	70.3	70.6	70.9	71.2	71.6	71.9	72.2	72.5	72.8	73.1	73.4	73.7	74.0	74.3	74.6	74.9	75.2
.3	67.5	67.8	68.1	68.4	68.7	69.0	69.3	69.6	69.9	70.2	70.5	70.8	71.1	71.4	71.7	72.0	72.3	72.6	72.9	73.2	73.5	73.8	74.1	74.4	74.7
.4	67.0	67.3	67.6	67.9	68.2	68.5	68.8	69.1	69.4	69.7	70.0	70.3	70.6	70.9	71.2	71.5	71.8	72.1	72.4	72.7	73.0	73.3	73.6	73.9	74.2
.5	66.6	66.9	67.2	67.5	67.8	68.1	68.4	68.7	69.0	69.3	69.6	69.9	70.2	70.5	70.8	71.1	71.4	71.6	71.9	72.2	72.5	72.8	73.1	73.4	73.7
.6	66.2	66.5	66.8	67.1	67.4	67.7	68.0	68.2	68.5	68.8	69.1	69.4	69.7	70.0	70.3	70.6	70.9	71.2	71.5	71.8	72.1	72.4	72.7	73.0	73.2
.7	65.8	66.1	66.3	66.6	66.9	67.2	67.5	67.8	68.1	68.4	68.7	69.0	69.3	69.6	69.9	70.1	70.4	70.7	71.0	71.3	71.6	71.9	72.2	72.5	72.8
.8	65.3	65.6	65.9	66.2	66.5	66.8	67.1	67.4	67.7	68.0	68.2	68.5	68.8	69.1	69.4	69.7	70.0	70.3	70.6	70.9	71.2	71.4	71.7	72.0	72.3
.9	64.9	65.2	65.5	65.8	66.1	66.4	66.7	67.0	67.2	67.5	67.8	68.1	68.4	68.7	69.0	69.3	69.5	69.8	70.1	70.4	70.7	71.0	71.3	71.6	71.9

TABLE 41 (*Continued*)

EXPANDED HORNE'S TABLE OF COEFFICIENTS OF PURITY

Pol. =	**50.0**	**50.2**	**50.4**	**50.6**	**50.8**	**51.0**	**51.2**	**51.4**	**51.6**	**51.8**	**52.0**	**52.2**	**52.4**	**52.6**	**52.8**	**53.0**	**53.2**	**53.4**	**53.6**	**53.8**	**54.0**	**54.2**	**54.4**	**54.6**	**54.8**
16.0	76.5	76.8	77.1	77.4	77.7	78.0	78.3	78.6	78.9	79.2	79.5	79.8	80.2	80.5	80.8	81.1	81.4	81.7	82.0	82.3	82.6	82.9	83.2	83.5	83.8
.1	76.0	76.3	76.6	76.9	77.2	77.5	77.8	78.1	78.4	78.7	79.0	79.3	79.6	79.9	80.2	80.5	80.8	81.1	81.4	81.7	82.1	82.4	82.7	83.0	83.3
.2	75.5	75.8	76.1	76.4	76.7	77.0	77.3	77.6	77.9	78.2	78.5	78.8	79.1	79.4	79.7	80.0	80.3	80.6	80.9	81.2	81.5	81.8	82.1	82.4	82.7
.3	75.0	75.3	75.6	75.9	76.2	76.5	76.8	77.1	77.4	77.7	78.0	78.3	78.6	78.9	79.2	79.5	79.8	80.1	80.4	80.7	81.0	81.3	81.6	81.9	82.2
.4	74.5	74.8	75.1	75.4	75.7	76.0	76.3	76.6	76.9	77.2	77.5	77.8	78.1	78.4	78.7	79.0	79.3	79.6	79.9	80.2	80.5	80.8	81.1	81.3	81.6
.5	74.0	74.3	74.6	74.9	75.2	75.5	75.8	76.1	76.4	76.7	77.0	77.3	77.6	77.9	78.2	78.5	78.8	79.0	79.3	79.6	79.9	80.2	80.5	80.8	81.1
.6	73.5	73.8	74.1	74.4	74.7	75.0	75.3	75.6	75.9	76.2	76.5	76.8	77.1	77.4	77.7	78.0	78.2	78.5	78.8	79.1	79.4	79.7	80.0	80.3	80.6
.7	73.1	73.4	73.7	73.9	74.2	74.5	74.8	75.1	75.4	75.7	76.0	76.3	76.6	76.9	77.2	77.5	77.7	78.0	78.3	78.6	78.9	79.2	79.5	79.8	80.1
.8	72.6	72.9	73.2	73.5	73.8	74.1	74.3	74.6	74.9	75.2	75.5	75.8	76.1	76.4	76.7	77.0	77.3	77.5	77.8	78.1	78.4	78.7	79.0	79.3	79.6
.9	72.1	72.4	72.7	73.0	73.3	73.6	73.9	74.2	74.5	74.7	75.0	75.3	75.6	75.9	76.2	76.5	76.8	77.1	77.3	77.6	77.9	78.2	78.5	78.8	79.1

Pol. =	**55.0**	**55.2**	**55.4**	**55.6**	**55.8**	**56.0**	**56.2**	**56.4**	**56.6**	**56.8**	**57.0**	**57.2**	**57.4**	**57.6**	**57.8**	**58.0**	**58.2**	**58.4**	**58.6**	**58.8**	**59.0**	**59.2**	**59.4**	**59.6**	**59.8**
16.0	84.1	84.4	84.7	85.0	85.4	85.7	86.0	86.3	86.6	86.9	87.2	87.5	87.8	88.1	88.4	88.7	89.0	89.3	89.6	89.9	90.2	90.6	90.9	91.2	91.5
.1	83.6	83.9	84.2	84.5	84.8	85.1	85.4	85.7	86.0	86.3	86.6	86.9	87.2	87.5	87.8	88.1	88.4	88.7	89.0	89.3	89.7	90.0	90.3	90.6	90.9
.2	83.0	83.3	83.6	83.9	84.2	84.5	84.8	85.1	85.4	85.7	86.0	86.3	86.6	86.9	87.2	87.6	87.9	88.2	88.5	88.8	89.1	89.4	89.7	90.0	90.3
.3	82.5	82.8	83.1	83.4	83.7	84.0	84.3	84.6	84.9	85.2	85.5	85.8	86.1	86.4	86.7	87.0	87.3	87.6	87.9	88.2	88.5	88.8	89.1	89.4	89.7
.4	81.9	82.2	82.5	82.8	83.1	83.4	83.7	84.0	84.3	84.6	84.9	85.2	85.5	85.8	86.1	86.4	86.7	87.0	87.3	87.6	87.9	88.2	88.5	88.8	89.1
.5	81.4	81.7	82.0	82.3	82.6	82.9	83.2	83.5	83.8	84.1	84.4	84.7	85.0	85.3	85.6	85.9	86.2	86.4	86.7	87.0	87.3	87.6	87.9	88.2	88.5
.6	80.9	81.2	81.5	81.8	82.1	82.4	82.7	83.0	83.2	83.5	83.8	84.1	84.4	84.7	85.0	85.3	85.6	85.9	86.2	86.5	86.8	87.1	87.4	87.7	88.0
.7	80.4	80.7	81.0	81.3	81.5	81.8	82.1	82.4	82.7	83.0	83.3	83.6	83.9	84.2	84.5	84.8	85.1	85.3	85.6	85.9	86.2	86.5	86.8	87.1	87.4
.8	79.9	80.2	80.4	80.7	81.0	81.3	81.6	81.9	82.2	82.5	82.8	83.1	83.4	83.6	83.9	84.2	84.5	84.8	85.1	85.4	85.7	86.0	86.3	86.5	86.8
.9	79.4	79.6	79.9	80.2	80.5	80.8	81.1	81.4	81.7	82.0	82.2	82.5	82.8	83.1	83.4	83.7	84.0	84.3	84.6	84.8	85.1	85.4	85.7	86.0	86.3

Pol. =	**60.0**	**60.2**	**60.4**	**60.6**	**60.8**	**61.0**	**61.2**	**61.4**	**61.6**	**61.8**	**62.0**	**62.2**	**62.4**	**62.6**	**62.8**	**63.0**	**63.2**	**63.4**	**63.6**	**63.8**	**64.0**	**64.2**	**64.4**	**64.6**	**64.8**
16.0	91.8	92.1	92.4	92.7	93.0	93.3	93.6	93.9	94.2	94.5	94.8	95.1	95.4	95.8	96.1	96.4	96.7	97.0	97.3	97.6	97.9	98.2	98.5	98.8	99.1
.1	91.2	91.5	91.8	92.1	92.4	92.7	93.0	93.3	93.6	93.9	94.2	94.5	94.8	95.1	95.4	95.7	96.0	96.3	96.6	96.9	97.2	97.6	97.9	98.2	98.5
.2	90.6	90.9	91.2	91.5	91.8	92.1	92.4	92.7	93.0	93.3	93.6	93.9	94.2	94.5	94.8	95.1	95.4	95.7	96.0	96.3	96.6	96.9	97.2	97.5	97.8
.3	90.0	90.3	90.6	90.9	91.2	91.5	91.8	92.1	92.4	92.7	93.0	93 3	93.6	93.9	94.2	94.5	94.8	95.1	95.4	95.7	96.0	96.3	96.6	96.9	97.2
.5	89.4	89.7	90.0	90.3	90.6	90.9	91.2	91.5	91.8	92.1	92.4	92.7	93.0	93.3	93.6	93.9	94.2	94.5	94.8	95.1	95.4	95.7	95.9	96.2	96.5
.5	88.8	89.1	89.4	89.7	90.0	90.3	90.6	90.9	91.2	91.5	91.8	92.1	92.4	92.7	93.0	93.3	93.6	93.9	94.1	94.4	94.7	95.0	95.3	95.6	95.9
.6	88.2	88.5	88.8	89.1	89.4	89.7	90.0	90.3	90.6	90.9	91.2	91.5	91.8	92.1	92.4	92.7	93.0	93.2	93.5	93.8	94.1	94.4	94.7	95.0	95.3
.7	87.7	88.0	88.3	88.6	88.9	89.1	89.4	89.7	90.0	90.3	90.6	90.9	91.2	91.5	92.8	92.1	92.4	92.7	92.9	93.2	93.5	93.8	94.1	94.4	94.7
.8	87.1	87.4	87.7	88.0	88.3	88.6	88.9	89.2	89.4	89.7	90.0	90.3	90.6	90.9	91.2	91.5	91.8	92.1	92.4	92.6	92.9	93.2	93.5	93.8	94.1
.9	86.6	86.9	87.2	87.4	87.7	88.0	88.3	88.6	88.9	89.2	89.5	89.7	90.0	90.3	90.6	90.9	91.2	91.5	91.8	92.1	92.3	92.6	92.9	93.2	93.5

TABLE 41 (*Continued*)

EXPANDED HORNE'S TABLE OF COEFFICIENTS OF PURITY

Pol. =	**45.0**	**45.2**	**45.4**	**45.6**	**45.8**	**46.0**	**46.2**	**46.4**	**46.6**	**46.8**	**47.0**	**47.2**	**47.4**	**47.6**	**47.8**	**48.0**	**48.2**	**48.4**	**48.6**	**48.8**	**49.0**	**49.2**	**49.4**	**49.6**	**49.8**
17.0	64.5	64.8	65.0	65.4	65.6	65.9	66.2	66.5	66.8	67.1	67.3	67.6	67.9	68.2	68.5	68.8	69.1	69.3	69.6	69.9	70.2	70.5	70.8	71.1	71.4
.1	64.1	64.4	64.6	64.9	65.2	65.5	65.8	66.1	66.4	66.6	66.9	67.2	67.5	67.8	68.1	68.3	68.6	68.9	69.2	69.5	69.8	70.1	70.3	70.6	70.9
.2	63.7	64.0	64.3	64.6	64.9	65.1	65.4	65.7	66.0	66.3	66.6	66.8	67.1	67.4	67.7	68.0	68.3	68.5	68.8	69.1	69.4	69.7	70.0	70.2	70.5
.3	63.3	63.6	63.9	64.2	64.4	64.7	65.0	65.3	65.6	65.9	66.1	66.4	66.7	67.0	67.3	67.5	67.8	68.1	68.4	68.7	69.0	69.2	69.5	69.8	70.1
.4	62.9	63.2	63.5	63.8	64.1	64.3	64.6	64.9	65.2	65.5	65.7	66.0	66.3	66.6	66.9	67.1	67.4	67.7	68.0	68.3	68.5	68.8	69.1	69.4	69.7
.5	62.6	62.8	63.1	63.4	63.7	63.9	64.2	64.5	64.8	65.1	65.3	65.6	65.9	66.2	66.4	66.7	67.0	67.3	67.6	67.8	68.1	68.4	68.7	68.9	69.2
.6	62.2	62.4	62.7	63.0	63.3	63.6	63.8	64.1	64.4	64.7	64.9	65.2	65.5	65.8	66.0	66.3	66.6	66.9	67.1	67.4	67.7	68.0	68.3	68.5	68.8
.7	61.8	62.1	62.3	62.6	62.9	63.2	63.4	63.7	64.0	64.3	64.5	64.8	65.1	65.4	65.6	65.9	66.2	66.5	66.7	67.0	67.3	67.6	67.8	68.1	68.4
.8	61.4	61.7	62.0	62.2	62.5	62.8	63.1	63.3	63.6	63.9	64.2	64.4	64.7	65.0	65.2	65.5	65.8	66.1	66.3	66.6	66.9	67.2	67.4	67.7	68.0
.9	61.1	61.3	61.6	61.9	62.1	62.4	62.7	63.0	63.2	63.5	63.8	64.0	64.3	64.6	64.9	65.1	65.4	65.7	65.9	66.2	66.5	66.7	67.0	67.3	67.6

Pol. =	**50.0**	**50.2**	**50.4**	**50.6**	**50.8**	**51.0**	**51.2**	**51.4**	**51.6**	**51.8**	**52.0**	**52.2**	**52.4**	**52.6**	**52.8**	**53.0**	**53.2**	**53.4**	**53.6**	**53.8**	**54.0**	**54.2**	**54.4**	**54.6**	**54.8**
17.0	71.6	71.9	72.2	72.5	72.8	73.1	73.4	73.6	73.9	74.2	74.5	74.8	75.1	75.4	75.7	75.9	76.2	76.5	76.8	77.1	77.4	77.7	77.9	78.2	78.5
.1	71.2	71.5	71.8	72.0	72.3	72.6	72.9	73.2	73.5	73.8	74.0	74.3	74.6	74.9	75.2	75.5	75.8	76.0	76.3	76.6	76.9	77.2	77.5	77.7	78.0
.2	70.8	71.1	71.4	71.6	71.9	72.2	72.5	72.8	73.1	73.3	73.6	73.9	74.2	74.5	74.8	75.0	75.2	75.6	75.9	76.2	76.5	76.7	77.0	77.3	77.6
.3	70.4	70.6	70.9	71.2	71.5	71.8	72.0	72.3	72.6	72.9	73.2	73.5	73.7	74.0	74.3	74.6	74.9	75.1	75.4	75.7	76.0	76.3	76.6	76.8	77.1
.4	69.9	70.2	70.5	70.8	71.0	71.3	71.6	71.9	72.2	72.4	72.7	73.0	73.3	73.6	73.8	74.1	74.4	74.7	75.0	75.2	75.5	75.8	76.1	76.4	76.6
.5	69.5	69.8	70.1	70.3	70.6	70.9	71.2	71.4	71.7	72.0	72.3	72.5	72.8	73.1	73.4	73.7	73.9	74.2	74.5	74.8	75.1	75.3	75.6	75.9	76.2
.6	69.1	69.4	69.6	69.9	70.2	70.5	70.7	71.0	71.3	71.6	71.8	72.1	72.4	72.7	72.9	73.2	73.5	73.8	74.1	74.3	74.6	74.9	75.2	75.4	75.7
.7	68.7	68.9	69.2	69.5	69.8	70.0	70.3	70.6	70.9	71.1	71.4	71.7	72.0	72.2	72.5	72.8	73.1	73.3	73.6	73.9	74.2	74.4	74.7	75.0	75.3
.8	68.2	68.5	68.8	69.1	69.3	69.6	69.9	70.2	70.4	70.7	71.0	71.2	71.5	71.8	72.1	72.3	72.6	72.9	73.2	73.4	73.7	74.0	74.3	74.5	74.8
.9	67.8	68.1	68.4	68.6	68.9	69.2	69.5	69.7	70.0	70.3	70.5	70.8	71.1	71.4	71.6	71.9	72.2	72.4	72.7	73.0	73.3	73.5	73.8	74.1	74.3

Pol. =	**55.0**	**55.2**	**55.4**	**55.6**	**55.8**	**56.0**	**56.2**	**56.4**	**56.6**	**56.8**	**57.0**	**57.2**	**57.4**	**57.6**	**57.8**	**58.0**	**58.2**	**58.4**	**58.6**	**58.8**	**59.0**	**59.2**	**59.4**	**59.6**	**59.8**
17.0	78.8	79.1	79.4	79.7	80.0	80.2	80.5	80.8	81.1	81.4	81.7	82.0	82.3	82.5	82.8	83.1	83.4	83.7	84.0	84.3	84.5	84.8	85.1	85.4	85.7
.1	78.3	78.6	78.9	79.2	79.5	79.7	80.0	80.3	80.6	80.9	81.2	81.5	81.7	82.0	82.3	82.6	82.9	83.2	83.4	83.7	84.0	84.3	84.6	84.9	85.2
.2	77.9	78.2	78.4	78.7	79.0	79.3	79.6	79.9	80.1	80.4	80.7	81.0	81.3	81.6	81.8	82.1	82.4	82.7	83.0	83.3	83.5	83.8	84.1	84.4	84.7
.3	77.4	77.7	78.0	78.2	78.5	78.8	79.1	79.4	79.6	79.9	80.2	80.5	80.8	81.1	81.3	81.6	81.9	82.2	82.5	82.7	83.0	83.3	83.6	83.9	84.2
.4	76.9	77.2	77.5	77.8	78.0	78.3	78.6	78.9	79.2	79.4	79.7	80.0	80.3	80.6	80.8	81.1	81.4	81.7	82.0	82.2	82.5	82.8	83.1	83.4	83.6
.5	76.5	76.7	77.0	77.3	77.6	77.8	78.1	78.4	78.7	79.0	79.2	79.5	79.8	80.1	80.3	80.6	80.9	81.2	81.5	81.7	82.0	82.3	82.6	82.8	83.1
.6	76.0	76.3	76.5	76.8	77.1	77.4	77.6	77.9	78.2	78.5	78.8	79.0	79.3	79.6	79.9	80.1	80.4	80.7	81.0	81.2	81.5	81.8	82.1	82.3	82.6
.7	75.5	75.8	76.1	76.3	76.6	76.9	77.2	77.4	77.7	78.0	78.3	78.5	78.8	79.1	79.4	79.6	79.9	80.2	80.5	80.7	81.0	81.3	81.6	81.8	82.1
.8	75.1	75.3	75.6	75.9	76.2	76.4	76.7	77.0	77.3	77.5	77.8	78.1	78.3	78.6	78.9	79.2	79.4	79.7	80.0	80.3	80.5	80.8	81.1	81.3	81.6
.9	74.6	74.9	75.2	75.4	75.7	76.0	76.2	76.5	76.8	77.1	77.3	77.6	77.9	78.1	78.4	78.7	79.0	79.2	79.5	79.8	80.0	80.3	80.6	80.9	81.1

TABLE 41 (*Continued*)

EXPANDED HORNE'S TABLE OF COEFFICIENTS OF PURITY

Pol. =	60.0	60.2	60.4	60.6	60.8	61.0	61.2	61.4	61.6	61.8	62.0	62.2	62.4	62.6	62.8	63.0	63.2	63.4	63.6	63.8	64.0	64.2	64.4	64.6	64.8
17.0	86.0	86.3	86.6	86.8	87.1	87.4	87.7	88.0	88.3	88.6	88.8	89.1	89.4	89.7	90.0	90.3	90.6	90.9	91.1	91.4	91.7	92.0	92.3	92.6	92.9
.1	85.4	85.7	86.0	86.3	86.6	86.9	87.2	87.4	87.7	88.0	88.3	88.6	88.9	89.1	89.4	89.7	90.0	90.3	90.6	90.9	91.1	91.4	91.7	92.0	92.3
.2	85.0	85.2	85.5	85.8	86.1	86.4	86.7	86.9	87.2	87.5	87.8	88.1	88.4	88.6	88.9	89.2	89.5	89.8	90.1	90.3	90.6	90.9	91.2	91.5	91.8
.3	84.4	84.7	85.0	85.3	85.6	85.8	86.1	86.4	86.7	87.0	87.2	87.5	87.8	88.1	88.4	88.7	88.9	89.2	89.5	89.8	90.1	90.3	90.6	90.9	91.2
.4	83.9	84.2	84.5	84.8	85.0	85.3	85.6	85.9	86.2	86.4	86.7	87.0	87.3	87.6	87.8	88.1	88.4	88.7	89.0	89.2	89.5	89.8	90.1	90.3	90.6
.5	83.4	83.7	84.0	84.2	84.5	84.8	85.1	85.3	85.6	85.9	86.2	86.5	86.7	87.0	87.3	87.6	87.8	88.1	88.4	88.7	89.0	89.2	89.5	89.8	90.1
.6	82.9	83.2	83.4	83.7	84.0	84.3	84.6	84.8	85.1	85.4	85.7	85.9	86.2	86.5	86.8	87.0	87.3	87.6	87.9	88.1	88.4	88.7	89.0	89.3	89.5
.7	82.4	82.7	82.9	83.2	83.5	83.8	84.0	84.3	84.6	84.9	85.1	85.4	85.7	86.0	86.2	86.5	86.8	87.1	87.3	87.6	87.9	88.2	88.4	88.7	89.0
.8	81.9	82.2	82.4	82.7	83.0	83.3	83.5	83.8	84.1	84.4	84.6	84.9	85.2	85.4	85.7	86.0	86.3	86.5	86.8	87.1	87.4	87.6	87.9	88.2	88.4
.9	81.4	81.7	81.9	82.2	82.5	82.8	83.0	83.3	83.6	83.8	84.1	84.4	84.7	84.9	85.2	85.5	85.7	86.0	86.3	86.6	86.8	87.1	87.4	87.6	87.9

Pol. =	65.0	65.2	65.4	65.6	65.8	66.0	66.2	66.4	66.6	66.8	67.0	67.2	67.4	67.6	67.8	68.0	68.2	68.4	68.6	68.8	69.0	69.2	69.4	69.6	69.8
17.0	93.1	93.4	93.7	94.0	94.3	94.6	94.9	95.2	95.4	95.7	96.0	96.3	96.6	96.9	97.2	97.4	97.7	98.0	98.3	98.6	98.9	99.2	99.5	99.7	100.0
.1	92.6	92.9	93.1	93.4	93.7	94.0	94.3	94.6	94.9	95.1	95.4	95.7	96.0	96.3	96.6	96.8	97.1	97.4	97.7	98.0	98.3	98.6	98.9	99.2	99.5
.2	92.0	92.3	92.6	92.9	93.2	93.5	93.7	94.0	94.3	94.6	94.9	95.2	95.4	95.7	96.0	96.3	96.6	96.9	97.1	97.4	97.7	98.0	98.3	98.6	98.8
.3	91.5	91.7	92.0	92.3	92.6	92.9	93.2	93.4	93.7	94.0	94.3	94.6	94.8	95.1	95.4	95.7	96.0	96.3	96.5	96.8	97.1	97.4	97.7	97.9	98.2
.4	90.9	91.2	91.5	91.7	92.0	92.3	92.6	92.9	93.1	93.4	93.7	94.0	94.3	94.5	94.8	95.1	95.4	95.7	95.9	96.2	96.5	96.8	97.1	97.3	97.6
.5	90.4	90.6	90.9	91.2	91.5	91.7	92.0	92.3	92.6	92.9	93.1	93.4	93.7	94.0	94.2	94.5	94.8	95.1	95.4	95.6	95.9	96.2	96.5	96.7	97.0
.6	89.8	90.1	90.4	90.6	90.9	91.2	91.5	91.7	92.0	92.3	92.6	92.8	93.1	93.4	93.7	93.9	94.2	94.5	94.8	95.1	95.3	95.6	95.9	96.2	96.4
.7	89.3	89.5	89.8	90.1	90.4	90.6	90.9	91.2	91.5	91.7	92.0	92.3	92.6	92.8	93.1	93.4	93.7	93.9	94.2	94.5	94.8	95.0	95.3	95.6	95.8
.8	88.7	89.0	89.3	89.5	89.8	90.1	90.4	90.6	90.9	91.2	91.4	91.7	92.0	92.3	92.5	92.8	93.1	93.4	93.6	93.9	94.2	94.5	94.7	95.0	95.3
.9	88.2	88.5	88.7	89.0	89.3	89.5	89.8	90.1	90.4	90.6	90.9	91.2	91.4	91.7	92.0	92.3	92.5	92.8	93.1	93.3	93.6	93.9	94.2	94.4	94.7

Pol. =	70.0	70.2	70.4	70.6	70.8	71.0	71.2	71.4	71.6	71.8	72.0	72.2	72.4	72.6	72.8	73.0	73.2	73.4	73.6	73.8	74.0	74.2	74.4	74.6	74.8
17.0																									
.1	99.7	100.0																							
.2	99.1	99.4	99.7	100.0																					
.3	98.5	98.8	99.1	99.3	99.6	99.9	100.2																		
.4	97.9	98.2	98.5	98.7	99.0	99.3	99.6	99.9	100.1																
.5	97.3	97.6	97.9	98.1	98.4	98.7	99.0	99.2	99.5	99.8	100.1														
.6	96.7	97.0	97.3	97.5	97.8	98.1	98.4	98.6	98.9	99.2	99.5	99.8	100.0												
.7	96.1	96.4	96.7	96.9	97.2	97.5	97.8	98.0	98.3	98.6	98.9	99.1	99.4	99.7	100.0										
.8	95.5	95.8	96.1	96.4	96.6	96.9	97.2	97.5	97.7	98.0	98.3	98.5	98.8	99.1	99.4	99.6	99.9	100.2							
.9	95.0	95.2	95.5	95.8	96.1	96.3	96.6	96.9	97.1	97.4	97.7	98.0	98.2	98.5	98.8	99.0	99.3	99.6	99.9	100.1					

TABLE 41 (*Continued*)

EXPANDED HORNE'S TABLE OF COEFFICIENTS OF PURITY

Pol. =	**50.0**	**50.2**	**50.4**	**50.6**	**50.8**	**51.0**	**51.2**	**51.4**	**51.6**	**51.8**	**52.0**	**52.2**	**52.4**	**52.6**	**52.8**	**53.0**	**53.2**	**53.4**	**53.6**	**53.8**	**54.0**	**54.2**	**54.4**	**54.6**	**54.8**
18.0	67.4	67.7	68.0	68.2	68.5	68.8	69.1	69.3	69.6	69.9	70.1	70.4	70.7	70.9	71.2	71.5	71.8	72.0	72.3	72.6	72.8	73.1	73.4	73.6	73.9
.1	67.0	67.3	67.6	67.8	68.1	68.4	68.6	68.9	69.2	69.4	69.7	70.0	70.3	70.5	70.8	71.1	71.3	71.6	71.9	72.1	72.4	72.9	72.9	73.2	73.5
.2	66.6	66.9	67.2	67.4	67.7	68.0	68.2	68.5	68.8	69.0	69.3	69.6	69.8	70.1	70.4	70.6	70.9	71.2	71.4	71.7	72.0	72.2	72.5	72.8	73.0
.3	66.2	66.5	66.8	67.0	67.3	67.6	67.8	68.1	68.4	68.6	68.9	69.2	69.4	69.7	70.0	70.2	70.5	70.7	71.0	71.3	71.5	71.8	72.1	72.3	72.6
.4	65.9	66.1	66.4	66.7	66.9	67.2	67.4	67.7	68.0	68.2	68.5	68.8	69.0	69.3	69.5	69.8	70.1	70.3	70.6	70.9	71.1	71.4	71.7	71.9	72.2
.5	65.5	65.7	66.0	66.3	66.5	66.8	67.0	67.3	67.6	67.8	68.1	68.4	68.6	68.9	69.1	69.4	69.7	69.9	70.2	70.5	70.7	71.0	71.2	71.5	71.8
.6	65.1	65.4	65.6	65.9	66.1	66.4	66.7	66.9	67.2	67.4	67.7	68.0	68.2	68.5	68.7	69.0	69.3	69.5	69.8	70.0	70.3	70.6	70.8	71.1	71.3
.7	64.7	65.0	65.2	65.5	65.8	66.0	66.3	66.5	66.8	67.1	67.3	67.6	67.8	68.1	68.3	68.6	68.9	69.1	69.4	69.6	69.9	70.2	70.4	70.7	70.9
.8	64.4	64.6	64.9	65.1	65.4	65.6	65.9	66.2	66.4	66.7	66.9	67.2	67.4	67.7	68.0	68.2	68.5	68.7	69.0	69.2	69.5	69.8	70.0	70.3	70.5
.9	64.0	64.2	64.5	64.8	65.0	65.3	65.5	65.8	66.0	66.3	66.5	66.8	67.1	67.3	67.6	67.8	68.1	68.3	68.6	68.8	69.1	69.4	69.6	69.9	70.1

Pol. =	**55.0**	**55.2**	**55.4**	**55.6**	**55.8**	**56.0**	**56.2**	**56.4**	**56.6**	**56.8**	**57.0**	**57.2**	**57.4**	**57.6**	**57.8**	**58.0**	**58.2**	**58.4**	**58.6**	**58.8**	**59.0**	**59.2**	**59.4**	**59.6**	**59.8**
18.0	74.2	74.4	74.7	75.0	75.3	75.5	75.8	76.1	76.3	76.6	76.9	77.1	77.4	77.7	78.0	78.2	78.5	78.8	79.0	79.3	79.6	79.8	80.1	80.4	80.7
.1	73.7	74.0	74.3	74.5	74.8	75.1	75.3	75.6	75.9	76.2	76.4	76.7	77.0	77.2	77.5	77.8	78.0	78.3	78.6	78.8	79.1	79.4	79.6	79.9	80.2
.2	73.3	73.6	73.8	74.1	74.4	74.6	74.9	75.2	75.4	75.7	76.0	76.2	76.5	76.8	77.0	77.3	77.6	77.8	78.1	78.4	78.6	78.9	79.2	79.4	79.7
.3	72.9	73.1	73.4	73.7	73.9	74.2	74.5	74.7	75.0	75.3	75.5	75.8	76.0	76.3	76.6	76.8	77.1	77.4	77.6	77.9	78.2	78.4	78.7	79.0	79.2
.4	72.4	72.7	73.0	73.2	73.5	73.8	74.0	74.3	74.6	74.8	75.1	75.3	75.6	75.9	76.1	76.4	76.7	76.9	77.2	77.5	77.7	78.0	78.2	78.5	78.8
.5	72.0	72.3	72.5	72.8	73.1	73.3	73.6	73.9	74.1	74.4	74.6	74.9	75.2	75.4	75.7	76.0	76.2	76.5	76.7	77.0	77.3	77.5	77.8	78.0	78.3
.6	71.6	71.9	72.1	72.4	72.6	72.9	73.2	73.4	73.7	74.0	74.2	74.5	74.7	75.0	75.3	75.5	75.8	76.0	76.3	76.6	76.8	77.1	77.3	77.6	77.9
.7	71.2	71.5	71.7	72.0	72.2	72.5	72.8	73.0	73.3	73.5	73.8	74.0	74.3	74.6	74.8	75.1	75.3	75.6	75.9	76.1	76.4	76.6	76.9	77.2	77.4
.8	70.8	71.0	71.3	71.6	71.8	72.1	72.3	72.6	72.8	73.1	73.4	73.6	73.9	74.1	74.4	74.7	74.9	75.2	75.4	75.7	75.9	76.2	76.5	76.7	77.0
.9	70.4	70.6	70.9	71.2	71.4	71.7	71.9	72.2	72.4	72.7	72.9	73.2	73.5	73.7	74.0	74.2	74.5	74.7	75.0	75.2	75.5	75.8	76.0	76.3	76.5

Pol. =	**60.0**	**60.2**	**60.4**	**60.6**	**60.8**	**61.0**	**61.2**	**61.4**	**61.6**	**61.8**	**62.0**	**62.2**	**62.4**	**62.6**	**62.8**	**63.0**	**63.2**	**63.4**	**63.6**	**63.8**	**64.0**	**64.2**	**64.4**	**64.6**	**64.8**
18.0	80.9	81.2	81.5	81.7	82.0	82.3	82.5	82.8	83.1	83.3	83.6	83.9	84.2	84.4	84.7	85.0	85.2	85.5	85.8	86.0	86.3	86.6	86.9	87.1	87.4
.1	80.4	80.7	81.0	81.2	81.5	81.8	82.1	82.3	82.6	82.9	83.1	83.4	83.7	83.9	84.2	84.5	84.7	85.0	85.3	85.5	85.8	86.1	86.3	86.6	86.9
.2	80.0	80.2	80.5	80.8	81.0	81.3	81.6	81.8	82.1	82.4	82.6	82.9	83.2	83.4	83.7	84.0	84.2	84.5	84.8	85.0	85.3	85.6	85.8	86.1	86.4
.3	79.5	79.8	80.0	80.3	80.6	80.8	81.1	81.3	81.6	81.9	82.1	82.4	82.7	82.9	83.2	83.5	83.7	84.0	84.3	84.5	84.8	85.1	85.3	85.6	85.9
.4	79.0	79.3	79.6	79.8	80.1	80.3	80.6	80.9	81.1	81.4	81.7	81.9	82.2	82.5	82.7	83.0	83.2	83.5	83.8	84.0	84.3	84.6	84.8	85.1	85.4
.5	78.6	78.8	79.1	79.4	79.6	79.9	80.1	80.4	80.7	80.9	81.3	81.5	81.7	82.0	82.2	82.5	82.8	83.0	83.3	83.5	83.8	84.1	84.3	84.6	84.9
.6	78.1	78.4	78.6	78.9	79.2	79.4	79.7	79.9	80.2	80.5	80.7	81.0	81.2	81.5	81.8	82.0	82.3	82.5	82.8	83.1	83.3	83.6	83.8	84.1	84.4
.7	77.7	77.9	78.2	78.4	78.7	79.0	79.2	79.5	79.7	80.0	80.3	80.5	80.8	81.0	81.3	81.6	81.8	82.1	82.3	82.6	82.8	83.1	83.4	83.6	83.9
.8	77.2	77.5	77.7	78.0	78.3	78.5	78.8	79.0	79.3	79.5	79.8	80.1	80.3	80.6	80.8	81.1	81.3	81.6	81.9	82.1	82.4	82.6	82.9	83.1	83.4
.9	76.8	77.0	77.3	77.5	77.8	78.1	78.3	78.6	78.8	79.1	79.3	79.6	79.9	80.1	80.4	80.6	80.9	81.1	81.4	81.6	81.9	82.2	82.4	82.7	82.9

TABLE 41 (*Continued*)

Expanded Horne's Table of Coefficients of Purity

Pol. =	65.0	65.2	65.4	65.6	65.8	66.0	66.2	66.4	66.6	66.8	67.0	67.2	67.4	67.6	67.8	68.0	68.2	68.4	68.6	68.8	69.0	69.2	69.4	69.6	69.8
18.0	87.7	87.9	88.2	88.5	88.7	89.0	89.3	89.6	89.8	90.1	90.4	90.6	90.9	91.2	91.4	91.7	92.0	92.3	92.5	92.8	93.1	93.3	93.6	93.9	94.1
.1	87.1	87.4	87.7	87.9	88.2	88.5	88.8	89.0	89.3	89.6	89.8	90.1	90.4	90.6	90.9	91.2	91.4	91.7	92.0	92.2	92.5	92.8	93.0	93.3	93.6
.2	86.6	86.9	87.2	87.4	87.7	88.0	88.2	88.5	88.8	89.0	89.3	89.6	89.8	90.1	90.4	90.6	90.9	91.2	91.4	91.7	92.0	92.2	92.5	92.8	93.0
.3	86.1	86.4	86.6	86.9	87.2	87.4	87.7	88.0	88.2	88.5	88.8	89.0	89.3	89.6	89.8	90.1	90.4	90.6	90.9	91.2	91.4	91.7	91.9	92.2	92.5
.4	85.6	85.9	86.1	86.4	86.7	86.9	87.2	87.5	87.7	88.0	88.3	88.5	88.8	89.0	89.3	89.6	89.8	90.1	90.4	90.6	90.9	91.2	91.4	91.7	91.9
.5	85.1	85.4	85.6	85.9	86.2	86.4	86.7	87.0	87.2	87.5	87.7	88.0	88.3	88.5	88.8	89.0	89.3	89.6	89.8	90.1	90.4	90.6	90.9	91.1	91.4
.6	84.6	84.9	85.2	85.4	85.7	85.9	86.2	86.5	86.7	87.0	87.2	87.5	87.8	88.0	88.3	88.5	88.8	89.1	89.3	89.6	89.8	90.1	90.4	90.6	90.9
.7	84.1	84.4	84.7	84.9	85.2	85.4	85.7	86.0	86.2	86.5	86.7	87.0	87.2	87.5	87.8	88.0	88.3	88.5	88.8	89.1	89.3	89.6	89.8	90.1	90.4
.8	83.7	83.9	84.2	84.4	84.7	84.9	85.2	85.5	85.7	86.0	86.2	86.5	86.8	87.0	87.3	87.5	87.8	88.0	88.3	88.6	88.8	89.1	89.3	89.6	89.8
.9	83.2	83.4	83.7	83.9	84.2	84.5	84.7	85.0	85.2	85.5	85.7	86.0	86.3	86.5	86.8	87.0	87.3	87.5	87.8	88.0	88.3	88.6	88.8	89.1	89.3

Pol. =	70.0	70.2	70.4	70.6	70.8	71.0	71.2	71.4	71.6	71.8	72.0	72.2	72.4	72.6	72.8	73.0	73.2	73.4	73.6	73.8	74.0	74.2	74.4	74.6	74.8
18.0	94.4	94.7	94.9	95.2	95.5	95.8	96.0	96.3	96.6	96.8	97.1	97.4	97.6	97.9	98.2	98.5	98.7	99.0	99.3	99.5	99.8	100.1			
.1	93.8	94.1	94.4	94.7	94.9	95.2	95.5	95.7	96.0	96.3	96.5	96.8	97.1	97.3	97.6	97.9	98.1	98.4	98.7	98.9	99.2	99.5	99.7	100.0	
.2	93.3	93.6	93.8	94.1	94.4	94.6	94.9	95.2	95.4	95.7	96.0	96.2	96.5	96.8	97.0	97.3	97.6	97.8	98.1	98.4	98.6	98.9	99.2	99.4	99.7
.3	92.7	93.0	93.3	93.5	93.8	94.1	94.3	94.6	94.9	95.1	95.4	95.7	95.9	96.2	96.5	96.7	97.0	97.2	97.5	97.8	98.0	98.3	98.6	98.8	99.1
.4	92.2	92.5	92.7	93.0	93.3	93.5	93.8	94.0	94.3	94.6	94.8	95.1	95.4	95.6	95.9	96.2	96.4	96.7	96.9	97.2	97.5	97.7	98.0	98.3	98.5
.5	91.7	91.9	92.2	92.5	92.7	93.0	93.2	93.5	93.8	94.0	94.3	94.5	94.8	95.1	95.3	95.6	95.9	96.1	96.4	96.6	96.9	97.2	97.4	97.7	98.0
.6	91.1	91.4	91.7	91.9	92.2	92.4	92.7	93.0	93.2	93.5	93.7	94.0	94.3	94.5	94.8	95.0	95.3	95.6	95.8	96.1	96.3	96.6	96.9	97.1	97.4
.7	90.6	90.9	91.1	91.4	91.7	91.9	92.2	92.4	92.7	92.9	93.2	93.5	93.7	94.0	94.2	94.5	94.8	95.0	95.3	95.5	95.8	96.1	96.3	96.6	96.8
.8	90.1	90.4	90.6	90.9	91.1	91.4	91.6	91.9	92.2	92.4	92.7	92.9	93.2	93.4	93.7	94.0	94.2	94.5	94.7	95.0	95.2	95.5	95.8	96.0	96.3
.9	89.6	89.8	90.1	90.3	90.6	90.9	91.1	91.4	91.6	91.9	92.1	92.4	92.7	92.9	93.2	93.4	93.7	93.9	94.2	94.4	94.7	95.0	95.2	95.5	95.7

Pol. =	75.0	75.2	75.4	75.6	75.8	76.0	76.2	76.4	76.6	76.8	77.0	77.2	77.4	77.6	77.8	78.0	78.2	78.4	78.6	78.8	79.0	79.2	79.4	79.6	79.8
18.0																									
.1																									
.2	100.0																								
.3	99.4	99.6	99.9	100.2																					
.4	98.8	99.1	99.3	99.6	99.8	100.1																			
.5	98.2	98.5	98.7	99.0	99.3	99.5	99.8	100.0																	
.6	97.7	97.9	98.2	98.4	98.7	99.0	99.2	99.5	99.7	100.0															
.7	97.1	97.3	97.6	97.9	98.1	98.4	98.6	98.9	99.2	99.4	99.7	99.9	100.2												
.8	96.5	96.8	97.0	97.3	97.6	97.8	98.1	98.3	98.6	98.8	99.1	99.4	99.6	99.9	100.1										
.9	96.0	96.2	96.5	96.7	97.0	97.3	97.5	97.8	98.0	98.3	98.5	98.8	99.0	99.3	99.6	99.8	100.1								

TABLE 41 (*Continued*)

EXPANDED HORNE'S TABLE OF COEFFICIENTS OF PURITY

Pol. =	55.0	55.2	55.4	55.6	55.8	56.0	56.2	56.4	56.6	56.8	57.0	57.2	57.4	57.6	57.8	58.0	58.2	58.4	58.6	58.8	59.0	59.2	59.4	59.6	59.8
19.0	70.0	70.2	70.5	70.8	71.0	71.3	71.5	71.8	72.0	72.3	72.5	72.8	73.0	73.3	73.6	73.8	74.1	74.3	74.6	74.8	75.1	75.3	75.6	75.8	76.1
.1	69.6	69.8	70.1	70.4	70.6	70.9	71.1	71.4	71.6	71.9	72.1	72.3	72.4	72.6	73.1	73.4	73.6	73.9	74.1	74.4	74.7	74.9	75.2	75.4	75.7
.2	69.2	69.5	69.7	70.0	70.2	70.5	70.7	71.0	71.2	71.5	71.7	72.0	72.2	72.5	72.7	73.0	73.2	73.5	73.7	74.0	74.2	74.5	74.7	75.0	75.2
.3	68.8	69.1	69.3	69.6	69.8	70.1	70.3	70.6	70.8	71.1	71.3	71.6	71.8	72.1	72.3	72.6	72.8	73.1	73.3	73.6	73.8	74.1	74.3	74.6	74.8
.4	68.4	68.7	68.9	69.2	69.4	69.7	69.9	70.2	70.4	70.7	70.9	71.2	71.4	71.7	71.9	72.2	72.4	72.7	72.9	73.2	73.4	73.7	73.9	74.2	74.4
.5	68.1	68.3	68.5	68.8	69.0	69.3	69.5	69.8	70.0	70.3	70.5	70.8	71.0	71.3	71.5	71.8	72.0	72.3	72.5	72.8	73.0	73.2	73.5	73.7	74.0
.6	67.7	67.9	68.2	68.4	68.7	68.9	69.2	69.4	69.6	69.9	70.1	70.4	70.6	70.9	71.1	71.4	71.6	71.9	72.1	72.4	72.6	72.8	73.1	73.3	73.6
.7	67.3	67.5	67.8	68.0	68.3	68.5	68.8	69.0	69.3	69.5	69.8	70.0	70.2	70.5	70.7	71.0	71.2	71.5	71.7	72.0	72.2	72.4	72.7	72.9	73.2
.8	66.9	67.2	67.4	67.7	67.9	68.2	68.4	68.6	68.9	69.1	69.4	69.6	69.9	70.1	70.3	70.6	70.8	71.1	71.3	71.6	71.8	72.1	72.3	72.5	72.8
.9	66.6	66.8	67.1	67.3	67.5	67.8	68.0	68.3	68.5	68.8	69.0	69.2	69.5	69.7	70.0	70.2	70.4	70.7	70.9	71.2	71.4	71.7	71.9	72.1	72.4

Pol. =	60.0	60.2	60.4	60.6	60.8	61.0	61.2	61.4	61.6	61.8	62.0	62.2	62.4	62.6	62.8	63.0	63.2	63.4	63.6	63.8	64.0	64.2	64.4	64.6	64.8
19.0	76.4	76.6	76.9	77.1	77.4	77.6	77.9	78.1	78.4	78.6	78.9	79.1	79.4	79.7	79.9	80.2	80.4	80.7	80.9	81.2	81.4	81.7	81.9	82.2	82.5
.1	75.9	76.2	76.4	76.7	76.9	77.2	77.4	77.7	77.9	78.2	78.4	78.7	79.0	79.2	79.5	79.7	80.0	80.2	80.5	80.7	81.0	81.2	81.5	81.7	82.0
.2	75.5	75.7	76.0	76.2	76.5	76.8	77.0	77.3	77.5	77.8	78.0	78.3	78.5	78.8	79.0	79.3	79.5	79.8	80.0	80.3	80.5	80.8	81.0	81.3	81.5
.3	75.1	75.3	75.6	75.8	76.1	76.3	76.6	76.8	77.1	77.3	77.6	77.8	78.1	78.3	78.6	78.8	79.1	79.3	79.6	79.8	80.1	80.3	80.6	80.8	81.1
4	74.7	74.9	75.1	75.4	75.6	75.9	76.1	76.4	76.6	76.9	77.1	77.4	77.6	77.9	78.1	78.4	78.6	78.9	79.1	79.4	79.6	79.9	80.1	80.4	80.6
.5	74.2	74.5	74.7	75.0	75.3	75.5	75.7	76.0	76.2	76.5	76.7	77.0	77.2	77.5	77.7	77.9	78.2	78.4	78.7	78.9	79.2	79.4	79.7	79.9	80.2
.6	73.8	74.1	74.3	74.6	74.8	75.1	75.3	75.6	75.8	76.0	76.3	76.5	76.8	77.0	77.3	77.5	77.8	78.0	78.3	78.5	78.8	79.0	79.2	79.5	79.7
.7	73.4	73.7	73.9	74.2	74.4	74.6	74.9	75.1	75.4	75.6	75.9	76.1	76.4	76.6	76.8	77.1	77.3	77.6	77.8	78.1	78.3	78.6	78.8	79.1	79.3
.8	73.0	73.3	73.5	73.8	74.0	74.2	74.5	74.7	75.0	75.2	75.5	75.7	75.9	76.2	76.4	76.7	76.9	77.2	77.4	77.7	77.9	78.1	78.4	78.6	78.9
.9	72.6	72.9	73.1	73.4	73.6	73.8	74.1	74.3	74.6	74.8	75.0	75.3	75.5	75.8	76.0	76.3	76.5	76.7	77.0	77.2	77.5	77.7	77.9	78.2	78.4

Pol. =	65.0	65.2	65.4	65.6	65.8	66.0	66.2	66.4	66.6	66.8	67.0	67.2	67.4	67.6	67.8	68.0	68.2	68.4	68.6	68.8	69.0	69.2	69.4	69.6	69.8
19.0	82.7	83.0	83.2	83.5	83.7	84.0	84.2	84.5	84.7	85.0	85.3	85.5	85.8	86.0	86.3	86.5	86.8	87.0	87.3	87.5	87.8	88.1	88.3	88.6	88.8
.1	82.2	82.5	82.8	83.0	83.3	83.5	83.8	84.0	84.3	84.5	84.8	85.0	85.3	85.5	85.8	86.0	86.3	86.5	86.8	87.1	87.3	87.6	87.8	88.1	88.3
.2	81.8	82.0	82.3	82.5	82.8	83.0	83.3	83.5	83.8	84.0	84.3	84.6	84.8	85.1	85.3	85.6	85.8	86.1	86.3	86.6	86.8	87.1	87.3	87.6	87.8
.3	81.3	81.6	81.8	82.1	82.3	82.6	82.8	83.1	83.3	83.6	83.8	84.1	84.3	84.6	84.8	85.1	85.3	85.6	85.8	86.1	86.3	86.6	86.8	87.1	87.3
.4	80.9	81.1	81.4	81.6	81.9	82.1	82.4	82.6	82.9	83.1	83.4	83.6	83.9	84.1	84.4	84.6	84.9	85.1	85.4	85.6	85.9	86.1	86.3	86.6	86.8
.5	80.4	80.7	80.9	81.2	81.4	81.7	81.9	82.2	82.4	82.7	82.9	83.1	83.4	83.6	83.9	84.1	84.4	84.6	84.9	85.1	85.4	85.6	85.9	86.1	86.4
.6	79.8	80.2	80.5	80.7	81.0	81.2	81.5	81.7	82.0	82.2	82.4	82.7	82.9	83.2	83.4	83.7	83.9	84.2	84.4	84.7	84.9	85.2	85.4	85.6	85.9
.7	79.5	79.8	80.0	80.3	80.5	80.8	81.0	81.3	81.5	81.7	82.0	82.2	82.5	82.7	83.0	83.2	83.5	83.7	83.9	84.2	84.4	84.7	84.9	85.2	85.4
.8	79.1	79.4	79.6	79.8	80.1	80.3	80.6	80.8	81.1	81.3	81.5	81.8	82.0	82.3	82.5	82.8	83.0	83.2	83.5	83.7	84.0	84.2	84.5	84.7	85.0
.9	78.7	78.9	79.2	79.4	79.6	79.9	80.1	80.4	80.6	80.9	81.1	81.3	81.6	81.8	82.1	82.3	82.5	82.8	83.0	83.3	83.5	83.8	84.0	84.2	84.5

TABLE 41 (*Continued*)

EXPANDED HORNE'S TABLE OF COEFFICIENTS OF PURITY

Pol. =	70.0	70.2	70.4	70.6	70.8	71.0	71.2	71.4	71.6	71.8	72.0	72.2	72.4	72.6	72.8	73.0	73.2	73.4	73.6	73.8	74.0	74.2	74.4	74.6	74.8
19.0	89.1	89.3	89.6	89.8	90.1	90.3	90.6	90.9	91.1	91.4	91.6	91.9	92.1	92.4	92.6	92.9	93.1	93.4	93.7	93.9	94.2	94.4	94.7	94.9	95.2
.1	88.6	88.8	89.1	89.3	89.6	89.8	90.1	90.3	90.6	90.8	91.1	91.3	91.6	91.9	92.1	92.4	92.6	92.9	93.1	93.4	93.6	93.9	94.1	94.4	94.6
.2	88.1	88.3	88.6	88.8	89.1	89.3	89.6	89.8	90.1	90.3	90.6	90.8	91.1	91.3	91.6	91.8	92.1	92.4	92.6	92.9	93.1	93.4	93.6	93.9	94.1
.3	87.6	87.8	88.1	88.3	88.6	88.8	89.1	89.3	89.6	89.8	90.1	90.3	90.6	90.8	91.1	91.3	91.6	91.8	92.1	92.3	92.6	92.8	93.1	93.3	93.6
.4	87.1	87.3	87.6	87.8	88.1	88.3	88.6	88.8	89.1	89.3	89.6	89.8	90.1	90.3	90.6	90.8	91.1	91.3	91.6	91.8	92.1	92.3	92.6	92.8	93.1
.5	86.6	86.9	87.1	87.4	87.6	87.8	88.1	88.3	88.6	88.8	89.1	89.3	89.6	89.8	90.1	90.3	90.6	90.8	91.1	91.3	91.6	91.8	92.1	92.3	92.6
.6	86.1	86.4	86.6	86.9	87.1	87.4	87.6	87.9	88.1	88.3	88.6	88.8	89.1	89.3	89.6	89.8	90.1	90.3	90.6	90.8	91.1	91.3	91.5	91.8	92.0
.7	85.7	85.9	86.1	86.4	86.6	86.9	87.1	87.4	87.6	87.9	88.1	88.4	88.6	88.8	89.1	89.3	89.6	89.8	90.1	90.3	90.6	90.8	91.0	91.3	91.5
.8	85.2	85.4	85.7	85.9	86.2	86.4	86.7	86.9	87.1	87.4	87.6	87.9	88.1	88.4	88.6	88.8	89.1	89.3	89.6	89.8	90.1	90.3	90.6	90.8	91.0
.9	84.7	85.0	85.2	85.5	85.7	85.9	86.2	86.4	86.7	86.9	87.1	87.4	87.6	87.9	88.1	88.4	88.6	88.8	89.1	89.3	89.6	89.8	90.1	90.3	90.5

Pol. =	75.0	75.2	75.4	75.6	75.8	76.0	76.2	76.4	76.6	76.8	77.0	77.2	77.4	77.6	77.8	78.0	78.2	78.4	78.6	78.8	79.0	79.2	79.4	79.6	79.8
19.0	95.4	95.7	95.9	96.2	96.5	96.7	97.0	97.2	97.5	97.7	98.0	98.2	98.5	98.7	99.0	99.3	99.5	99.8	100.0						
.1	94.9	95.2	95.4	95.7	95.9	96.2	96.4	96.7	96.9	97.2	97.4	97.7	97.9	98.2	98.4	98.7	98.9	99.2	99.5	99.7	100.0				
.2	94.4	94.6	94.9	95.1	95.4	95.6	95.9	96.1	96.4	96.6	96.9	97.1	97.4	97.6	97.9	98.1	98.4	98.6	98.9	99.1	99.4	99.6	99.9	100.2	
.3	93.8	94.1	94.3	94.6	94.8	95.1	95.3	95.6	95.8	96.1	96.3	96.6	96.8	97.1	97.3	97.6	97.8	98.1	98.3	98.6	98.8	99.1	99.3	99.6	99.8
.4	93.3	93.6	93.8	94.1	94.3	94.6	94.8	95.1	95.3	95.6	95.8	96.1	96.3	96.5	96.8	97.0	97.3	97.5	97.8	98.0	98.3	98.5	98.8	99.9	99.3
.5	92.8	93.0	93.3	93.5	93.8	94.0	94.3	94.5	94.8	95.0	95.3	95.5	95.8	96.0	96.3	96.5	96.8	97.0	97.3	97.5	97.7	98.0	98.2	98.5	98.7
.6	92.3	92.5	92.8	93.0	93.3	93.5	93.8	94.0	94.3	94.5	94.7	95.0	95.2	95.5	95.7	96.0	96.2	96.5	96.7	97.0	97.2	97.5	97.7	97.9	98.2
.7	91.8	92.0	92.3	92.5	92.8	93.0	93.2	93.5	93.7	94.0	94.2	94.5	94.7	95.0	95.2	95.4	95.7	95.9	96.2	96.4	96.7	96.9	97.2	97.4	97.7
.8	91.3	91.5	91.8	92.0	92.3	92.5	92.7	93.0	93.2	93.5	93.7	94.0	94.2	94.4	94.7	94.9	95.2	95.4	95.7	95.9	96.2	96.4	96.6	96.9	97.1
.9	90.8	91.0	91.3	91.5	91.7	92.0	92.2	92.5	92.7	93.0	93.2	93.4	93.7	93.9	94.2	94.4	94.7	94.9	95.1	95.4	95.6	95.9	96.1	96.3	96.6

Pol. =	80.0	80.2	80.4	80.6	80.8	81.0	81.2	81.4	81.6	81.8	82.0	82.2	82.4	84.6	84.8	83.0	83.2	83.4	83.6	83.8	84.0	84.2	84.4	84.6	84.8
19.0																									
.1																									
.2																									
.3	100.1																								
.4	99.5	99.8	100.0																						
.5	99.0	99.2	99.5	99.7	100.0																				
.6	98.4	98.7	98.9	99.2	99.4	99.7	99.9	100.2																	
.7	97.9	98.1	98.4	98.6	98.9	99.1	99.4	99.6	99.9	100.1															
.8	97.4	97.6	97.9	98.1	98.3	98.6	98.8	99.1	99.3	99.6	100.0														
.9	96.8	97.1	97.3	97.6	97.8	98.0	98.3	98.5	98.8	99.0	99.3	99.5	99.7	100.0											

TABLE 41 (*Continued*)

Expanded Horne's Table of Coefficients of Purity

Pol. =	60.0	60.2	60.4	60.6	60.8	61.0	61.2	61.4	61.6	61.8	62.0	62.2	62.4	62.6	62.8	63.0	63.2	63.4	63.6	63.8	64.0	64.2	64.4	64.6	64.8
20.0	72.2	72.5	72.7	73.0	73.2	73.4	73.7	73.9	74.2	74.4	74.6	74.9	75.1	75.4	75.6	75.8	76.1	76.3	76.6	76.8	77.0	77.3	77.5	77.8	78.0
.1	71.8	72.1	72.3	72.6	72.8	73.0	73.3	73.5	73.8	74.0	74.2	74.5	74.7	75.0	75.2	75.4	75.7	75.9	76.2	76.4	76.6	76.9	77.1	77.4	77.6
.2	71.5	71.7	71.9	72.2	72.4	72.7	72.9	73.1	73.4	73.6	73.8	74.1	74.3	74.6	74.8	75.0	75.3	75.5	75.7	76.0	76.2	76.5	76.7	76.9	77.2
.3	71.1	71.3	71.5	71.8	72.0	72.3	72.5	72.7	73.0	73.2	73.4	73.7	73.9	74.2	74.4	74.6	74.9	75.1	75.3	75.6	75.8	76.1	76.3	76.5	76.8
.4	70.7	70.9	71.2	71.4	71.6	71.9	72.1	72.4	72.6	72.8	73.1	73.3	73.5	73.8	74.0	74.2	74.5	74.7	74.9	75.2	75.4	75.7	75.9	76.1	76.4
.5	70.3	70.6	70.8	71.0	71.3	71.5	71.7	72.0	72.2	72.4	72.7	72.9	73.1	73.4	73.6	73.8	74.1	74.3	74.5	74.8	75.0	75.2	75.5	75.7	76.0
.6	70.0	70.2	70.4	70.7	70.9	71.1	71.4	71.6	71.8	72.1	72.3	72.5	72.8	73.0	73.2	73.5	73.7	73.9	74.2	74.4	74.6	74.9	75.1	75.3	75.6
.7	69.6	69.8	70.1	70.3	70.5	70.7	71.0	71.2	71.4	71.7	71.9	72.1	72.4	72.6	72.8	73.1	73.3	73.5	73.8	74.0	74.2	74.5	74.7	74.9	75.2
.8	69.2	69.5	69.7	69.9	70.2	70.4	70.6	70.8	71.1	71.3	71.5	71.8	72.0	72.2	72.5	72.7	72.9	73.2	73.4	73.6	73.8	74.1	74.3	74.5	74.8
.9	68.9	69.1	69.3	69.6	69.8	70.0	70.2	70.5	70.7	70.9	71.2	71.4	71.6	71.9	72.1	72.3	72.5	72.8	73.0	73.2	73.5	73.7	73.9	74.1	74.4

Pol. =	65.0	65.2	65.4	65.6	65.8	66.0	66.2	66.4	66.6	66.8	67.0	67.2	67.4	67.6	67.8	68.0	68.2	68.4	68.6	68.8	69.0	69.2	69.4	69.6	69.8
20.0	78.3	78.5	78.7	79.0	79.2	79.5	79.7	79.9	80.2	80.4	80.7	80.9	81.1	81.4	81.6	81.9	82.1	82.3	82.6	82.8	83.1	83.3	83.6	83.8	84.0
.1	77.8	78.1	78.3	78.5	78.8	79.0	79.3	79.5	79.7	80.0	80.2	80.5	80.7	80.9	81.2	81.4	81.7	81.9	82.1	82.4	82.6	82.9	83.1	83.3	83.6
.2	77.4	77.7	77.9	78.1	78.4	78.6	78.8	79.1	79.3	79.6	79.8	80.0	80.3	80.5	80.7	81.0	81.2	81.5	81.7	81.9	82.2	82.4	82.7	82.9	83.1
.3	77.0	77.2	77.5	77.7	77.9	78.2	78.4	78.7	78.9	79.1	79.4	79.6	79.8	80.1	80.3	80.6	80.8	81.0	81.3	81.5	81.7	82.0	82.2	82.4	82.7
.4	76.6	76.8	77.1	77.3	77.5	77.8	78.0	78.2	78.5	78.7	79.0	79.2	79.4	79.7	79.9	80.1	80.4	80.6	80.8	81.1	81.3	81.5	81.8	82.0	82.3
.5	76.2	76.4	76.7	76.9	77.1	77.4	77.6	77.8	78.1	78.3	78.5	78.8	79.0	79.2	79.5	79.7	79.9	80.2	80.4	80.6	80.9	81.1	81.3	81.6	81.8
.6	75.8	76.0	76.3	76.5	76.7	77.0	77.2	77.4	77.7	77.9	78.1	78.4	78.6	78.8	79.1	79.3	79.5	79.8	90.0	80.2	80.5	80.7	80.9	81.2	81.4
.7	75.4	75.6	75.9	76.1	76.3	76.5	76.8	77.0	77.2	77.5	77.7	77.9	78.2	78.4	78.6	78.9	79.1	79.3	79.6	79.8	80.0	80.3	80.5	80.7	81.0
.8	75.0	75.2	75.5	75.7	75.9	76.2	76.4	76.6	76.8	77.1	77.3	77.5	77.8	78.0	78.2	78.5	78.7	78.9	79.2	79.4	79.6	79.8	80.1	80.3	80.5
.9	74.6	74.8	75.0	75.3	75.5	75.8	76.0	76.2	76.4	76.7	76.9	77.1	77.4	77.6	77.8	78.1	78.3	78.5	78.7	79.0	79.2	79.4	79.7	79.9	80.1

Pol. =	70.0	70.2	70.4	70.6	70.8	71.0	71.2	71.4	71.6	71.8	72.0	72.2	72.4	72.6	72.8	73.0	73.2	73.4	73.6	73.8	74.0	74.2	74.4	74.6	74.8
20.0	84.3	84.5	84.8	85.0	85.2	85.5	85.7	86.0	86.2	86.4	86.7	86.9	87.2	87.4	87.6	87.9	88.1	88.4	88.6	88.8	89.1	89.3	89.6	89.8	90.1
.1	83.8	84.1	84.3	84.5	84.8	85.0	85.3	85.5	85.7	86.0	86.2	86.5	86.7	86.9	87.2	87.4	87.6	87.9	88.1	88.4	88.6	88.8	89.1	89.3	89.6
.2	83.4	83.6	83.8	84.1	84.3	84.6	84.8	85.0	85.3	85.5	85.8	86.0	86.2	86.5	86.7	86.9	87.2	87.4	87.7	87.9	88.1	88.4	88.6	88.8	89.1
.3	82.9	83.2	83.4	83.6	83.9	84.1	84.3	84.6	84.8	85.1	85.3	85.5	85.8	86.0	86.2	86.5	86.7	86.9	87.2	87.4	87.7	87.9	88.1	88.4	88.6
.4	82.5	82.7	83.0	83.2	83.4	83.7	83.9	84.1	84.4	84.6	84.8	85.1	85.3	85.6	85.8	86.0	86.3	86.5	86.7	87.0	87.2	87.4	87.7	87.9	88.1
.5	82.0	82.3	82.5	82.8	83.0	83.2	83.5	83.7	83.9	84.2	84.4	84.6	84.9	85.1	85.3	85.6	85.8	86.0	86.3	86.5	86.7	87.0	87.2	87.4	87.7
.6	81.6	81.9	82.1	82.3	82.6	82.8	83.0	83.3	83.5	83.7	84.0	84.2	84.4	84.7	84.9	85.1	85.4	85.6	85.8	86.1	86.3	86.5	86.8	87.0	87.2
.7	81.2	81.4	81.6	81.9	82.1	82.3	82.6	82.8	83.0	83.3	83.5	83.7	84.0	84.2	84.4	84.7	84.9	85.1	85.4	85.6	85.8	86.1	86.3	86.5	86.8
.8	80.8	81.0	81.2	81.5	81.7	81.9	82.2	82.4	82.6	82.8	83.1	83.3	83.5	83.8	84.0	84.2	84.5	84.7	84.9	85.2	85.4	85.6	85.8	86.1	86.3
.9	80.3	80.6	80.8	81.0	81.3	81.5	81.7	82.0	82.2	82.4	82.6	82.9	83.1	83.3	83.6	83.8	84.0	84.2	84.5	84.7	84.9	85.2	85.4	85.6	85.9

TABLE 41 (*Continued*)

EXPANDED HORNE'S TABLE OF COEFFICIENTS OF PURITY

Pol. =	**75.0**	**75.2**	**75.4**	**75.6**	**75.8**	**76.0**	**76.2**	**76.4**	**76.6**	**76.8**	**77.0**	**77.2**	**77.4**	**77.6**	**77.8**	**78.0**	**78.2**	**78.4**	**78.6**	**78.8**	**79.0**	**79.2**	**79.4**	**79.6**	**79.8**
20.0	90.3	90.5	90.8	91.0	91.3	91.5	91.7	92.0	92.2	92.5	92.7	92.9	93.2	93.4	93.7	93.9	94.1	94.4	94.6	94.9	95.1	95.3	95.6	95.8	96.1
.1	89.8	90.0	90.3	90.5	90.8	91.0	91.2	91.5	91.7	92.0	92.2	92.4	92.7	92.9	93.2	93.4	93.6	93.9	94.1	94.4	94.6	94.8	95.1	95.3	95.6
.2	89.3	89.6	89.8	90.0	90.3	90.5	90.8	91.0	91.2	91.5	91.7	91.9	92.2	92.4	92.7	92.9	93.1	93.4	93.6	93.9	94.1	94.3	94.6	94.8	95.0
.3	88.8	89.1	89.3	89.6	89.8	90.0	90.3	90.5	90.7	91.0	91.2	91.5	91.7	91.9	92.2	92.4	92.6	92.9	93.1	93.3	93.6	93.8	94.1	94.3	94.5
.4	88.4	88.6	88.9	89.1	89.3	89.6	89.8	90.0	90.3	90.5	90.7	91.0	91.2	91.4	91.7	91.9	92.2	92.4	92.6	92.9	93.1	93.3	93.6	93.8	94.0
.5	87.9	88.1	88.4	88.6	88.8	89.1	89.3	89.5	89.8	90.0	90.3	90.5	90.7	91.0	91.2	91.4	91.7	91.9	92.1	92.4	92.6	92.8	93.1	93.3	93.5
.6	87.5	87.7	87.9	88.1	88.3	88.6	88.8	89.1	89.3	89.5	89.8	90.0	90.2	90.5	90.7	90.9	91.2	91.4	91.6	91.9	92.1	92.3	92.6	92.8	93.0
.7	87.0	87.2	87.4	87.7	87.9	88.1	88.4	88.6	88.8	89.1	89.3	89.5	89.8	90.0	90.2	90.5	90.7	90.9	91.2	91.4	91.6	91.9	92.1	92.3	92.6
.8	86.5	86.8	87.0	87.2	87.5	87.7	87.9	88.2	88.4	88.6	88.8	89.1	89.3	89.5	89.8	90.0	90.2	90.5	90.7	90.9	91.2	91.4	91.6	91.8	92.1
.9	86.1	86.3	86.5	86.8	87.0	87.2	87.5	87.7	87.9	88.2	88.4	88.6	88.8	89.1	89.3	89.5	89.8	90.0	90.2	90.4	90.7	90.9	91.1	91.4	91.6

Pol. =	**80.0**	**80.2**	**80.4**	**80.6**	**80.8**	**81.0**	**81.2**	**81.4**	**81.6**	**81.8**	**82.0**	**82.2**	**82.4**	**82.6**	**82.8**	**83.0**	**83.2**	**83.4**	**83.6**	**83.8**	**84.0**	**84.2**	**84.4**	**84.6**	**84.8**
20.0	96.3	96.6	96.8	97.0	97.3	97.5	97.8	98.0	98.2	98.5	98.7	99.0	99.2	99.4	99.7	99.9	100.2								
.1	95.8	96.0	96.3	96.5	96.7	97.0	97.2	97.5	97.7	97.9	98.2	98.4	98.7	98.9	99.1	99.4	99.6	99.9	100.1						
.2	95.3	95.5	95.8	96.0	96.2	96.5	96.7	96.9	97.2	97.4	97.7	97.9	98.1	98.4	98.6	98.9	99.1	99.3	99.6	99.8	100.0				
.3	94.8	95.0	95.2	95.5	95.7	96.0	96.2	96.4	96.7	96.9	97.1	97.4	97.6	97.8	98.1	98.3	98.6	98.8	99.0	99.3	99.5	99.7	100.0		
.4	94.3	94.5	94.7	95.0	95.2	95.5	95.7	95.9	96.2	96.4	96.6	96.9	97.1	97.3	97.6	97.8	98.0	98.3	98.5	98.7	99.0	99.2	99.5	99.7	99.9
.5	93.8	94.0	94.2	94.5	94.7	94.9	95.2	95.4	95.6	95.9	96.1	96.3	96.6	96.8	97.0	97.3	97.5	97.8	98.0	98.2	98.5	98.7	98.9	99.2	99.4
.6	93.3	93.5	93.7	93.9	94.2	94.4	94.6	94.9	95.2	95.4	95.6	95.8	96.1	96.3	96.5	96.7	97.0	97.2	97.5	97.7	97.9	98.2	98.4	98.6	98.8
.7	92.8	93.0	93.2	93.5	93.7	93.9	94.2	94.4	94.6	94.9	95.1	95.3	95.6	95.8	96.0	96.3	96.5	96.7	97.0	97.2	97.4	97.7	97.9	98.1	98.4
.8	92.3	92.5	92.8	93.0	93.2	93.5	93.7	93.9	94.2	94.4	94.6	94.8	95.1	95.3	95.5	95.8	96.0	96.2	96.5	96.7	96.9	97.1	97.4	97.6	97.8
.9	91.8	92.1	92.3	92.5	92.7	93.0	93.2	93.4	93.7	93.9	94.1	94.3	94.5	94.8	95.0	95.3	95.5	95.7	96.0	96.2	96.4	96.6	96.9	97.1	97.3

Pol. =	**85.0**	**85.2**	**85.4**	**85.6**	**85.8**	**86.0**	**86.2**	**86.4**	**86.6**	**86.8**	**87.0**	**87.2**	**87.4**	**87.6**	**87.8**	**88.0**	**88.2**	**88.4**	**88.6**	**88.8**	**89.0**	**89.2**	**89.4**	**89.6**	**89.8**
20.0																									
.1																									
.2																									
.3																									
.4	100.2																								
.5	99.6	99.9	100.1																						
.6	99.1	99.3	99.6	99.8	100.0																				
.7	98.6	98.8	99.0	99.3	99.5	99.7	100.0																		
.8	98.1	98.3	98.5	98.8	99.0	99.2	99.5	99.7	99.9	100.1															
.9	97.6	97.8	98.0	98.3	98.5	98.7	98.9	99.2	99.4	99.6	99.9	100.1													

TABLE 42

EXPANDED MEISSL-HILLER TABLE FOR INVERT SUGAR * (E. W. RICE)

Weight of Sample in 100 Ml.			1 Gram		2 Grams		5 Grams		10 Grams		20 Grams	
Polarization			30°	35°	20°	30°	85°	95°	85°	'95°	85°	95°
Weight Obtained as												
Cu	Cu_2O	CuO										
.0999	.1125	.1250	10.28	10.26	5.13	5.12	1.661	1.600	.76	.76		
.1019	.1147	.1275	10.48	10.46	5.24	5.23	1.708	1.648	.78	.78		
.1039	.1170	.1300	10.69	10.67	5.34	5.33	1.755	1.696	.80	.80		
.1059	.1192	.1325	10.89	10.87	5.44	5.43	1.802	1.744	.82	.82		
.1070	.1215	.1350	11.10	11.08	5.54	5.53	1.849	1.792	.84	.84		
.1099	.1237	.1375	11.31	11.29	5.65	5.63	1.896	1.839	.86	.86		
.1119	.1260	.1400	11.52	11.50	5.76	5.74	1.942	1.886	.88	.88		
.1138	.1282	.1425	11.73	11.71	5.86	5.84	1.989	1.933	.90	.90		
.1158	.1305	.1450	11.94	11.92	5.96	5.95	2.036	1.980	.92	.92		
.1178	.1327	.1475	12.15	12.13	6.07	6.05	2.082	2.027	.94	.94		
.1198	.1350	.1500	12.36	12.34	6.18	6.16	2.128	2.074	.96	.96		
.1218	.1372	.1525	12.57	12.55	6.28	6.26	2.175	2.121	.98	.98		
.1238	.1395	.1550	12.78	12.76	6.38	6.36	2.221	2.168	1.00	1.00		
.1258	.1417	.1575	12.99	12.97	6.49	6.47	2.267	2.215	1.02	1.02		
.1278	.1440	.1600	13.21	13.19	6.60	6.58	2.313	2.262	1.05	1.04		
.1298	.1462	.1625	13.42	13.40	6.70	6.68	2.359	2.309	1.07	1.06		
.1318	.1485	.1650	13.64	13.62	6.81	6.79	2.405	2.356	1.09	1.08		
.1338	.1507	.1675	13.85	13.83	6.92	6.90	2.451	2.403	1.11	1.10		
.1358	.1530	.1700	14.07	14.05	7.03	7.01	2.497	2.449	1.13	1.12		
.1378	.1552	.1725	14.28	14.26	7.13	7.11	2.543	2.496	1.15	1.14		
.1398	.1575	.1750	14.50	14.48	7.24	7.22	2.589	2.543	1.17	1.16		
.1418	.1597	.1775	14.72	14.69	7.35	7.33	2.635	2.589	1.19	1.18		
.1438	.1620	.1800	14.93	14.91	7.46	7.44	2.680	2.635	1.22	1.21		

NOTE.—The fourth and fifth pages of this table have been extended to include concentrations of 1.5 grams per 100 ml. (See method for reducing sugars in molasses, p. 543.)

TABLE 42 (*Continued*)

EXPANDED MEISSL-HILLER TABLE FOR INVERT SUGAR (E. W. RICE)

Weight of Sample in 100 Ml.			1 Gram		2 Grams		5 Grams		10 Grams		20 Grams	
Polarization			30°	35°	20°	30°	85°	95°	85°	95°	85°	95°
Weight Obtained as												
Cu	Cu_2O	CuO										
.1458	.1642	.1825	15.15	15.12	7.56	7.54	2.726	2.682	1.24	1.23		
.1478	.1665	.1850	15.37	15.43	7.67	7.65	2.772	2.728	1.27	1.25		
.1498	.1687	.1875	15.59	15.56	7.78	7.76	2.817	2.774	1.29	1.27		
.1518	.1710	.1900	15.81	15.78	7.89	7.87	2.862	2.820	1.31	1.30		
.1538	.1732	.1925	16.03	16.00	7.99	7.97	2.907	2.867	1.33	1.32		
.1558	.1755	.1950	16.25	16.22	8.10	8.08	2.952	2.913	1.35	1.34		
.1578	.1777	.1975	16.47	16.44	8.21	8.19	2.997	2.959	1.37	1.36		
.1598	.1800	.2000	16.69	16.66	8.32	8.30	3.042	3.005	1.40	1.38		
.1618	.1822	.2025	16.91	16.88	8.43	8.40	3.087	3.051	1.42	1.40		
.1638	.1845	.2050	17.13	17.10	8.54	8.51	3.132	3.097	1.44	1.42		
.1658	.1867	.2075	17.35	17.32	8.65	8.62	3.177	3.143	1.46	1.44		
.1678	.1890	.2100	17.57	17.54	8.76	8.73	3.221	3.188	1.49	1.47		
.1698	.1912	.2125	17.79	17.76	8.87	8.83	3.266	3.234	1.51	1.49		
.1718	.1935	.2150	18.01	17.98	8.98	8.94	3.310	3.280	1.53	1.51	.72	
.1738	.1957	.2175	18.23	18.20	9.09	9.05	3.354	3.325	1.55	1.53	.73	
.1758	.1980	.2200	18.45	18.42	9.20	9.16	3.398	3.370	1.58	1.56	.75	
.1778	.2002	.2225	18.67	18.64	9.31	9.26	3.433	3.416	1.60	1.58	.76	
.1798	.2025	.2250	18.89	18.86	9.42	9.37	3.488	3.461	1.62	1.60	.77	
.1817	.2047	.2275	19.11	19.08	9.53	9.48	3.532	3.506	1.64	1.62	.78	
.1837	.2070	.2300	19.34	19.30	9.64	9.59	3.576	3.551	1.67	1.65	.79	
.1857	.2092	.2325	19.56	19.52	9.75	9.70	3.621	3.597	1.69	1.67	.80	
.1877	.2115	.2350	19.78	19.74	9.86	9.81	3.666	3.642	1.71	1.69	.81	
.1897	.2137	.2375	20.00	19.96	9.97	9.92	3.710	3.687	1.73	1.72	.82	.81
.1917	.2160	.2400	20.23	20.19	10.08	10.03	3.754	3.732	1.76	1.75	.83	.83
.1937	.2182	.2425	20.45	20.41	10.19	10.14	3.799	3.777	1.78	1.77	.84	.84
.1957	.2205	.2450	20.67	20.63	10.30	10.25	3.844	3.822	1.80	1.79	.85	.85

TABLE 42 (*Continued*)

EXPANDED MEISSL-HILLER TABLE FOR INVERT SUGAR (E. W. RICE)

Weight of Sample in 100 Ml.			1 Gram		2 Grams		5 Grams		10 Grams		20 Grams	
Polarization			30°	35°	20°	30°	85°	95°	85°	95°	85°	95°
Weight Obtained as												
Cu	Cu_2O	CuO										
.1977	.2227	.2475	20.89	20.85	10.41	10.36	3.886	3.867	1.83	1.81	.86	.86
.1997	.2250	.2500	21.12	21.08	10.52	10.47	3.932	3.912	1.86	1.84	.88	.87
.2017	.2272	.2525	21.34	21.30	10.63	10.58	3.977	3.957	1.88	1.86	.89	.88
.2037	.2295	.2550	21.56	21.52	10.74	10.69	4.022	4.002	1.90	1.88	.90	.89
.2057	.2317	.2575	21.78	21.74	10.85	10.80	4.066	4.047	1.92	1.90	.91	.90
.2077	.2340	.2600	22.00	21.96	10.96	10.91	4.110	4.092	1.95	1.93	.92	.91
.2097	.2362	.2625	22.22	22.18	11.07	11.02	4.155	4.137	1.97	1.95	.93	.92
.2117	.2385	.2650	22.44	22.40	11.18	11.13	4.200	4.183	1.99	1.97	.94	.93
.2137	.2407	.2675	22.66	22.62	11.29	11.24	4.244	4.227	2.02	1.99	.95	.94
.2157	.2430	.2700	22.89	22.85	11.40	11.35	4.288	4.271	2.05	2.02	.97	.96
.2177	.2452	.2725	23.11	23.07	11.51	11.46	4.333	4.316	2.07	2.04	.98	.97
.2197	.2475	.2750	23.33	23.29	11.62	11.57	4.378	4.361	2.09	2.06	.99	.98
.2217	.2497	.2775	23.55	23.51	11.73	11.68	4.422	4.405	2.11	2.08	1.00	.99
.2237	.2520	.2800	23.78	23.74	11.84	11.79	4.466	4.449	2.14	2.12	1.01	1.00
.2257	.2542	.2825	24.00	23.96	11.95	11.90	4.511	4.494	2.16	2.14	1.02	1.01
.2277	.2565	.2850	24.22	24.18	12.06	12.01	4.556	4.538	2.18	2.16	1.03	1.02
.2297	.2587	.2875	24.44	24.40	12.17	12.12	4.600	4.582	2.20	2.18	1.04	1.03
.2317	.2610	.2900	24.67	24.63	12.28	12.23	4.644	4.626	2.23	2.21	1.06	1.05
.2337	.2633	.2925	24.89	24.85	12.39	12.34	4.689	4.671	2.25	2.23	1.07	1.06
.2357	.2655	.2950	25.11	25.07	12.50	12.45	4.734	4.716	2.27	2.25	1.08	1.07
.2377	.2677	.2975	25.33	25.29	12.61	12.56	4.779	4.761	2.29	2.27	1.09	1.08
.2397	.2700	.3000	25.56	25.52	12.73	12.67	4.823	4.805	2.32	2.30	1.10	1.09
.2417	.2722	.3025	25.78	25.74	12.84	12.78	4.868	4.850	2.34	2.32	1.11	1.10
.2437	.2745	.3050	26.00	25.96	12.95	12.89	4.913	4.895	2.36	2.34	1.12	1.11
.2457	.2767	.3075	26.22	26.18	13.06	13.00	4.958	4.940	2.39	2.37	1.13	1.12
.2477	.2790	.3100	26.45	26.41	13.18	13.11	5.003	4.985	2.42	2.40	1.15	1.14

TABLE 42 (*Continued*)

EXPANDED MEISSL-HILLER TABLE FOR INVERT SUGAR (E. W. RICE)

Wt. of Sample in 100 Ml.		1 Gram		1.5 Grams		2 Grams		5 Grams		10 Grams		20 Grams	
Polarization		30°	35°	25°	35°	20°	30°	85°	95°	85°	95°	85°	95°
Wt. Obtained as													
Cu	CuO												
.2497	.3125	26.67	26.63	17.71	17.64	13.29	13.22	5.046	5.031	2.44	2.42	1.16	1.15
.2517	.3150	26.90	26.85	17.85	17.79	13.40	13.33	5.095	5.076	2.46	2.44	1.17	1.16
.2537	.3175	27.12	27.07	18.01	17.94	13.51	13.44	5.141	5.121	2.48	2.46	1.18	1.17
.2556	.3200	27.35	27.30	18.16	18.08	13.63	13.56	5.186	5.166	2.51	2.49	1.19	1.18
.2576	.3225	27.57	27.52	18.31	18.23	13.74	13.67	5.232	5.212	2.53	2.51	1.20	1.19
.2596	.3250	27.80	27.75	18.46	18.38	13.85	13.78	5.278	5.258	2.55	2.53	1.21	1.20
.2616	.3275	28.02	27.97	18.62	18.54	13.96	13.89	5.324	5.303	2.57	2.55	1.22	1.21
.2636	.3300	28.25	28.20	18.77	18.69	14.08	14.01	5.371	5.348	2.60	2.58	1.24	1.23
.2656	.3325	28.47	28.42	18.92	18.84	14.19	14.12	5.418	5.394	2.62	2.60	1.25	1.24
.2676	.3350	28.70	28.65	19.07	18.99	14.30	14.23	5.465	5.440	2.64	2.62	1.26	1.25
.2696	.3375	28.93	28.87	19.22	19.14	14.42	14.35	5.512	5.486	2.66	2.64	1.27	1.26
.2716	.3400	29.16	29.10	19.38	19.30	14.54	14.47	5.558	5.532	2.69	2.67	1.28	1.27
.2736	.3425	29.39	29.32	19.53	19.45	14.65	14.59	5.605	5.578	2.71	2.69	1.29	1.28
.2756	.3450	29.62	29.55	19.69	19.61	14.76	14.70	5.652	5.624	2.73	2.71	1.30	1.29
.2776	.3475	29.85	29.77	19.85	19.77	14.88	14.81	5.699	5.671	2.75	2.73	1.31	1.30
.2796	.3500	30.08	30.00	20.00	19.92	15.00	14.93	5.746	5.718	2.78	2.76	1.33	1.32
.2816	.3525	30.31	30.23	20.16	20.08	15.11	15.04	5.793	5.765	2.80	2.78	1.34	1.33
.2836	.3550	30.54	30.46	20.31	20.23	15.22	15.15	5.840	5.812	2.82	2.80	1.35	1.34
.2856	.3575	30.77	30.69	20.47	20.39	15.34	15.27	5.888	5.859	2.84	2.82	1.36	1.35
.2876	.3600	31.00	30.93	20.61	20.53	15.46	15.39	5.936	5.906	2.87	2.85	1.37	1.36
.2896	.3625	31.23	31.16	20.76	20.68	15.57	15.50	5.983	5.953	2.89	2.87	1.38	1.37
.2916	.3650	31.46	31.40	20.92	20.84	15.69	15.61	6.031	6.000	2.91	2.88	1.39	1.38
.2936	.3675	31.69	31.63	21.07	20.99	15.81	15.73	6.079	6.048	2.93	2.91	1.40	1.39
.2956	.3700	31.93	31.87	21.23	21.15	15.93	15.85	6.127	6.096	2.96	2.94	1.42	1.41
.2976	.3725	32.16	32.10	21.38	21.30	16.04	15.96	6.174	6.144	2.98	2.96	1.43	1.42
.2996	.3750	32.40	32.34	21.53	21.45	16.16	16.08	6.222	6.192	3.00	2.98	1.44	1.43

TABLE 42 (*Continued*)

EXPANDED MEISSL-HILLER TABLE FOR INVERT SUGAR. (E. W. RICE)

Wt. of Sample in 100 Ml.		1 Gram		1.5 Grams		2 Grams		5 Grams		10 Grams		20 Grams	
Polarization		30°	35°	25°	35°	20°	30°	85°	95°	85°	95°	85°	95°
Wt. Obtained as													
Cu	CuO												
.3016	.3775	32.63	32.57	21.69	21.61	16.28	16.20	6.270	6.240	3.03	3.00	1.45	1.44
.3036	.3800	32.87	32.81	21.85	21.77	16.40	16.32	6.318	6.288	3.06	3.03	1.46	1.45
.3056	.3825	33.10	33.04	22.00	21.93	16.52	16.44	6.366	6.337	3.08	3.05	1.47	1.47
.3076	.3850	33.34	33.28	22.16	22.09	16.64	16.56	6.414	6.386	3.10	3.07	1.48	1.48
.3096	.3875	33.58	33.52	22.32	22.25	16.76	16.68	6.462	6.434	3.12	3.09	1.49	1.49
.3116	.3900	33.82	33.76	22.48	22.41	16.88	16.80	6.510	6.482	3.15	3.12	1.51	1.50
.3136	.3925	34.00	34.00	22.64	22.57	17.00	16.92	6.558	6.531	3.17	3.14	1.52	1.51
.3156	.3950	34.30	34.24	22.81	22.73	17.12	17.04	6.608	6.580	3.19	3.16	1.53	1.52
.3176	.3975	34.54	34.48	22.97	22.89	17.24	17.16	6.654	6.629	3.21	3.18	1.54	1.53
.3196	.4000	34.78	34.72	23.13	23.05	17.36	17.28	6.703	6.678	3.24	3.21	1.55	1.54
.3216	.4025	35.02	34.96	23.29	23.21	17.48	17.40	6.751	6.727	3.26	3.23	1.56	1.55
.3236	.4050	35.26	35.20	23.45	23.38	17.60	17.52	6.799	6.776	3.28	3.26	1.57	1.56
.3256	.4075	35.50	35.44	23.62	23.54	17.72	17.64	6.848	6.825	3.30	3.27	1.58	1.57
.3275	.4100	35.75	35.68	23.77	23.70	17.84	17.76	6.897	6.875	3.33	3.30	1.60	1.59
.3295	.4125	35.99	35.92	23.93	23.86	17.96	17.88	6.945	6.924	3.35	3.32	1.61	1.60
.3315	.4150	36.24	36.16	24.09	24.02	18.08	18.00	6.993	6.973	3.37	3.34	1.62	1.61
.3335	.4175	36.48	36.40	24.25	24.18	18.20	18.12	7.042	7.023	3.39	3.36	1.63	1.62
.3355	.4200	36.73	36.65	24.42	24.35	18.33	18.25	7.091	7.073	3.42	3.39	1.64	1.63
.3375	.4225	36.97	36.89	24.58	24.51	18.45	18.37	7.139	7.122	3.44	3.41	1.65	1.64
.3395	.4250	37.22	37.13	24.74	24.67	18.57	18.49	7.188	7.172	3.46	3.43	1.66	1.65
.3415	.4275	37.47	37.37	24.90	24.83	18.69	18.61	7.237	7.222	3.48	3.45	1.67	1.66
.3435	.4300	37.72	37.62	25.07	25.00	18.82	18.74	7.286	7.272	3.51	3.48	1.69	1.68
.3455	.4325	37.96	37.86	25.23	25.16	18.94	18.86	7.334	7.321	3.53	3.50	1.70	1.69
.3475	.4350	38.21	38.10	25.40	25.33	19.06	18.99	7.383	7.371	3.55	3.52	1.71	1.70
.3495	.4375	38.46	38.44	25.56	25.49	19.19	19.12	7.432	7.421	3.58	3.55	1.72	1.71

TABLE 43

Expanded Meissl-Hiller Table for Invert Sugar in Cane Juice

Grams Juice in in 100 Ml.		10			20			30			40		
Polarization		10	14	18	10	14	18	10	14	18	10	14	18
Cu Gram	CuO Gram	Percentages of Invert Sugar											
.150	.1877	1.55	1.55	1.52	0.75	0.73	0.72	0.49	0.46	0.46	0.36	0.35	0.33
.155	.1939	1.61	1.60	1.58	0.78	0.76	0.75	0.51	0.50	0.49	0.37	0.36	0.34
.160	.2003	1.66	1.65	1.63	0.81	0.78	0.77	0.52	0.51	0.50	0.38	0.37	0.35
.165	.2065	1.71	1.70	1.69	0.83	0.82	0.80	0.54	0.53	0.52	0.39	0.38	0.36
.170	.2128	1.76	1.76	1.75	0.86	0.85	0.82	0.56	0.55	0.53	0.41	0.40	0.38
.175	.2191	1.82	1.81	1.80	0.89	0.87	0.84	0.58	0.57	0.55	0.43	0.42	0.39
.180	.2252	1.87	1.87	1.85	0.92	0.90	0.87	0.60	0.58	0.57	0.44	0.43	0.40
.185	.2315	1.93	1.92	1.91	0.94	0.93	0.90	0.63	0.60	0.59	0.45	0.44	0.41
.190	.2378	1.98	1.98	1.98	0.97	0.95	0.93	0.64	0.62	0.60	0.47	0.45	0.43
.195	.2440	2.03	2.03	2.03	1.00	0.98	0.96	0.65	0.64	0.62	0.48	0.46	0.45
.200	.2503	2.08	2.08	2.08	1.03	1.01	0.99	0.67	0.66	0.65	0.49	0.48	0.48
.205	.2566	....	....	....	1.06	1.04	1.03	0.69	0.68	0.67	0.51	0.50	0.49
.210	.2628	....	....	....	1.09	1.07	1.06	0.71	0.69	0.69	0.52	0.52	0.50
.215	.2690	....	....	....	1.12	1.10	1.09	0.73	0.71	0.70	0.54	0.53	0.51
.220	.2754	....	....	....	1.15	1.12	1.11	0.74	0.72	0.72	0.55	0.54	0.53
.225	.2817	....	....	....	1.18	1.14	1.13	0.76	0.74	0.74	0.56	0.55	0.54
.230	.2879	....	....	....	1.21	1.18	1.16	0.78	0.77	0.76	0.57	0.57	0.56
.235	.2942	....	....	....	1.24	1.21	1.19	0.80	0.79	0.78	0.59	0.59	0.57
.240	.3004	....	....	....	1.26	1.24	1.22	0.82	0.82	0.80	0.61	0.60	0.58
.245	.3066	....	....	....	1.29	1.27	1.25	0.84	0.84	0.82	0.62	0.61	0.60
.250	.3129	....	....	....	1.32	1.30	1.28	0.86	0.85	0.83	0.63	0.62	0.61
.255	.3192	....	....	....	1.35	1.32	1.31	0.88	0.87	0.85	0.65	0.64	0.62
.260	.3254	....	....	....	1.37	1.34	1.34	0.90	0.88	0.88	0.66	0.65	0.63
.265	.3317	....	....	....	1.40	1.37	1.36	0.92	0.90	0.90	0.68	0.67	0.65
.270	.3379	....	....	....	1.43	1.41	1.39	0.93	0.92	0.91	0.69	0.68	0.68
.275	.3441	....	....	....	1.46	1.44	1.42	0.95	0.94	0.93	0.71	0.69	0.69
.280	.3504	....	....	....	1.48	1.47	1.45	0.97	0.96	0.94	0.72	0.70	0.70
.285	.3568	....	....	....	1.51	1.50	1.48	0.99	0.98	0.96	0.74	0.72	0.71
.290	.3630	....	....	....	1.54	1.52	1.51	1.01	0.99	0.99	0.76	0.74	0.73
.295	.3692	....	....	....	1.57	1.55	1.54	1.03	1.01	1.01	0.77	0.76	0.75
.300	.3755	....	....	....	1.59	1.58	1.56	1.05	1.03	1.02	0.78	0.77	0.76

TABLE 44

Expanded Meissl-Hiller Table for Invert Sugar in Cane Sirup

Grams Sirup in 100 Ml.		4				6				10			
Polarization		45.0	50.0	55.0	60.0	45.0	50.0	55.0	60.0	45.0	50.0	55.0	60.0
Cu Gram	CuO Gram	Percentages of Invert Sugar											
.150	.1877	3.79	3.77	3.75	3.73	2.46	2.43	2.41	2.39	1.40	1.38	1.36	1.34
.155	.1939	3.93	3.91	3.89	3.87	2.56	2.53	2.51	2.49	1.46	1.44	1.42	1.40
.160	.2003	4.07	4.04	4.02	4.00	2.64	2.62	2.59	2.57	1.51	1.49	1.48	1.45
.165	.2065	4.21	4.18	4.16	4.14	2.74	2.72	2.69	2.67	1.57	1.55	1.54	1.51
.170	.2128	4.35	4.32	4.30	4.27	2.83	2.80	2.78	2.76	1.62	1.61	1.59	1.57
.175	.2191	4.49	4.46	4.44	4.41	2.93	2.90	2.88	2.86	1.68	1.67	1.65	1.63
.180	.2252	4.63	4.60	4.57	4.55	3.01	2.99	2.96	2.94	1.74	1.72	1.70	1.69
.185	.2315	4.77	4.74	4.71	4.69	3.11	3.09	3.06	3.04	1.80	1.78	1.76	1.75
.190	.2378	4.92	4.88	4.85	4.83	3.20	3.18	3.15	3.12	1.85	1.83	1.82	1.80
.195	.2440	5.06	5.02	4.99	4.97	3.30	3.28	3.25	3.22	1.91	1.89	1.88	1.86
.200	.2503	5.21	5.17	5.13	5.11	3.38	3.36	3.34	3.31	1.96	1.95	1.93	1.92
.205	.2566	5.35	5.31	5.27	5.25	3.48	3.46	3.44	3.41	2.02	2.01	1.99	1.98
.210	.2628	5.48	5.46	5.42	5.39	3.56	3.54	3.53	3.50	2.07	2.06	2.05	2.03
.215	.2690	5.62	5.60	5.56	5.53	3.66	3.64	3.63	3.60	2.13	2.12	2.11	2.09
.220	.2754	5.75	5.75	5.70	5.67	3.75	3.73	3.71	3.69	2.18	2.17	2.16	2.15
.225	.2817	5.89	5.89	5.84	5.81	3.85	3.83	3.81	3.79	2.24	2.23	2.22	2.21
.230	.2879	6.03	6.03	5.99	5.96	3.94	3.91	3.89	3.88	2.29	2.28	2.27	2.26
.235	.2942	6.17	6.17	6.13	6.10	4.04	4.01	3.99	3.98	2.35	2.34	2.33	2.32
.240	.3004	6.30	6.30	6.29	6.25	4.13	4.10	4.08	4.06	2.41	2.40	2.39	2.38
.245	.3066	6.44	6.44	6.43	6.39	4.23	4.20	4.18	4.16	2.47	2.46	2.45	2.44
.250	.3129	6.58	6.58	6.58	6.54	4.34	4.29	4.27	4.25	2.52	2.51	2.50	2.49
.255	.3192	6.72	6.72	6.72	6.68	4.44	4.39	4.37	4.35	2.58	2.57	2.56	2.55
.260	.3254	6.86	6.85	6.85	6.84	4.52	4.48	4.46	4.44	2.63	2.62	2.61	2.60
.265	.3317	7.00	6.99	6.99	6.98	4.62	4.58	4.56	4.54	2.69	2.68	2.67	2.66
.270	.3379	7.13	7.13	7.13	7.13	4.71	4.67	4.65	4.64	2.75	2.74	2.73	2.72
.275	.3441	7.27	7.27	7.27	7.27	4.81	4.77	4.75	4.74	2.81	2.80	2.79	2.78
.280	.3504	7.41	7.41	7.41	7.41	4.91	4.87	4.84	4.83	2.87	2.85	2.84	2.83
.285	.3568	7.55	7.55	7.55	7.55	5.01	4.97	4.94	4.93	2.93	2.91	2.90	2.89
.290	.3630	7.70	7.69	7.69	7.69	5.11	5.07	5.03	5.02	2.98	2.97	2.96	2.95
.295	.3692	7.84	7.83	7.83	7.83	5.21	5.17	5.13	5.12	3.04	3.03	3.02	3.01
.300	.3755	7.98	7.97	7.97	7.97	5.31	5.27	5.23	5.21	3.10	3.09	3.08	3.07

TABLE 45

Herzfeld's Table for Determining Invert Sugar in Raw Sugars

(Invert sugar not to exceed 1.5 per cent)

Copper (Cu), Mgs.	Invert Sugar, Per Cent	Copper (Cu), Mgs.	Invert Sugar, Per Cent	Copper (Cu), Mgs.	Invert Sugar, Per Cent	Copper (Cu), Mgs.	Invert Sugar, Per Cent	Copper (Cu), Mgs.	Invert Sugar, Per Cent
50	0.050	105	0.325	160	0.621	215	0.929	270	1.242
51	0.054	106	0.330	161	0.627	216	0.935	271	1.248
52	0.058	107	0.335	162	0.633	217	0.940	272	1.253
53	0.062	108	0.340	163	0.639	218	0.946	273	1.259
54	0.066	109	0.346	164	0.645	219	0.951	274	1.265
55	0.070	110	0.351	165	0.651	220	0.957	275	1.271
56	0.074	111	0.356	166	0.657	221	0.962	276	1.276
57	0.078	112	0.361	167	0.663	222	0.968	277	1.282
58	0.082	113	0.366	168	0.669	223	0.973	278	1.288
59	0.086	114	0.371	169	0.675	224	0.979	279	1.294
60	0.090	115	0.376	170	0.680	225	0.984	280	1.299
61	0.094	116	0.381	171	0.686	226	0.990	281	1.305
62	0.098	117	0.386	172	0.692	227	0.996	282	1.311
63	0.103	118	0.392	173	0.698	228	1.001	283	1.317
64	0.108	119	0.397	174	0.704	229	1.007	284	1.322
65	0.113	120	0.402	175	0.709	230	1.013	285	1.328
66	0.118	121	0.407	176	0.715	231	1.018	286	1.334
67	0.123	122	0.412	177	0.720	232	1.024	287	1.339
68	0.128	123	0.417	178	0.726	233	1.030	288	1.345
69	0.133	124	0.423	179	0.731	234	1.036	289	1.351
70	0.138	125	0.428	180	0.737	235	1.041	290	1.357
71	0.143	126	0.433	181	0.742	236	1.047	291	1.362
72	0.148	127	0.438	182	0.748	237	1.053	292	1.368
73	0.152	128	0.443	183	0.753	238	1.058	293	1.374
74	0.157	129	0.448	184	0.759	239	1.064	294	1.380
75	0.162	130	0.453	185	0.764	240	1.070	295	1.385
76	0.167	131	0.458	186	0.770	241	1.076	296	1.391
77	0.172	132	0.463	187	0.775	242	1.081	297	1.397
78	0.177	133	0.468	188	0.781	243	1.087	298	1.403
79	0.182	134	0.473	189	0.786	244	1.093	299	1.408
80	0.187	135	0.478	190	0.792	245	1.099	300	1.414
81	0.192	136	0.483	191	0.797	246	1.104	301	1.420
82	0.197	137	0.488	192	0.803	247	1.110	302	1.425
83	0.202	138	0.493	193	0.808	248	1.116	303	1.431
84	0.208	139	0.498	194	0.814	249	1.122	304	1.437
85	0.213	140	0.503	195	0.819	250	1.127	305	1.443
86	0.219	141	0.509	196	0.825	251	1.133	306	1.448
87	0.225	142	0.515	197	0.830	252	1.139	307	1.454
88	0.231	143	0.521	198	0.836	253	1.144	308	1.460
89	0.236	144	0.527	199	0.841	254	1.150	309	1.466
90	0.242	145	0.533	200	0.847	255	1.156	310	1.471
91	0.248	146	0.538	201	0.852	256	1.162	311	1.477
92	0.254	147	0.544	202	0.858	257	1.167	312	1.483
93	0.260	148	0.550	203	0.863	258	1.173	313	1.489
94	0.265	149	0.556	204	0.869	259	1.179	314	1.494
95	0.271	150	0.562	205	0.874	260	1.185	315	1.500
96	0.277	151	0.568	206	0.880	261	1.190		
97	0.283	152	0.574	207	0.885	262	1.196		
98	0.288	153	0.580	208	0.891	263	1.202		
99	0.294	154	0.586	209	0.896	264	1.207		
100	0.300	155	0.592	210	0.902	265	1.213		
101	0.305	156	0.598	211	0.907	266	1.219		
102	0.310	157	0.604	212	0.913	267	1.225		
103	0.315	158	0.609	213	0.918	268	1.231		
104	0.320	159	0.615	214	0.924	269	1.236		

TABLE 46

Condensed Munson and Walker's Table for Invert Sugar Alone and Invert Sugar in the Presence of Sucrose *

(Expressed in Milligrams)

Copper	Invert Sugar	Invert Sugar and Sucrose			Copper	Invert Sugar	Invert Sugar and Sucrose		
		0.3 gram of total sugar	0.4 gram of total sugar	2.0 gram of total sugar			0.3 gram of total sugar	0.4 gram of total sugar	2.0 gram of total sugar
10	5.2	3.2	2.9	.....	235	124.9	124.0	123.5	117.9
15	7.7	5.8	5.4	.....	240	127.8	126.8	126.3	120.7
20	10.2	8.3	7.9	1.9	245	130.6	129.6	129.2	123.6
25	12.8	10.9	10.5	4.4	250	133.4	132.5	132.0	126.5
30	15.3	13.4	13.0	7.0	255	136.3	135.3	134.9	129.4
35	17.8	16.0	15.6	9.5	260	139.1	138.2	137.8	132.3
40	20.4	18.6	18.2	12.1	265	142.0	141.1	140.7	135.2
45	22.9	21.2	20.7	14.7	270	144.8	144.0	143.5	138.1
50	25.5	23.8	23.3	17.3	275	147.7	146.8	146.4	141.0
55	28.1	26.3	25.9	19.9	280	150.6	149.7	149.3	143.9
60	30.6	28.9	28.5	22.5	285	153.5	152.6	152.2	146.9
65	33.2	31.6	31.1	25.1	290	156.4	155.5	155.2	149.8
70	35.8	34.2	33.7	27.7	295	159.3	158.5	158.1	152.8
75	38.4	36.8	36.3	30.3	300	162.2	161.4	161.0	155.7
80	41.0	39.4	38.9	32.9	305	165.1	164.3	164.0	158.7
85	43.6	42.0	41.6	35.5	310	168.0	167.2	166.9	161.7
90	46.2	44.7	44.2	38.2	315	171.0	170.2	169.9	164.7
95	48.8	47.3	46.8	40.8	320	173.9	173.1	172.8	167.7
100	51.5	50.0	49.5	43.5	325	176.9	176.1	175.8	170.7
105	54.1	52.6	52.1	46.1	330	179.8	179.1	178.8	173.7
110	56.7	55.3	54.8	48.8	335	182.8	182.1	181.8	176.7
115	59.4	57.9	57.4	51.5	340	185.8	185.0	184.8	179.8
120	62.0	60.6	60.1	54.1	345	188.8	188.0	187.8	182.8
125	64.7	63.3	62.8	56.8	350	191.8	191.0	190.8	185.9
130	67.4	66.0	65.5	59.5	355	194.8	194.0	193.8	188.9
135	70.0	68.7	68.2	62.2	360	197.8	197.1	196.9	192.0
140	72.7	71.4	70.9	64.9	365	200.8	200.1	199.9	195.1
145	75.4	74.1	73.6	67.7	370	203.8	203.1	203.0	198.2
150	78.1	76.8	76.3	70.4	375	206.9	206.2	206.0	201.3
155	80.8	79.5	79.0	73.1	380	209.9	209.2	209.1	204.4
160	83.5	82.2	81.7	75.9	385	213.0	212.3	212.2	207.5
165	86.2	85.0	84.5	78.6	390	216.0	215.4	215.3	210.6
170	88.9	87.7	87.2	81.4	395	219.1	218.5	218.4	213.8
175	91.7	90.5	90.0	84.1	400	222.2	221.5	221.5	217.0
180	94.4	93.2	92.7	86.9	405	225.4	224.7	224.7	220.1
185	97.1	96.0	95.5	89.7	410	228.5	227.8	227.8	223.3
190	99.9	98.7	98.2	92.4	415	231.7	231.0	231.0	226.6
195	102.6	101.5	101.0	95.2	420	234.8	234.2	234.2	229.8
200	105.4	104.3	103.8	98.0	425	238.1	237.5	237.5	233.1
205	108.2	107.1	106.6	100.9	430	241.5	240.9	241.0	236.5
210	110.9	109.9	109.4	103.7	435	245.6	245.1	245.1	240.4
215	113.7	112.7	112.2	106.5					
220	116.5	115.5	115.0	109.3					
225	119.3	118.3	117.8	112.2					
230	122.1	121.1	120.7	115.0					

* Values as redetermined by Hammond (*Bur. Standards Circ. C 440* (1942), p. 564)

TABLE 47

INVERT SUGAR TABLE FOR 10 ML. OF FEHLING'S SOLUTION

(Eynon and Lane Method)

Solutions containing, besides invert sugar

Ml. of Sugar Solution Required	No Sucrose		1 Gm. Sucrose per 100 Ml.		5 Gm. Sucrose per 100 Ml.		10 Gm. Sucrose per 100 Ml.		25 Gm. Sucrose per 100 Ml.	
	Invert Sugar Factor *	Mg. Invert Sugar per 100 Ml.	Invert Sugar Factor *	Mg. Invert Sugar per 100 Ml.	Invert Sugar Factor *	Mg. Invert Sugar per 100 Ml.	Invert Sugar Factor *	Mg. Invert Sugar per 100 Ml.	Invert Sugar Factor *	Mg. Invert Sugar per 100 Ml.
15	50.5	336	49.9	333	47.6	317	46.1	307	43.4	289
16	50.6	316	50.0	312	47.6	297	46.1	288	43.4	271
17	50.7	298	50.1	295	47.6	280	46.1	271	43.4	255
18	50.8	282	50.1	278	47.6	264	46.1	256	43.3	240
19	50.8	267	50.2	264	47.6	250	46.1	243	43.3	227
20	50.9	254.5	50.2	251.0	47.6	238.0	46.1	230.5	43.2	216
21	51.0	242.9	50.2	239.0	47.6	226.7	46.1	219.5	43.2	206
22	51.0	231.8	50.3	228.2	47.6	216.4	46.1	209.5	43.1	196
23	51.1	222.2	50.3	218.7	47.6	207.0	46.1	200.4	43.0	187
24	51.2	213.3	50.3	209.8	47.6	198.3	46.1	192.1	42.9	179
25	51.2	204.8	50.4	201.6	47.6	190.4	46.0	184.0	42.8	171
26	51.3	197.4	50.4	193.8	47.6	183.1	46.0	176.9	42.8	164
27	51.4	190.4	50.4	186.7	47.6	176.4	46.0	170.4	42.7	158
28	51.4	183.7	50.5	180.2	47.7	170.3	46.0	164.3	42.7	152
29	51.5	177.6	50.5	174.1	47.7	164.5	46.0	158.6	42.6	147
30	51.5	171.7	50.5	168.3	47.7	159.0	46.0	153.3	42.5	142
31	51.6	166.3	50.6	163.1	47.7	153.9	45.9	148.1	42.5	137
32	51.6	161.2	50.6	158.1	47.7	149.1	45.9	143.4	42.4	132
33	51.7	156.6	50.6	153.3	47.7	144.5	45.9	139.1	42.3	128
34	51.7	152.2	50.6	148.9	47.7	140.3	45.8	134.9	42.2	124
35	51.8	147.9	50.7	144.7	47.7	136.3	45.8	130.9	42.2	121
36	51.8	143.9	50.7	140.7	47.7	132.5	45.8	127.1	42.1	117
37	51.9	140.2	50.7	137.0	47.7	128.9	45.7	123.5	42.0	114
38	51.9	136.6	50.7	135.5	47.7	125.5	45.7	120.3	42.0	111
39	52.0	133.3	50.8	130.2	47.7	122.3	45.7	117.1	41.9	107
40	52.0	130.1	50.8	127.0	47.7	119.2	45.6	114.1	41.8	104
41	52.1	127.1	50.8	123.9	47.7	116.3	45.6	111.2	41.8	102
42	52.1	124.2	50.8	121.0	47.7	113.5	45.6	108.5	41.7	99
43	52.2	121.4	50.8	118.2	47.7	110.9	45.5	105.8	41.6	97
44	52.2	118.7	50.9	115.6	47.7	108.4	45.5	103.4	41.5	94

* Mg. of invert sugar corresponding to 10 ml. of Fehling's solution.

TABLE 48

INVERT SUGAR TABLE FOR 25 ML. OF FEHLING'S SOLUTION

(Eynon and Lane Method)

Solutions containing, besides invert sugar

Ml. of Sugar Solution Required	No Sucrose		1 Gm. Sucrose per 100 Ml.	
	Invert Sugar Factor *	Mg. Invert Sugar per 100 Ml.	1 Gm. Invert Sugar Factor *	Mg. Invert Sugar per 100 Ml.
15	123.6	824	122.6	817
16	123.6	772	122.7	767
17	123.6	727	122.7	721
18	123.7	687	122.7	682
19	123.7	651	122.8	646
20	123.8	619.0	122.8	614.0
21	123.8	589.5	122.8	584.8
22	123.9	563.2	122.9	558.2
23	123.9	538.7	122.9	534.0
24	124.0	516.7	122.9	512.1
25	124.0	496.0	123.0	492.0
26	124.1	477.3	123.0	473.1
27	124.1	459.7	123.0	455.6
28	124.2	443.6	123.1	439.6
29	124.2	428.3	123.1	424.4
30	124.3	414.3	123.1	410.4
31	124.3	401.0	123.2	397.4
32	124.4	388.7	123.2	385.0
33	124.4	377.0	123.2	373.4
34	124.5	366.2	123.3	362.6
35	124.5	355.8	123.3	352.3
36	124.6	346.1	123.3	342.5
37	124.6	336.8	123.4	333.5
38	124.7	328.1	123.4	324.7
39	124.7	319.7	123.4	316.4
40	124.8	311.9	123.4	308.6
41	124.8	304.4	123.5	301.2
42	124.9	297.3	123.5	294.1
43	124.9	290.5	123.5	287.3
44	125.0	284.1	123.6	280.9
45	125.0	277.9	123.6	274.7
46	125.1	272.0	123.6	268.7
47	125.1	266.3	123.7	263.1
48	125.2	260.8	123.7	257.7
49	125.2	255.5	123.7	252.5
50	125.3	250.6	123.8	247.6

* Mg. of invert sugar corresponding to 25 ml. of Fehling's solution.

TABLE 49

Jackson-Gillis Clerget Method IV*

Column No. 1 is the algebraic sum of $P-P'$ corrected to normality. Column No. 2 is the Clerget Divisor corresponding to that value of $P-P'$ Apply temperature correction for exact temperature at which the invert polarization was read. Corrected Clerget Divisor divided into $100(P-P') =$ Clerget Sucrose.

CLERGET DIVISOR

No. 1	No. 2	No. 1	No. 2	No. 1	No. 2	No. 1	No. 2
5	131.78	40	132.01	77	132.26	117	132.52
7	131.80	45	132.04	80	132.28	120	132.54
10	131.82	50	132.08	83	132.29	122	132.56
12	131.83	55	132.11	85	132.31	125	132.58
15	131.85	60	132.15	87	132.33	127	132.59
17	131.87	63	132.16	90	132.35	130	132.61
20	131.89	65	132.18	95	132.38	132.63	132.63
22	131.90	67	132.19	100	132.41	133	132.63
25	131.92	70	132.21	105	132.44		
30	131.95	72	132.22	110	132.47		
35	131.98	75	132.24	115	132.51		

The above table is based on sucrose concentration instead of on solids concentration, which has been shown to be the correct method. See pp. 404 and 534 for discussion.

SUBTRACTIVE TEMPERATURE CORRECTIONS

C°	0.0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
20	0.00	0.05	0.11	0.16	0.21	0.27	0.32	0.37	0.42	0.48
21	0.53	0.58	0.64	0.69	0.74	0.80	0.85	0.90	0.95	1.01
22	1.06	1.11	1.17	1.22	1.27	1.33	1.38	1.43	1.48	1.54
23	1.59	1.64	1.70	1.75	1.80	1.86	1.91	1.96	2.01	2.07
24	2.12	2.17	2.23	2.28	2.33	2.39	2.44	2.49	2.54	2.60
25	2.65	2.70	2.76	2.81	2.86	2.92	2.97	3.02	3.07	3.13
26	3.18	3.23	3.29	3.34	3.39	3.44	3.50	3.55	3.60	3.66
27	3.71	3.76	3.82	3.87	3.92	3.98	4.03	4.08	4.13	4.19
28	4.24	4.29	4.35	4.40	4.45	4.51	4.56	4.61	4.66	4.72
29	4.77	4.82	4.88	4.93	4.98	5.04	5.09	5.14	5.19	5.25
30	5.30	5.35	5.41	5.46	5.51	5.57	5.62	5.67	5.72	5.78
31	5.83	5.88	5.94	5.99	6.04	6.10	6.15	6.20	6.25	6.31
32	6.36	6.41	6.47	6.52	6.57	6.63	6.68	6.73	6.78	6.84
33	6.89	6.94	7.00	7.05	7.10	7.16	7.21	7.26	7.31	7.37
34	7.42	7.47	7.53	7.58	7.63	7.69	7.74	7.79	7.84	7.90
35	7.95	8.00	8.06	8.11	8.16	8.22	8.27	8.32	8.37	8.43

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* *Bur. Standards Scientific Papers 375.*

TABLE 50

Calculating the Per Cent Sucrose (Pol) in Bagasse. (Spencer)

Conditions: 100 gm. of bagasse; 1000 gm. water; dry lead clarification; 400 mm. observation tube; W = (100 + Water) − % fiber (marc)

W (Grams)	980	990	1000	1010	1020	1030	1040	1050	1060	1070	1080
Observation	Per Cent Sucrose (Polarization) in the Bagasse										
1.5	1.91	1.93	1.95	1.97	1.99	2.01	2.03	2.05	2.07	2.09	2.11
.6	2.04	2.06	2.08	2.10	2.12	2.14	2.16	2.18	2.20	2.23	2.25
.7	2.17	2.19	2.21	2.23	2.25	2.28	2.30	2.32	2.34	2.36	2.39
.8	2.29	2.32	2.34	2.36	2.39	2.41	2.43	2.46	2.48	2.50	2.53
.9	2.42	2.45	2.47	2.49	2.52	2.54	2.57	2.59	2.62	2.64	2.67
2.0	2.55	2.57	2.60	2.63	2.65	2.68	2.70	2.73	2.76	2.78	2.81
.1	2.68	2.70	2.73	2.76	2.78	2.81	2.84	2.87	2.89	2.92	2.95
.2	2.80	2.83	2.86	2.89	2.92	2.95	2.97	3.00	3.03	3.06	3.09
.3	2.93	2.96	2.99	3.02	3.05	3.08	3.11	3.14	3.19	3.20	3.23
.4	3.06	3.09	3.12	3.15	3.18	3.21	3.24	3.28	3.31	3.34	3.37
.5	3.19	3.22	3.25	3.28	3.32	3.35	3.38	3.41	3.45	3.48	3.51
.6	3.31	3.35	3.38	3.41	3.45	3.48	3.52	3.55	3.58	3.62	3.65
.7	3.44	3.47	3.51	3.55	3.58	3.62	3.65	3.69	3.72	3.76	3.79
.8	3.57	3.60	3.64	3.68	3.71	3.75	3.79	3.82	3.86	3.89	3.93
.9	3.69	3.73	3.77	3.81	3.85	3.88	3.92	3.96	4.00	4.03	4.07
3.0	3.82	3.86	3.90	3.94	3.98	4.02	4.06	4.10	4.13	4.17	4.21
.1	3.95	3.99	4.03	4.07	4.11	4.15	4.19	4.23	4.27	4.31	4.35
.2	4.08	4.12	4.16	4.20	4.24	4.28	4.33	4.37	4.41	4.45	4.49
.3	4.20	4.25	4.29	4.33	4.38	4.42	4.46	4.50	4.55	4.59	4.63
.4	4.33	4.38	4.42	4.46	4.51	4.55	4.60	4.64	4.69	4.73	4.77
.5	4.46	4.50	4.55	4.60	4.64	4.69	4.73	4.78	4.82	4.87	4.91
.6	4.59	4.63	4.68	4.73	4.77	4.82	4.87	4.91	4.96	5.01	5.05
.7	4.71	4.76	4.81	4.86	4.91	4.95	5.00	5.05	5.10	5.15	5.19
.8	4.84	4.89	4.94	4.99	5.04	5.09	5.14	5.19	5.24	5.29	5.34
.9	4.97	5.02	5.07	5.12	5.17	5.22	5.27	5.32	5.37	5.42	5.48
4.0	5.10	5.15	5.20	5.25	5.30	5.36	5.41	5.46	5.51	5.56	5.62
.1	5.22	5.28	5.33	5.38	5.44	5.49	5.54	5.60	5.65	5.70	5.76
.2	5.35	5.41	5.46	5.51	5.57	5.62	5.68	5.73	5.79	5.84	5.90
.3	5.48	5.53	5.59	5.65	5.70	5.76	5.81	5.87	5.93	5.98	6.04
.4	5.61	5.66	5.72	5.78	5.83	5.89	5.95	6.01	6.06	6.12	6.18

TABLE 51

INDEX OF SUBSTANCES THAT ARE OR HAVE BEEN USED FOR PURIFYING, DECOLORIZING, AND CLARIFYING SUGAR-CONTAINING SOLUTIONS

Compilation by Prof. Dr. Edmund O. von Lippmann, printed in Deutsche Zuckerindustrie, Vol. XXXIV, page 9 (Jan., 1909).—Translation supplied through the courtesy of Dr. Charles A. Browne, Bureau of Chemistry, Washington.
See also supplementary Table 51*a*, p. 812, extending this list to 1937.

(A list of the abbreviations of references is given at the end of this index.)

I. SULPHUR; ITS ACIDS, COMPOUNDS AND DERIVATIVES

1. Sulphur (Leuchs III, 86; 1836).
2. Hydrogen Sulphide (Sievier, 1847, in Woodcroft, 94; Hlavati, Chz., 28, 1180).
3. Hydrogen Persulphide (Hlavati, Chz., 28, 1180).
4. Sulphuric Acid (Achard, about 1800, Gesch., 407; Kessler, Z., 16, 760; Hagemann, D. Z., 12, 491).
5. Sulphuric Acid with Lime (Mége, D., 115, 215).
6. Sulphuric Acid with Zinc Chloride (Thiele, Chz., 20, 404).
7. Sulphuric Acid with Zinc Sulphate (Terry, 1833, in Woodcroft, 54).
8. Potassium or Sodium Sulphate (Macfadyen, 1830, Gesch., 423).
9. Ammonium Sulphate (Dullo, D., 155, 71; Beanes, D., 167, 220).
10. Sulphuric with Sulphurous Acid (Possoz, D., 170, 64).

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11. Sulphurous Acid (Drapiez, "Bull. de la Société d'encourag.," Paris, 1811, X. 56; Perpere, 1812, and Dubrunfaut, 1829, Zerban, 1908.
12. Sulphurous Acid and Hydrogen Sulphide (Hlavati, Bl. Ass., 16, 759).
13. Sulphurous Acid with Calcium Bisulphite (Stolle, D., 114, 305).
14. Sulphurous Acid with Chloride of Lime and Phenol (Meiner, Bl. Ass., 10, 165).
15. Sulphurous Acid and Phenol (Kowalski, Z., 55, 396).
16. Sodium Sulphite (Perrier and Possoz, Z., 12, 128; Rümpler, N.Z., 30, 204).
17. Potassium Sulphite (Cassel and Kempe, S. ind., 47, 684).
18. Ammonium Sulphite (Beanes, D., 167, 220).
19. Calcium Sulphite (Prout, 1810; Melsens, C. r., 55, 729; Calvert, Z., 12, 500).
20. Barium Sulphite with Ozygen (Bouillant, S. ind., 50, 189).
21. Magnesium Sulphite (Mehay, Z., 23, 27; Drost and Schulz, Oe., 1885, 891; Degener, D. Z., 24, 203).
22. Lead Sulphite (Scoffern, 1847, in Woodcroft, 98).
23. Ferrous Sulphite (Englert and Becker, N. Z., 16, 70).
24. Aluminum Sulphite (Boulin, 1846, in Zerban, 15; Brandé, 1846, Z., 44, 455; Mehay, Z., 23, 27).
25. Aluminum Sulphite with Calcium Hydrate (Schubarth, Z., 2, 129).
26. Aluminum Sulphite with Manganese Sulphate (Massé, Z., 10, 256).
27. Acid Potassium Sulphite (Z., 1, 254; Cassel and Kempe, S. ind., 47, 684).
28. Acid Sodium Sulphite (Perrier and Possoz, Z., 12, 128).
29. Acid Alkali Sulphite with Calcium Bisulphite (Allabard, Engl. Patent No. ?).
30. Acid Calcium Sulphite (Stolle, 1838, in Zerban, 15; Melsens, D., 117, 136; Reynoso, Z., 12, 500).
31. Acid Calcium Sulphite with Alum (Leyde, Z., 1, 365).
32. Acid Calcium Sulphite and Calcium Hydrate and Alum (Lapeyrere, S. ind., 27, 568).
33. Acid Barium Sulphite, also with Alum (Lapeyrere, see above).
34. Acid Strontium Sulphite (Melsens, S. ind., 9, 379).
35. Acid Magnesium Sulphite (Mehay, Z., 23, 26; Hulwa, Oe., 13, 465; Saillard, S. ind., 42, 82).
36. Acid Iron Sulphite (Becker, N. Z., 16, 6).
37. Acid Aluminum Sulphite (Stolle, 1838, S. ind., 8, 295; Becker, Z., 35, 924).
38. Acid Aluminum Sulphite with Aluminum Phosphate (Schiller, Z. B., 12, 509).

TABLE 51 (*Continued*)

39. Calcium Trisulphite (?) (Labarre, Oe., 18, 36).
40. Basic Magnesium Sulphite (Berggreen, B., 16, 2542).

41. Hyposulphurous Acid (Talamo, N. Z., 29, 211; Baudry, Z., 53, 260).
42. Sodium Hyposulphite (Thiele, Chz., 20, 204).
43. Sodium Hyposulphite with Lime and Aluminum Acetate (D. Z., 33, 912).
44. Sodium Hyposulphite with Phosphoric Acid or Phosphates (Stein and Crosfield, Z., 53, 1334).
45. Hyposulphites of the Alkaline Earths and Magnesia (Reece and Price, 1849, in Woodcroft, 106).
46. Hydrosulphurous Acid (Ranson, Oe., 26, 737).
47. Ammonium Hydrosulphite (Descamps, S. ind., 65, 673).
48. Sodium Hydrosulphite (Thiele, Chz., 20, 404; Schiller, Z. B., 22, 683).
49. Calcium Hydrosulphite with Barium Hydrate (Descamps, S. ind , 65, 673).
50. Hydrosulphite of Calcium, Barium or Strontium (Descamps, S ind., 65, 673).
51. Magnesium Hydrosulphite (Becker, Z., 36, 978).
52. Cadmium Hydrosulphite (Urbain, S. ind., 50, 31).
53. Zinc Hydrosulphite (Urbain, see above).
54. Double Salt of Zinc Hydrosulphite with Sodium Chloride or Bromide and Ammonium Chloride or Fluoride (Harding, S. ind., 66, 742).
55. Iron or Manganese Hydrosulphite (Descamps, S. ind., 65, 673).
56. Aluminum Hydrosulphite (Descamps, see above).
57. Hydrosulphite of Alumina (Becker, Z., 36, 978).
58. Hydrosulphurous Acid and Phenol (Kowalski, Z., 55, 396).

II. PHOSPHORUS; ITS ACIDS, COMPOUNDS AND DERIVATIVES

59. Phosphorus Sulphide (Hlavati, Chz., 27, 254).

60. Phosphoric Acid (Stammer, Z., 9, 433).
61. Sodium Phosphate (Kuhlmann, Z., 2, 130).
62. Potassium Phosphate (Blanchard, B., 6, 153).
63. Ammonium Phosphate (Kuhlmann, Z., 2, 92; Beanes, Amer. patent, 1862).
64. Sodium Calcium Phosphate (Gwynne, Z., 3, 292).
65. Calcium Phosphate (Oxland, Z., 2, 130; Ostermann, S. ind., 40, 598).
66. Barium Phosphate (Heffter, Oe., 22, 71).
67. Strontium Phosphate (Heffter, see above).
68. Magnesium Phosphate (Kessler, Z., 15, 525).
69. Phosphate of Alumina (Oxland, Z., 2, 130).
70. Acid Ammonium Phosphate (Packert, S. ind., 25, 25).
71. Acid Ammonium Phosphate with Barium Hydrate (Chameroy, S. ind., 51, 173).
72. Acid Calcium Phosphate (Richter, 1834, Z., 44, 446; Schott, N. Z., 14, 314).
73. Acid Calcium Phosphate with Calcium Bisulphite (Barthelémy, S. ind., 52, 468).
74. Acid Calcium Phosphate with Magnesium Sulphate (Kessler, Z., 15, 51).
75. Acid Barium Phosphate (Manoury, J. Fabr., 29, 24).
76. Acid Magnesium Phosphate (Kessler, Z., 15, 51).
77. Acid Phosphate of Alumina (Oxland, Z., 2, 130).
78. Calcium Superphosphate (Maguin, J. Fabr., 29, 23).
79. Superphosphate of Alumina (Daubeny, 1857, in Ling-Roth, 23; Stubbs, Bl. Ass., 9, 912).
80. Commercial Superphosphate (Casamajor, Z., 34, 1269).
81. Tribasic Calcium Phosphate, also with Sulphurous Acid (Packert, S. ind., 25, 25).
82. Tribasic Calcium Phosphate with Alum (Kessler, Z., 15, 51).
83. Tribasic Calcium Phosphate with Ammonium Phosphate (Leplay, Z., 12, 193).
84. Tribasic Phosphate of Alumina with Sulphurous Acid (Packert, S. ind., 25, 25).
85. Manganese Phosphate (Lefranc, S. ind., 58, 410).

86. Metaphosphoric Acid (Bielmann, S. C., 28, 386; Müller, S. ind., 47, 410).

TABLE 51 (*Continued*)

87. Sodium Calcium Metaphosphate (Gwynne and Young, 1836, in Woodcroft, 59).
88. Sodium Calcium Pyrophosphate (Gwynne and Young, see above).
89. Phosphorous Acid (Hlavati, Chz., 27, 254).
90. Phosphite of Alumina (Spence, Z., 31, 231).
91. Acid Phosphites and Sulphites (Kühnel, Prager Marktb., 1888, 168).
92. Phospho-sulphites of the Alkalies and Alkaline Earths (Prangey and Grobert, S. ind., 54, 425).
93. Hypophosphorous Acid (Hlavati, Chz., 27, 254).

III. BORON, SILICON, CARBON, THEIR ACIDS, COMPOUNDS AND DERIVATIVES

94. Boric Acid (Payen, 1828, in Weber I, 565).
95. Boric Acid with Sulphur Powder (Fancher and Clarke, Bl. Ass., 9, 912).
96. Boric Acid and Borates of the Alkaline Earths (Oppermann, Z., 30, 533; Brear, B., 15, 1224).
97. Ammonium Borate (Besson, J. Fabr., 43, 1).
98. Borax (Brear, B., 15, 1224).
99. Hydrofluoboric Acid (Hlavati, Z., 53, 258).

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100. Silicon Fluoride (Hlavati, Z., 52, 758).
101. Silicic Acid (Leuchs III, 86, 1836).
102. Silicic Acid (Kieselguhr), (Heddle, Oe., 16, 441).
103. Kieselguhr and Saw-dust (Soxhlet, Z., 43, 972).
104. Hydrated Silicic Acid (Schubarth, Z., 2, 92).
105. Potassium Silicate, also with Gypsum (Schott, D., 251,'91).
106. Sodium Silicate (Wagner, Z., 9, 331).
107. Polysilicates of Magnesium and Aluminum (Hlavati, Chz., 28, 1180).
108. Zinc Silicate (Hlavati, see above).
109. Silicate of Alumina, *e.g.*, Brick Dust (Maumené, textbook).
110. Silicate of Alumina, *e.g.*, Brick Dust, with Caustic Lime (Breyer, Z., 54, 1271).
111. Hydrofluosilicic Acid (Kessler, Z., 16, 760; Gin, Z., 46, 627; Schoonjans, Chz., 30, 382).
112. Ammonium Hydrofluosilicate (Mills, N. Z., 39, 115; Whiteman, S. C., 1903, 565).
113. Ammonium Hydrofluosilicate with Lime (Hlavati, Chz., 28, 1110).
114. Hydrofluosilicic Acid with Calcium Carbonate (Marix, Bl., 1869, 346).
115. Magnesium Hydrofluosilicate (Kessler, Z., 16, 760).
116. Zinc Hydrofluosilicate (Rivière, Bl. Ass., 25, 603).
117. Lead Hydrofluosilicate (Vivien, Bl. Ass., 8, 24; Sokol, Chz., 21, R., 68).
118. Basic Lead Salt of Hydrofluosilicic Acid (Hlavati, Chz., 28, 1180).
119. Aluminum Hydrofluosilicate (Rivière, J. Fabr., 49, 18).
120. Iron Hydrofluosilicate (Lefranc, Z., 41, 498; Drost Patent, 54, 372).
121. Hydrofluosilicic Acid with Powdered Iron or Aluminum (Mertens, S. ind., 63, 659).
122. Manganese Hydrofluosilicate (Kessler, Z., 16, 760).
123. Hydrofluosilicate of Alumina (Kessler, Z., 15, 525).
124. Hydrofluosilicic Acid with Alumina (Gin, S. ind., 46, 48).
125. Carbonic Acid (Barruell, 1811; Oe., 23, 946; Leuchs, 1836, III, 86).
126. Potassium Carbonate, also with Fuller's Earth (Freund, 1827, Gesch, 369).
127. Sodium Carbonate (Dubrunfaut, about 1830 (?), Clémandot in Weber, III, 568).
128. Sodium Potassium Carbonate (Richard, 1856, in Woodcroft, 211).
129. Ammonium Carbonate (Payen, 1828, in Weber, I, 565; Nind, Z., 1, 595; Stammer, Z., 9, 430).
130. Acid Sodium Carbonate (Perrier and Possoz, St. J., 1863, 350).
131. Acid Sodium Carbonate with Alum (Salisbury, Z., 54, 849).
132. Acid Ammonium Carbonate (Dubrunfaut, about 1830 (?),).
133. Potassium Percarbonate (Bismer, Oe., 28, 534).

TABLE 51 (*Continued*)

IV. HYDROGEN, OXYGEN, HALOGENS, NITROGEN, THEIR ACIDS, COMPOUNDS AND DERIVATIVES

134. Nascent Hydrogen, from Hydroperoxide with Zinc, Lead or Manganese (Manoury, Z., 48, 140).
135. Hydrogen Peroxide (Frank, Z., 11, 392).
136. Hydrogen Peroxide with Phosphoric Acid or Alkaline Phosphates (Stein and Crosfield, Oe., 28, 181).
137. Hydrogen Peroxide with Phosphoric Acid and Magnesia (Pechnik and Bögel in Z., 25, 127).
138. Hydrogen Peroxide and Bone Black (Ranson, Oe., 26, 737)
139. Oxygen Gas (Reboux, S. ind., 36, 150; Wayland, S. C., 1893, 611).
140. Ozonized Air (Schneller and Wisse, S ind., 39, 467).
141. Air and Ozonized Air (Steffens, S. ind., 72, 214).
142. Ozone (Beanes, 1866, in Woodcroft, 392; Lee, B., 2, 64).
143. Ozone with Chlorine and Soda (Brin, Engl. Patent, 2297).
144. Ozonized Chlorine (*) (Lewicki, Z., 54, 245).
145. Ozone with Sulphurous Acid and Barium Hydrate (Verley, S. ind., 53, 301).
146. Ozone with Chloride of Lime and Alumina (Brin, Engl. Patent, 2297).

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147. Chlorine Gas (Strathing and Smit, 1820, Z., 49, 370; Z., 1, 258; Siemens, 1859, Z., 44, 458; Duncan, St. J., 1882, 274).
148. Liquefied Chlorine (Reboux, S. ind., 36, 150).
149. Chlorine with Carbonic Acid (Bismer, Oe., 38, 532).
150. Chlorine with Acetylene (Carlee, D. Z., 33, 738).
151. Chlorine with Ethylene (Kitsee, S. C., II, 2, 49).
152. Hydrochloric Acid (Margueritte, S. ind., 8, 71; Kessler, Z., 16, 761; Erk, Z., 26, 288).
153. Hydrochloric Acid with Metallic Powders (Hlavati, Z., 52, 758).
154. Hydrochloric Acid with Alum (Thiele, Chz., 20, 404).
155. Ammonium Chloride (Macfadyen, 1830, Gesch., 423; Reboux, Z., 84, 94; Licht, St. J., 24, 415).
156. Potassium Chloride (Macfadyen, 1830, Gesch., 423).
157. Sodium Chloride (Nash, 1852, in Woodcroft, 151).
158. Hypochlorous Acid (Z., 1, 255; Bismer, Oe., 34, 532).
159. Hypochlorous Acid Anhydride (Lagarigue, S. ind., 35, 549).
160. Hypochlorites of Alkalies (Dobler, S. ind., 66, 517; Hafner, Oe., 37, 86).
161. Hypochlorites of Alkaline Earths (Herapath, 1862, in Woodcroft, 320).
162. Hypochlorite of Alumina (used in England about 1880).

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163. Bromine (Maumené, S. ind., 1895, 577).
164. Hydrofluoric Acid (Frickenhaus, Z., 15, 43; Schoonjans, Chz., 29, 889).
165. Ammonium Fluoride (Besson, Chz., 27, 863, Bartz, 125).
166. Ammonium Fluoride with Aluminum (Voss, Z., 50, 438).
167. Magnesium Fluoride (Kessler, S. ind., 1, 363).
168. Calcium Fluoride (Kessler, S. ind., 1, 363; Abraham, C Z., 11, 886).

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169. Nitrous Oxide (Melsens, 1849, D. Z., 25, 1360; Hlavati, Chz., 28, 1180).
170. Nitrous Acid (Drapiez, in Blachette-Zoega, 1833, 264; Newton, 1849, in Woodcroft, 111).
171. Nitrites of the Alkalies and Alkaline Earths (Decastro, Z., 29, 270).
172. Nitric Acid (Kessler, Z., 16, 61).
173. Calcium Nitrate (Decastro, Z., 29, 270).
174. Potassium Nitrate (Macfadyen, 1830; Gesch., 423).

TABLE 51 (*Continued*)

V. ALKALIES, ALKALINE EARTHS, AND THEIR COMPOUNDS

175. Ammonia (Nash, 1852, in Woodcroft, 152; Michaelis, Z., 2, 448).
176. Ammonia, also with Caustic Lime (Marot, B., 9, 643).
177. Ammonia with Magnesium or Aluminum Sulphate (Hlavati, S. ind., 65, 673).
178. Ammonia with Oxalic Acid (Hlavati, Z., 56, 300).
179. Ammonium Sulphide (Bandris, 1853, in Ling-Roth, 107).
180. Caustic Potash with Alkali Carbonate (partially causticated plant ash), about 700 in Egypt, Gesch., 134 and 287).
181. Potassium Sulphide or Sodium Sulphide (Bandris, see 179).
182. Sodium acetate (Margueritte and Maumené, Z., 28, 845).

183. Calcium Peroxide (Hlavati, Chz., 27, 254).
184. Caustic Lime and Hydrated Lime (in Egypt about 700, Gesch., 134 and 287).
185. Calcium Hydrate with Soda (Beuster, J. Fabr., 32, 2).
186. Calcium Hydrate with Gypsum (Nathusius, in Bley, 75).
187. Calcium Chloride (Balling, 1837, Z., 44, 452; Michaelis, Z., 2, 65).
188. Chloride of Lime (Brandes, 1824, Z., 44, 447, Z, 7, 423).
189. Chloride of Lime with Sulphurous Acid (Hafner and Bismer, Oe., 37, 199).
190. Calcium Chloride with Lime or Magnesia (Guignard, Z., 53, 446).
191. Calcium Carbonate (Maumené, J. Fabr., 17, 22).
192. Calcium Carbonate with Milk of Lime (Dabrowski, Z., 50, 615).
193. Calcium Bicarbonate (Reece and Price, 1849, in Woodcroft, 106).
194. Calcium Nitrate with Sulphate of Alumina (Pape, Chz., 12, 30).
195. Calcium Sulphate (Howard, 1810, Gesch., 368; Druke, 1816, in Woodcroft, 23; Leyde, Z., 1, 378; Duquesne, D., 196, 83).
196. Calcium Sulphate with Lime (Kassner, D. Z., 29, 2151).
197. Calcined Gypsum with Lime (Leisy, D. Z., 29, 919).
198. Calcium Acetate (Barth, 1832, Z., 44, 449; Durieux, St. J., 8, 334).
199. Calcium Borate (Klein, B., 9, 1433).
200. Calcium Sulphide (Drapiez in Blachette-Zoega, 1833, 264).
201. Calcium Sulphide with Magnesium Sulphate (Drummond, D., 203, 325).
202. Calcium Persulphide (Talamo, S. ind., 40, 57).
203. Calcium Sulphuret (Reece and Price, 1849, in Woodcroft, 106).
204. Polysulphurets of Calcium or Calcium Sulphide with Ammonia and Sulphurous Acid (Hlavati, S. ind., 72, 487).
205. Calcium Carbide (Rivière, Bl. Ass., 15, 583).

206. Barium Oxide Hydrate (Lagrange, J. Fabr., 14, 34; Du Beaufret and Manoury, Z., 40, 590).
207. Barium Oxide Hydrate with Ammonium Phosphate (Lagrange, J. Fabr., 14, 34).
208. Barium Oxide Hydrate with Soda (Oppermann, Z., 40, 592).
209. Barium Oxide Hydrate with Iron Vitriol (Curley, S. ind., 43, 361).
210. Barium Peroxide (Beaudet, D. Z., 18, 1824).
211. Barium Peroxide with Phosphoric Acid (Stein and Crosfield, Z., 53, 1334).
212. Barium Peroxide Hydrate (Ranson, S. ind., 47, 251).
213. Barium Chloride (Licht, B., 15, 1471).
214. Barium Chloride with Caustic Soda (Plique, D. Z., 2, 51).
215. Barium Carbonate (Seyferth, Z., 25, 611; Heffter, Oe., 22, 71; Weisberg, S. ind., 64, 429).
216. Barium Carbonate with Sodium Phosphate and Sulphurous Acid (Packert, S. ind., 25, 25).
217. Barium Carbonate with Sulphate of Alumina (Eisenstuck, St. J., 3, 244).
218. Barium Carbonate with Potassium Permanganate (Talamo, N. Z., 29, 210).
219. Barium Sulphate with Barium Chloride and Lime (Haesendonck, S. ind., 43, 598).
220. Barium Sulphide and Sulphuret (Reece and Price, 1849, in Woodcroft, 106; Weisberg, S. ind., 64, 429).
221. Barium Sulphide with Caustic Soda (Romiguiéres, S. ind., 26, 682).

TABLE 51 (*Continued*)

222. Barium Sulphide with Magnesium Sulphate (Drummond, D., 203, 325).
223. Barium Manganate (Lefranc, Bl. Ass., 18, 962).
224. Barium Silicate (Hlavati, S. ind., 65, 675).
225. Barium Carbide (Rivière, Bl. Ass., 15, 583).
226. Barium Carbide with Barium Hydrate (Battistoni, S. ind., 68, 198).

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227. Strontium Oxide (Moureaux, Bl. Ass., 19, 1483).
228. Strontium Oxide Hydrate (Scheibler, Z., 32, 986).
229. Strontium Oxide Hydrate with Iron Sulphate (Curely, S. ind., 43, 361).
230. Strontium Chloride (Kottmann, Z., 32, 899).
231. Strontium Carbonate (Heffter, Oe., 22, 41).
232. Strontium Sulphide and Sulphuret (Reece and Price, 1849, in Woodcroft, 106).
233. Magnesium Alloyed with Potassium, Sodium, Copper, Mercury, Tin, Zinc, or Antimony (Besson, Oe., 36, 466).
234. Magnesium Powder with Alkalies (Ranson, Chz., 21, 1033).
235. Magnesium Powder with Acids (Manoury, S. ind., 51, 103).
236. Magnesium Oxide (Thénard, Z., 13, 128).
237. Magnesium Oxide Hydrate (Rümpler, D. Z., 4, 52; Oppermann, N. Z., 18, 216).
238. Magnesium Oxide Hydrate with Sulphuric Acid and Lime (Koebig, S. C., 1894, 274).
239. Magnesium Oxide Hydrate with Magnesium Carbonate (Rümpler, D. Z., 4, 180).
240. Magnesium Calcium (Dolomite), Oxide Hydrate (Oppermann and Manoury, S. ind., 1888, 240).
241. Magnesium Chloride (Nash, 1852, in Woodcroft, 151; Kessler, Z., 16, 760; Z., 23, 74; Drenckmann, D. Z., 17, 1468).
242. Magnesium Carbonate (Reich, Z., 6, 173; Spreckels. Chz., 28, 1070).
243. Magnesium Subcarbonate (Stenhouse, 1856, in Woodcroft, 216).
244. Magnesium Bicarbonate (Reece and Price, 1849, in Woodcroft, 106; N. Z., 25, 91).
245. Dolomite (Dubreul, B., 6, 155).
246. Magnesium Sulphate (Bayvet, Z., 10, 256).
247. Magnesium Sulphate with Lime or Baryta (Manoury, S. ind., 26, 680).
248. Magnesium Sulphate with Alcohol (Degener, Chz., 12, 174).
249. Magnesium Sulphide (Dubreul, J. Fabr., 13, 27; Rivière, Bl. Ass., 15, 583).
250. Magnesium Sulphide and Sulphuret (Reece and Price, 1849, in Woodcroft, 106).
251. Magnesium Silicate (Hlavati, S. ind., 65, 674).
252. Magnesium Carbide (Rivière, Bl. Ass., 15, 583).
253. Radium (?) (C. Z., 1902, 466).

VI. METALS AND THEIR COMPOUNDS

254. Aluminum Dust (Ranson, Chz., 21, 1033).
255. Aluminum Dust with Alkalies (Ranson, see above).
256. Aluminum Dust with Ammonium Sulphite (Besson, Bl. Ass., 19, 800).
257. Aluminum Dust with Hydrofluoric Acid or Hydrofluosilicic Acid (Mertens, Z., 54, 118).
258. Aluminum Alloys, also with Copper or Zinc Dust (Bessen, Chz., 28, 529).
259. Aluminum Chloride (Nash, 1852, in Woodcroft, 151; Heffter, Oe., 22, 71).
260. Aluminum Chloride with Lime (Siemens, St. J., 18, 256).
261. Aluminum Fluoride (Kessler, Z., 15, 525).
262. Alumina (about 700 in Egypt, Gesch., 135 and 295; Murray, about 1802, Gesch., 368).
263. Hydrate of Alumina (Howard, 1810, Gesch., 368; Z., 2, 92).
264. Colloidal Alumina (Löwig, Z., 29, 905).
265. Fuller's Earth (Fritsche, Z., 35, 361).
266. Sodium Aluminate also with Sulphurous Acid (Besson, Bl. Ass., 25, 733).
267. Aluminate of the Alkaline Earths (Plicque, D. Z., 2, 51).
268. Calcium Aluminate (Oxland, Z., 2, 92).
269. Basic Calcium Aluminate (Gui, Z., 46, 202).

TABLE 51 (*Continued*)

270. Tetra- and Hexa-Basic Aluminate of Calcium or Barium (Gin and Leleux, Bl. Ass., 16, 707).
271. Aluminate of Barium or Strontium (Jacquemart, French Patent, 51, 908, 1861; Rembert, Bl. Ass., 20, 747).
272. Barium Aluminate with Ammonia Alum (Geißtodt, Z., 28, 843).
273. Barium Aluminate with Sulphurous Acid (Jaluzot, S. ind., 63, 690).
274. Barium Aluminate with Aluminum Sulphate (Jaluzot, see above).
275. Magnesium Aluminate (used about 1888; Hlavati, S. ind., 65, 674).
276. Sulphite of Alumina (Kessler, Z., 15, 525; Massé, 1860, Z., 44, 458).
277. Sulphate of Alumina with Phosphoric Acid (Stein and Crosfield, Oe., 28, 183).
278. Basic Sulphate of Alumina (Hunt, Z., 30, 361; Brünjes, D. Z., 25, 19).
279. Alum (about 700 in Egypt, Gesch., 135; Hermbstaedt, "Anleit. z. Fabrik. des Zuckers," Berlin, 1811, 86).
280. Alum with Sodium Carbonate (Salisbury, Z., 54, 1274).
281. Alum with Lime and Alcohol (Derosne, Oe., 23, 948).
282. Alum also with Sulphate of Alumina (Howard, 1812, Z., 44, 446).
283. Aluminum Acetate (Oxland, 1850, in Ling-Roth., 121; Schubarth, Z., 2, 92).
284. Tartrate of Alumina (Dumas, C. Z., 1906, 939).
285. Oxalate of Alumina (Sievier, 1847, in Woodcroft, 94; Mialhe, D., 99, 482; Dumas, C. Z., 1906, 939).
286. Aluminum Phosphate (Oxland, Z., 2, 92, and 2, 130).
287. Aluminum Silicate (Maumené, Lehrbuch).
288. Aluminate Silicates (Gans, Z., 57, 206).
289. Iron- and Quartz-containing Clay (Harm, D. Z., 22, 1104).
290. Aluminum Sulphide (Hlavati, Chz., 27, 254).
291. Ferrous Oxide (Hills, 1850, in Woodcroft, 121).
292. Iron Hydroxide, also with Gypsum (Rousseau, Z., 11, 671).
293. Iron Sesquioxide, also with Ozone (Wayland, Chz., 19, 1519).
294. Iron Sesquioxide Hydrate (Wackernie, S. ind., 47, 251).
295. Iron Peroxide (Reynolds, 1859, in Woodcroft, 250).
296. Iron Ochre (Martineau, 1815, in Woodcroft, 21).
297. Iron Chloride (Sievier, 1847, in Woodcroft, 94; Karl, Z., 18, 317; Licht, N. Z., 11, 63).
298. Ferrous Chloride (Maumené, S. ind., 1895, 577).
299. Iron Oxy-chloride (Spunt and Schachtrupp, N. Z., 30, 216).
300. Ferrous Fluoride (Junius and Gouthiere, Chz., 25, 603).
301. Iron Carbonate (Reynolds, 1859, in Woodcroft, 250).
302. Ferric Sulphate (Sievier, 1847, in Woodcroft, 94; Karl, Z., 18, 317).
303. Basic Ferric Sulphate (Mehrle, Z., 32, 385).
304. Ferric Sulphate (Bayvet, Z., 10, 256; Mehrle, Z., 32, 385).
305. Iron Vitriol with Alkaline Earths (Curely, S. ind., 43, 361).
306. Iron Vitriol with Barium Carbonate (Beaufret, Bl. Ass., 10, 803).
307. Iron Vitriol with Gypsum (Lohmann, 1817, Z., 44, 447).
308. Iron Vitriol with Zinc (Schetke, Chz., 1906, 23).
309. Iron Vitriol with Albuminates (Karl, Z., 18, 317).
310. Iron Nitrate (Sievier, 1847, in Woodcroft, 94).
311. Salts of Ferric Acid, so-called "Ferrites" (Liesenberg, about 1892).
312. Iron Cyanide and Sulphurous Acid (Thompson, Z., 50, 957).
313. Potassium Ferrocyanide (Sievier, 1847, in Woodcroft, 94).
314. Potassium Ferrocyanide, also with Sulphurous Acid (Boot, Java Archiv., 1903, 1046).
315. Calcium Ferrocyanide (Therry, 1833, in Woodcroft, 54).
316. Chromium Peroxide (Piettre, Bl. Ass., 19, 1381).
317. Chromic Acid and Salts of Chromic Acid (Maumené, S. ind., 1895, 577).
318. Acid Chromic Acid Salts (Maumené, see above).
319. Chromium Sulphate (Lefranc, S. ind., 58, 410).
320. Chromium Phosphate (Lefranc, see above).

TABLE 51 (*Continued*)

321. Manganese Dust with Acids (Manoury, S. ind., 51, 103).
322. Manganese Oxide (Spreckels, Chz., 28, 1270).
323. Manganous Oxide (Eachran, D., 251, 91).
324. Manganese Dioxide (about 1836, Bley, 47; Frickenhaus, Z., 10, 301; Piettre, Bl. Ass., 19, 1351).
325. Manganese Chloride (Manoury, about 1880).
326. Manganese Chloride with Oxalic Acid (Fontenille, S. ind., 54, 425).
327. Manganese Carbonate (Newton, 1859, in Woodcroft, 253).
328. Manganese Sulphate (Massé, Z., 10, 256).
329. Manganates of the Alkalies and Alkaline Earths (Hawes, 1853, in Woodcroft, 163).
330. Sodium Manganate (Knaggs, 1866, in Woodcroft, 384).
331. Manganate of Lime (Z., 1, 256; Lefranc, Bl. Ass., 18, 962).
332. Calcium Permanganate (Maumené, J. Fabr., 1894, 51).
333. Sodium Permanganate (Knaggs, 1866, in Woodcroft, 384).
334. Calcium Permanganate (Fayolle, S. ind., 52, 554).
335. Aluminum Permanganate (Fayolle, see above).
336. Permanganates with Barium Carbonate and Oxalic Acid (Talamo, N. Z., 29, 210).
337. Copper Sulphate with Lime (Hlavati, Z., 56, 300).
338. Lead Dust, also with Acids (Manoury, S. ind., 51, 103).
339. Lead with Sulphides of the Alkalies (Bandris, Ling-Roth, 107).
340. Lead Oxide (about 1836, Bley, 126).
341. Plumbic Hydrate (Gwynne, Z., 3, 392; Lagrange, S. ind., 1892, 468; Wohl and Kollrepp, Z., 55, 60).
342. Litharge (Pfeifer and Langen, N. Z., 19, 131).
343. Lead Peroxide (Maumené, S. ind., 1895, 577; Piettre, Bl. Ass., 19, 1351).
344. Plumbites of the Alkaline Earths (Galloway, 1852, in Woodcroft, 147 and 171).
345. Lead Carbonate (Hills, 1850, in Woodcroft, 121; Besson, Chz., 28, 1270).
346. Lead Sulphate (Scoffern, 1850, in Woodcroft, 115).
347. Lead Nitrate (Lagrange, S. ind., 1892, 468).
348. Lead Nitrate with Sulphate of Alumina (Pape, Chz., 12, 30).
349. Basic Lead Nitrate (Wohl and Kollrepp, Z., 55, 60).
350. Lead Acetate (Scoffern, 1847, in Zerban, 15; Gwynne, Z., 3, 393).
351. Lead Subacetate, also with Sodium Sulphide (Maumené).
352. Lead Subacetate with Chalk (Pajot de Charmes, 1821, Gesch., 369).
353. Lead Subacetate with Sulphurous Acid (Scoffern, D., 117, 265; Ling-Roth, 81 and 82).
354. Lead Triacetate (?) (Gwynne and Young, in Woodcroft, 59).
355. Lead Saccharate (Gwynne, 1850, in Woodcroft, 116; Wohl and Kollrepp, Z., 54, 854).
356. Lead Albuminate (Gwynne and Young, 1836, in Woodcroft, 59).

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357. Zinc Dust with Mineral Acids (Manoury, S. ind., 51, 103).
358. Zinc Dust with Sulphuric Acid and Barium Sulphide (Cripo, St. J., 24, 416).
359. Zinc Dust with Sulphurous Acid, also with Ferrocyanides (Boot, Oe., 27, 717).
360. Zinc Dust with Hydrofluoric Acid (Mertens, Z., 54, 118).
361. Zinc Dust with Tartaric Acid (Koperski, Z., 54, 1271).
362. Zinc Dust with Alkalies (Ranson, Chz., 21, 1033).
363. Zinc Dust with Dolomite (Hlavati, Bl. Ass., 16, 759).
364. Zinc Dust with Ammonium Sulphide (Brünn, Chz., 31, R., 459).
365. Coppered Zinc-Powder (Verley, Chz., 24, 596).
366. Zinc Iron Alloys (Mertens, Z., 54, 118).
367. Zinc Chloride (Gauchy, N. Z., 13, 43; Heffter, Oe., 22, 71).
368. Zinc Fluoride (Hlavati, Z., 53, 258).
369. Zinc Oxide (about 1836, Bley, 126).
370. Zinc Hydrate (Wilson, 1815, Gesch., 368).
371. Zinc Hydrocarbonate (Perrin, Chz., 22, 54; Mittelstaedt, D. Z., 23, 1112).
372. Zinc Hydrocarbonate with Oxalic Acid (Moureaux, Bl. Ass., 19, 1483).

TABLE 51 (*Continued*)

373. Zinc Sulphate (Wilson, 1818, in Woodcroft, 27; Z., 44, 447; Hermbstädt, in Weber, 1829, 100).
374. Zinc Sulphate with Barium Hydrate (Wackernie, S. ind., 53, 201, 61, 718).
375. Zinc Nitrate (Decastro, St. J., 19, 340).
376. Zinc Nitrate with Alkali Sulphide (Decastro, see No. 375).
377. Zinc Nitrate with Calcium Sulphide or Barium Sulphide (Decastro, see No. 375).
378. *Zinc Aluminate (Hlavati, S. ind., 65, 674).*

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379. Cadmium Oxide (Mouraux, Bl. Ass., 19, 1483).
380. Cadmium Carbonate (Moraux, see No. 379).

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381. Tin Dust (Besson, Chz., 27, 863).
382. Stannic Oxide, also with Soda (B., 19, R., 520).
383. Stannic Hydrate (Wilson, 1815, Gesch., 368).
384. Stannic Chloride (Nash, 1852, in Woodcroft, 151; Maumené, J. Fabr., 20, 7).
385. Stannous Chloride (Nash, 1852, see No. 384; Havemeyer, 1869, in Zerban, 77; Manoury, Z., 34, 1275; Maumené; S. ind., 1895, 577).
386. Stannous Chloride with Sulphuric Acid (Thiele, Chz., 20, 404).
387. Stannous Nitrochloride (Nash, 1852, see No. 384).
388. Stannous Fluoride (Ranson, Chz., 24, 1026).
389. Stannic Sulphate (Anderson, 1856, in Woodcroft, 218).
390. Stannous Sulphate (Oe., 15, 76).
391. Tin Nitrate (Reynolds, 1859, in Woodcroft, 250).
392. Tin Chloronitrate (Reynolds, 1859, see No. 391).
393. Stannic Acid or Metastannic Acid (Reynolds, 1859, see No. 391).
394. Stannates of the Alkalies (Reynolds, 1859, see No. 391).
395. Stannates of the Alkaline Earths (Reynolds, 1859, see No. 391).
396. Metastannates of the Alkalies and Alkaline Earths (Reynolds, 1859, see No. 391).
397. Aluminum Metastannate (Reynolds, 1859, see No. 391).
398. Mercury Peroxide (Piettre, Bl. Ass., 19, 1351).
399. Mercuric Nitrate (S. C., II, 4, 216).

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400. Antimony Dust (Besson, Chz., 27, 863).
401. Antimony Tin Alloy (Mertens, S. ind., 63, 659).
402. Antimony Peroxide (Piettre, see No. 398).
403. Antimony Sulphide (about 1670, Gesch., 311).
404. Bismuth Nitrate (Sievier, 1847, in Woodcroft, 94).
405. Bismuth Salts (Hawes, 1853, in Woodcroft, 163).
406. Ammonium Molybdate (Wichardt, D. Z., 31, 652).

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407. Salts of Tungstic Acid (Reynolds, 1859, in Woodcroft, 259).

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408. Titanic Acid (Employed in England about 1880).
409. Ferrititanite (Liesenberg, about 1892).
410. Thorium- and Monazite-Earths (Browne, C. Z., 1904, 568).

VII. ORGANIC SUBSTANCES AND COMPOUNDS; BONE BLACK AND ITS SUBSTITUTES

411. Extract of Gall-Apples (about 700 in Egypt, Gesch., 135).
412. Tannins and Tanning Liquors (Dorion, 1816, Z., 49, 578; Leuchs, 1836; III. 86; Luther, Chz., 29, 1091).
413. Quebracho, Valonea, and Sumach (Hlavati, Chz., 27, 254).
414. Tannic Acid (Wagner, Z., 9, 331; Walkhoff, 1863, Z., 44, 459).
415. Liquid Tannic Acid (?) (Elias, S. ind., 1895, 20).
416. Tannate of Potassium or Ammonium (Galloway, 1853, in Woodcroft, 171).
417. Tannic Acid with Lime (about 1836, Bley, 126; Heffter, Oe., 16, 442).
418. Tannic Acid with Salts of Barium or Strontium (Heffter, see No. 417).

TABLE 51 (*Continued*)

419. Tannic Acid with Alumina (Heffter, see No. 417).
420. Tannic Acid with Tartaric Acid, Metaphosphoric Acid, and Hydrofluosilicic Acid (Royers, S. ind., 50, 32).
421. Tannic Acid with Glue, Starch or Albumen (Heffter, see No. 417).
422. Pertannic Acid (?) (Meritens, Z., 28, 800).
423. Gallic Acid (Royers, see No. 420; Kowalski, Chz., 26, 972).
424. Gallate of Potassium or Ammonium (Galloway, 1853, see No. 416).
425. Acetic Acid, also with Sulphurous Acid (Z , 20, 741; Stutzer and Wernekinck, S. ind., 51, 114). For acetates look under the list metals.
426. Wood Vinegar (Leidenfrost, Z., 20, 746).
427. Butyric Sulphonic Acid (?) (Spreckels, Chz., 28, 1270).
428. Fatty Acids with Sulphurous or Sulphuric Acid (Spreckels, Z. ang., 1902, 891; Chz., 28, 1072).
429. Stearic Acid (Z., 2, 91; Wagner, Z., 9, 331).
430. Ammonium Stearate (Besson, Chz., 27, 863).
431. Stearic Acid with Sulphites of the Alkalies or Magnesium (Stewart, Z., 57, 268).
432. Stearic-Sulphonic Acid (Spreckels, see No. 427).
433. Palmitic-Sulphonic Acid (Spreckels, see No. 427).
434. Margaric Acid (Pidding, 1853, in Woodcroft, 162).
435. Oleic Acid (Pidding, see No. 434; Thénard, Z., 8, 130).
436. Oleic-Sulphonic Acid (Spreckels, see No. 427).
437. Oxalic Acid (Leuchs, 1836, III, 86; Wagner, Z., 9, 331; Eissfeldt, Z., 21, 1102).
438. Oxalic Acid with Ammonia, Magnesium and Zinc (Besson, Bl. Ass., 18, 616).
439. Ammonium Oxalate (Sievier, 1847, in Woodcroft, 94; Besson, J. Fabr., 43, 1).
440. Oxalic Acid with Barium Carbonate and Permanganates (Talamo, N. Z., 29, 210).
441. Tartaric Acid (Possoz, Z., 23, 27; Stutzer and Wernekinck, S. ind., 51, 114).
442. Ammonium Tartrate (Besson, Chz., 27, 863).
443. Malic Acid with Metallic Bases or Carbonates (Moreaux, Bl. Ass., 19, 1483).
444. Citric Acid with Metallic Bases or Carbonates (Moreaux, Bl. Ass., 19, 1483).
445. Citric Acid, also with Polysilicates (Hlavati, Chz., 28, 1180).
446. Salicilic Acid (Hulwa, Z., 25, 640; D. Z. 9, 7).
447. Resin Acids (Leuchs, 1836; III, 86).
448. Pimaric Acid (Schiller, Z. B., 12, 33).
449. Pectic Acid (Acar in Wagner's Technologie, 12th Ed., 563).
450. Formaldehyde (Boulet, Chz., 20, 12; Friedrich, Chz., 27, 1183; Simpson, Bl. Ass., 25, 531).
451. Acetaldehyde (Newton, 1849, in Woodcroft, 111; Melsens, 1849, D. Z., 25, 1306; Boulet, Chz., 20, 12).
452. Methylalcohol (Trovach, D. Z., 11, 1302).
453. Alcohol (Jennings, 1825, in Woodcroft, 33; Pesier, Z., 11, 522).
454. Alcohol with Chlorine Gas (Duncan, St. J., 22, 274).
455. Alcohol with Acetic Acid (Paulet, 1837; Z., 14, 641; 19, 376).
456. Alcohol with Hydrochloric Acid, Nitric Acid or Sulphuric Acid (Ure, 1830, in Woodcroft, 49).
457. Alcohol with Sulphuric Acid and Gypsum (Duquesne, D., 196, 83).
458. Alcohol with Sulphurous Acid (Stolle, D., 114, 305).
459. Alcohol with Alum and Lime (Derosne, 1810, Oe., 23, 948).
460. Alcohol with Magnesium Sulphite (Degener, Chz., 12, 174).
461. Rum or Gin (Stokes, in Weber, III, 236).
462. Glycerine (Rabe, Z., 14, 124).
463. Glucose and its Salts (?) (Bielmann, S. C., 28, 386).
464. Saccharites of Lead or of the Alkaline Earths (Reece and Price, 1849, in Woodcroft, 106; Gwynne, Z., 3, 392; Stammer, Z., 12, 336).
465. Magnesium Saccharate (Galloway, Z., 3, 31).
466. Starch with Caustic Lime (Steinkamp, 1848, in Woodcroft, 102)
467. Hydrocarbons and Petroleum (Carbonelle, S. ind., 33, 455).
468. Kerosene with Alumina and Metallic Powder (Z., 53, 444).
469. Kerosene Oil (Spreckels and Kern, Z., 53, 878).
470. Tar Oils (Newton, 1849, in Woodcroft, 111).

TABLE 51 (*Continued*)

471. Benzol or Toluol, also with Sulphurous Acid or Hydrosulphurous Acid (Kowalski, Z., 53, 909).
472. Phenol (Fishman, Z., 21, 313; D. Z.,21, 9, 7).
473. Phenol with Benzol or Petroleum (Kowalski, Z., 55, 396).
474. Phenol with Chloride of Lime (Menier, Bl. Ass., 10, 165).
475. Oxybenzol (Kowalski, see No. 471).
476. Oxynaphthalin and Oxyanthracene, also with Sulphurous Acid or Hydrosulphurous Acid (Kowalski, see No. 471).
477. Oxyanthraquinone (Kowalski, see No. 473).
478. Fats with Sulphurous or Sulphuric Acid (Spreckels, Z. ang., 1902, 891; Chz., 28, 1072).
479. Tallow or Lard with Sulphurous or Sulphuric Acid (Spreckels & Kern, Z., 55, 571; 53, 878).
480. Fatty Oils and Mineral Oils (Bouvier, Z. B., 1896, 386).
481. Fatty Oils with Sulphurous or Sulphuric Acid (Spreckels, Z. ang., 1902, 891).
482. Fatty Oils with Soda (Brooman, 1857, in Woodcroft, 232).
483. Wax and Neutral Fat (Leuchs, III, 86).
484. Spermaceti and Spermaceti Oil (Pidding, 1853, in Woodcroft, 162).
485. Stearine and Palmatine (Carlee, D. Z., 33, 738).
486. Fish Oil with Sulphurous or Sulphuric Acid (Spreckels, Z., 55, 571; Z. ang., 1902, 891).
487. Linseed Oil with Sulphuric Acid (Spreckels and Kern, Z., 53, 878).
488. Castor Oil with Sulphuric Acid (Spreckels, Z., 55, 571).
489. Soap (Basset, Z., 7, 381).
490. Ammonia Soaps (Brooman, Z., 8, 449; Besson, J. Fabr., 43, 1).

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491. Turpentine (Newton, 1849, in Woodcroft, 111; Carlee, D. Z., 33, 738).
492. Turpentine and Sulphuric Acid (Spreckels and Kern, Z., 53, 878; 55, 571).
493. Tar with Sulphurous or Sulphuric Acid (Spreckels, Z. ang., 1902, 891).
494. Tar Oil (Pidding, 1853, in Woodcroft, 162).
495. Tar Oil also with Alumina and Metallic Powders (Z., 53, 444).
496. Tar Oils with Sulphurous or Sulphuric Acid (Spreckels, Z. ang., 1902, 891).
497. Resin (Pidding, 1853, in Woodcroft, 162).
498. Resin and Sulphuric Acid (Spreckels and Kern, Z., 53, 878).
499. Pitch (Pidding, 1853, see No. 497).
500. Creosote (Newton, 1849, in Woodcroft, 111).
501. Shellac (Greiger, S. ind., 54, 23).
502. Carbon Bisulphide (Ckiandi, S. ind., 25, 268).
503. Mustard Oil (Leuchs, 1836, III, 86; Newton, 1842, in Woodcroft, 111).
504. Radish Oil (Newton, 1842, see No. 503).
505. Sulphur-containing Ethereal Oils (Spreckels, Chz., 29, 1307).
506. Ethereal Oils with Sulphurous or Sulphuric Acid (Spreckels, Z. ang., 1902, 891; Chz., 28, 1072).
507. Animal Oil (Pidding, 1853, in Woodcroft, 162).
508. Eucalyptol (Fry, Amer. Patent, No. 472,989).
509. Eucalyptus Oil and Sulphuric Acid (Spreckels, Z., 55, 571).
510. Indigo White (Bielmann, S. C., 28, 386).
511. Milk (about 600, in Persia, Gesch., 102; Batley, 1810, Gesch., 369; Hermstadt, 1811).
512. Casein (Krüger, Z., 9, 221).
513. Albumen (about 700, in Egypt, Gesch., 135 and 209; Wilcox, Bl. Ass., 9, 912).
514. Calcium Albuminate (Karl, Z., 18, 317).
515. Calcium Albuminate with Iron Vitriol (Karl, see No. 514).
516. Blood (about 1700, Gesch., 324).
517. Hay or grass (Hlavati, Chz., 27, 254).
518. Bark of Trees, as Elm Bark (Stokes, in Weber, III, 236).
519. Cork (Wagner, Z., 43, 630).
520. Plane Shavings (Leuchs, 1836; III, 86).
521. Wood Dust (Wiechmann, 1885; D. Z., 28, 1544).

TABLE 51 (*Continued*)

522. Sawdust (Hills, 1853, in Woodcroft, 163; Casamajor, 34, 1269).
523. Wood Meal from Mechanical Pulp Process (Soxhlet, Z., 43, 972).
524. Wood Wool (Excelsior) (Müller and Schubert, Z., 44, 233).
525. Wood Pulp or Paper Pulp (Spreckels, Chz., 28, 1270).
526. Linen or Cotton Fabrics and Threads (Ost. Z., 58, 556).
527. Bran (Tyre, Z., 50, 475).
528. Dead Yeast (Clowes, Z., 54, 1286).
529. Yeast with Hydrochloric Acid (Effront, Z., 58, 326).
530. Peat with Calcium Sulphite (Nowak, Z., 53, 988).
531. Peat-Coal or Lignite (Maumené, Z., 4, 452).
532. Calcium Humate (Schmidt and Degener, D. Z., 20, 209).
533. Brown Coal (Böttcher, 1836, Gesch., 377).
534. Brown Coal Coke (Knauer, Z., 11, 350).
535. Wood Charcoal (Lowitz, 1793, Gesch., 368).
536. Charcoal Dust (Remmers, Z., 35, 369).
537. Coal-Dust, also with Alumina (Pajot de Charmes, 1821, Gesch., 369).
538. Lamp-Black (Martineau, 1815, in Woodcroft, 21).
539. Sugar Charcoal (Sievier, 1847, in Woodcroft, 94).
540. Plant-Blood Charcoal (Degener, Z., 46, 492).
541. Bituminous Coal (Martineau, 1815, in Woodcroft, 21; Payen, 1830, Z., 49, 594).
542. Carbon-Alumina (?) (Kachmarkiewicz, C. Z., 1906, 229).
543. Alumina Impregnated with Carbonized Blood (Olschewsky, Z., 32, 525).
544. Carbonized Kieselguhr (Infusorial Earth) (Heddle, Z., 37, 478).
545. Kieselguhr Impregnated with Carbonized Fatty Residues (about 1900, in America).
546. Bone Black (Figuier and Magnes, Gesch., 368).
547. Bone Black with Hydrogen Peroxide (Mastbaum, Z., 37, 704).
548. Bone Black Saturated with Carbonic Acid or Sulphurous Acid (Lach., Z., 46, 497).
549. Bone Meal (Hills, 1853, in Woodcroft, 163).
550. Osteine (Brunon and Rothé; Z., 54, 848).
551. Ferrocyanide Residues (So-called Coal-settlings) (Cavaillon, 1817, in Woodcroft, 25; Gawalowski, Oe., 18, 718).
552. Powder-Settlings of Stearin Factories (Lach and Benies, S. ind., 1895, 20).
553. Graphite with Bone Black and Zinc Bloom (Macherski and Koperski, Z., 57, 1121).
554. Graphite with Sand and Zinc Powder (Macherski and Koperski, Z., 57, 1044).
555. Anthracite (Hlavati, Z., 56, 300).
556. Coal-Tar with Lime (Lemaire, S. ind., 9, 56).
557. Carbonized Scums (Karlik, Oe., 32, 256).
558. Gravel (Bergmann, 1840, Z., 29, 1184; Meyer, 1879, Z., 30, 1149).
559. Bauxite (Hlavati, Z., 56, 300).
560. Calcined Phosphate-Slag (Lachaux, S. ind., 59, 677).
561. Cement (Harm, D. Z., 25, 1946).
562. Brick-Dust with Lime (Breyer, S. ind., 65, 655).
563. Pumice Stone (Saunders, 1835, in Woodcroft, 56).
564. Talc or Meerschaum (Hlavati Chz., 28, 1180).
565. Mica (Hlavati, Chz., 28, 1180).
566. Natural Zeolite (Riedel, S. ind., 70, 230).
567. Permutite = Artificial Zeolite (Riedel, see No. 566).
568. Soil from the Beet Storehouse (Kohlrausch, Z., 28, 215).

VIII. ELECTROLYTIC SUBSTANCES

569. Hydrogen (Kugler, Chz., 32; R., 454).
570. Ozone (Schollmeyer, Chz., 24, 825).
571. Chlorine, Bromine, Iodine, Fluorine (Spillern-Spitzer, Z., 53, 244).
572. Sulphurous Acid (Lallement, S. ind., 53, 301).

TABLE 51 (*Continued*)

573. Sulphurous Acid or Sulphites with Lead, Zinc, Aluminum, Iron, or Tin (Baudry and Charitonenko, Z., 50, 625).
574. Hydrosulphurous Acid (Ranson, Oe., 26, 737).
575. Coal (Despeissis, Battut, Z., 46, 624).
576. Wood Charcoal (Hlavati, Z., 53, 258).
577. Alkaline Earths (Gin and Leluex, Z., 53, 627).
578. Calcium Carbonate (Schwerin, D. Z., 29, 451).
579. Barium Salts (Bonillaut, S. ind., 50, 189).
580. Barium Aluminate (Rembert, Bl. Ass., 20, 966).
581. Magnesium (Urbain, Bl. Ass., 16, 719).
582. Magnalium (Murphy, J. Fabr., 44, 18).
583. Magnesium Hydroxide (Schwerin, Chz., 28, 626).
584. Magnesium Carbonate (Schwerin, D. Z., 29, 451).
585. Zinc (Schollmeyer, Z., 46, 624).
586. Zinc Alloy with Calcium or Antimony (Hlavati, Z., 53, 258).
587. Basic Zinc Salts (Wohl and Kollrepp, D. Z., 27, 1280).
588. Cadmium (Urbain, Bl. Ass., 16, 719).
589. Lead (Javaux, Gallois, Dupont, Z., 46, 626).
590. Lead-Antimony Alloy, also with Manganese Sulphate (Piettre and Nodon, D. Z., 27, 1211).
591. Lead Oxides (Z., 46, 626).
592. Lead Peroxide (Piettre and Nodon, Bl. Ass., 19, 1351).
593. Lead Saccharate (Wohl and Kollrepp, D. Z., 27, 1280).
594. Basic Lead Salts (Wohl and Kollrepp, see No. 593).
595. Aluminum (Z., 46, 626).
596. Aluminum-Magnesium (Browne, Z. ang., 1908, 174).
597. Aluminum Manganate with Zinc Hydroxide or Iron Hydroxide (Delavierre, Z., 53, 1106).
598. Alumina (Z., 46, 626).
599. Iron (Jennings, 1846, Clement, 1848, in Woodcroft, 89 and 103; Maigrot, Z., 46, 625).
600. Iron Bisulphide (Aschermann, Chz., 26, 683).
601. Manganese-Silicon Alloy (Hlavati, Z., 53, 258).
602. Manganese Dioxide (Hlavati, Z., 53, 626).
603. Hydrated Manganese Peroxide (Piettre and Nodon, Bl. Ass., 19, 1351).
604. Manganates of the Alkalies and Alkaline Earths (Lavollay and Bourgoin, D. Z., 25, 330).
605. Chromium Trioxide (Piettre and Nodon, see No. 603).
606. Nickel (Horsin-Déon, Oe., 28, 162).
607. Nickel with Sulphurous Acid or Sulphites (Baudry and Charitonenko, Z., 50, 625).
608. Copper (Görz, Z., 46, 624).
609. Iron (Horsin-Déon, Bl. Ass., 16, 729).
610. Antimony (Piettre and Nodon, D. Z., 27, 1211).
611. Antimony Peroxide (Piettre and Nodon, Bl. Ass., 27, 1315).
612. Mercury (Polaczek, Bl. Ass., 16, 720; Gurwitsch, Z., 54, 1030).
613. Mercury Amalgams (Polaczek, see No. 612).
614. Mercury Peroxide (Piettre and Nodon, Bl. Ass., 19, 1351).
615. Easily-Fluid Mercury Alloys (Palms, Bl. Ass., 17, 274).
616. Silver (Horsin-Déon, Oe., 28, 162).
617. Silver with Sulphurous Acid or Sulphites (Baudry and Charitonenko, Z., 50, 625).
618. Platinum (Collette, Z., 46, 623; Thomas and Howe, S. ind., 66, 624).
619. Platinum Antimony Alloy, also with Manganese Sulphate (Piettre and Nodon, D. Z., 27, 1211).
620. Platinized Copper (Charitonenko, S. ind., 53, 272).
621. Sebonaft = "Solid Mineral Oil" (Nowakowski, C. Z., 17, 277).
622. Straw-Meal (Lenze, D. Z., 33, 937).

TABLE 51 (*Continued*)

ABBREVIATIONS OF REFERENCES

Abbreviations	*Reference*
Bartz	Claassen-Bartz's "Zuckerfabrikation" (Leipzig, 1905).
B	Berichte der deutschen chemischem Gesellschaft (R = Referate).
Bl.	Bulletin de la Société chimique.
Blanchette Zoega	"Manuel du fabricant et du raffineur de sucre" (Paris, 1833).
Bley	Bley's "Zuckerbereitung aus Runkelrüben" (Halle, 1836).
Bl. Ass.	Bulletin de l'association des chimistes.
Chz.	Chemiker-Zeitung (R = Repertorium).
C. r.	Comptes rendus.
C. Z.	Centralblatt für die Zuckerindustrie.
D.	Dingler's polytechnisches Journal.
D. Z.	Die Deutsche Zuckerindustrie.
Gesch.	Lippmann's "Geschichte des Zuckers" (Leipzig, 1890).
J. fabr.	Journal des fabricants de sucre.
Ling-Roth	Ling-Roth's "Guide to the Literature of Sugar" (London, 1890).
Leuchs	Leuchs "10,000 Erfindungen und Ansichten" (Nürnberg, 1871).
Maumené	Maumené's "Traité de la fabrication du sucre" (Paris, 1878).
N. Z.	Neue Zeitschrift für Rübenzuckerindustrie.
Oe.	Oesterreichisch-Ungarische Zeitschrift für Zuckerindustrie.
Prager Marktb.	Prager Marktbericht.
S. C.	The Sugar Cane.
S. ind.	La sucrerie indigène et coloniale.
St. J.	Stammer's "Jahresbericht der Zuckerfabrikation."
Weber	Weber's "Zeitblatt für Gewerbetreibende."
Woodcroft	Woodcroft's "Abridgments of Specifications relating to Sugar" (London, 1871).
Z.	Zeitschrift des Vereins der Deutschen Zuckerindustrie.
Z. ang.	Zeitschrift für angewandte Chemie.
Z. B.	Zeitschrift für Zuckerindustrie in Böhmen.
Zerban	Zerban's "Louisiana Bulletin No. 103" (Baton Rouge, 1908).

ADDITIONAL ABBREVIATIONS FOR TABLE 51*a*

C.	Chem. Centr.
F. a. S.	Facts About Sugar.
I. S. J.	International Sugar Journal.
Newlands	"Sugar" (London, 1909).
S. B.	Sucré Belge.

TABLE 51*a*

Supplementary List of Substances Proposed for the Purification, Decolorization, and Clarification of Sugar Juices and Products

(Courtesy *International Sugar Journal*)

This list is an extension of Table 51, p. 798, and is compiled from the complete translation of von Lippmann's revised list (*Deut. Zuckerind.*, 1937) given in *International Sugar Journal*, May, 1937, p. 187, and subsequent issues to June, 1938. The earlier table has been compared and corrected with this later translation. Substances marked with an asterisk are listed in the earlier translation but the references here given are in addition to those given in the previous list.

I. SULPHUR: ITS ACIDS, COMPOUNDS, AND DERIVATIVES

Colloidal sulphur (Hugel, C., 1934, p. 4987).
*Sulphuric acid (Vassena, Z., 60, p. 751; Wiese, Z., 63, p. 587).
Sulphurous acid and ammonium biphosphate (François, C., 1933, p. 145).
Sulphurous acid and alcohol (Larrondo, Z., 55, p. 396).
*Sodium sulphite (Stanek, Z. B., 11, p. 14).
Aluminium sulphite with kaolin (Lipski, S. ind., 75, p. 137).
Double salt of zinc hydrosulphite with ammonia (Mestre and Dutillory, Chz., 1931 R., p. 151).

II. PHOSPHORUS: ITS ACIDS, COMPOUNDS, AND DERIVATIVES

*Sodium phosphate (Hood, Z., 71, p. 844).
Mono- and disodium phosphate (Naughe, C., 1934, p. 1539).
Trisodium phosphate (Hamous, Chz., 1932, R., p. 115).
*Calcium phosphate (Manoury, Z., 62, p. 1413).
Dicalcium phosphate with kieselguhr (Guerrero, Z., 1913, p. 190).
Aluminium phosphate with aluminium and carbon (Hayashi, S. C., II, 20, p. 385).
*Acid ammonium phosphate (Fleshmann, C., 1935, p. 3449).
*Commercial superphosphate (Wiesner, Z. B., 40, p. 472).

III. BORON, SILICON, AND CARBON: THEIR ACIDS, COMPOUNDS, AND DERIVATIVES

Colloidal silicic acid (Bielmann, D. Z., 1920, p. 493).
Electro-osmosed silicic acid (Marcus, D. Z., 1914, p. 823).
Silicic acid gel (Kullgrem and Lind, Z., 78, p. 283).
Alkali silicates with alcohol (Pollaczek, Chz., 1910, R., p. 459).
Calcium silicate (Schneller, Z., 69, p. 320).
Barium silicate (Deguide, Z., 76, p. 691).
Tribasic barium silicate (Spencer, F. A. S., 1932, p. 540).
Barium metasilicate (Deguide, Z., 76, p. 768).
Hydrated aluminium silicate with kieselguhr (Krieger, Z., 69, p. 401).
Zeolite (Elbogen, D. Z., 1929, p. 133; Bachler, Z. B., 1926, Beil., 43).
Ammoniacal zeolite (Jeanprost, Bl. Ass., 1932, pp. 195 and 457; Austerweil, Chz., 1934, R., p. 126).
Calcium zeolite (Riedel, Z., 63, p. 169).
Sodium carbonate with common salt and sand (Leclaire, D. Z., 1910, p. 341).
*Ammonium carbonate (Stutzer, Chz., 1912, R., p. 621).
Potassium nitrate (Macfadyen, 1830; Gesch., p. 423).
Potassium-ammonium carbonate (English patent, number unknown).

IV. HYDROGEN, OXYGEN, HALOGENS, NITROGEN, THEIR DERIVATIVES AND ACIDS, ALSO THEIR COMPOUNDS

Hydrogen peroxide and kieselguhr (Bachler, Z., 66, p. 504).
Hydrogen peroxide with activated carbon and "Silica-gel" (Roessler, C. b., 1935, p. 2464).

TABLE 51a (*Continued*)

Hydrogen peroxide and ammonium persulphate (Adolph, D. Z., 1936, p. 796).
Carbonyl hydride (?) and wood charcoal (Trowbridge, Chz., 1909, p. 989).
Nascent oxygen (Larronde, Z., 6A, p. 266).
Oxygen or air under pressure (Meyer, D. Z., 1923, p. 325).
*Ozone (Lundgren, Z., 63, p. 1092; Minz, Z. B., 16, R., 8).
Ozone with CO_2 under pressure (Delafond, S. B., 1924, p. 81).
Chlorine with decolorizing carbon (Shuichiro and Ochi, C., 1926, p. 3426).
Chlorine and Norit (Mintz and Chartakoff, S. B., 1932, p. 457).
Activated chlorine (?) with phosphoric acid, phosphates, borates, or oxalates, and hydrogen peroxide (Sanchez, C. b., 1936, p. 1630).
*Hydrochloric acid (Fremy, 1811, S. ind., 81, p. 512).
Alkali oxychloride with calcium carbonate and phosphoric acid (Wachtel, Z., 63, p. 1094).
Chlorine oxides (Murmann, C., 1924, B., p. 2547).
Iodine (Larrondo, S. ind., 84, p. 18).
Potassium iodate (*ibid.*).
*Hydrofluoric acid (Verbiese, B., 17, p. 309).
Nitrogen textroxide (Patent, C. b., 1927, p. 2241).

V. ALKALIS, ALKALINE EARTHS AND THEIR COMPOUNDS

*Ammonia (King, I. S. J., 1931, p. 141).
*Ammonium sulphide (Harvester, S. C., II, 18, p. 341).
Ammonium carbonate (Schwilger, C. b., 1931, p. 1941).
Ammonium bicarbonate (*ibid.*, C. b., 1931, p. 2251).
Ammonium and magnesium acetate (Joshida, S. C., II, 23, p. 282).
*Potassium or sodium sulphide (Horne, S. C., II, 18, p. 341).
Calcium oxide and limestone in powder (Stern and Carniol, Oe., 41, p. 531).
Calcium chloride with calcium hydrate and hydrochloric acid (Arsem, Z., 78, p. 283).
Calcium chloride with sodium phosphate and carbon (C., 1934, p. 3669).
Barium sulphate (Kangun, C. Z., 1924, p. 1349).
Barium trisilicate (Deguide, C., 1926, p. 3283).
*Magnesium oxide (Loginov, C., 1934, p. 2503).
Magnesium oxide with sulphurous acid (Meyer, Z., 82, p. 324).
*Magnesium oxide hydrate with sulphuric acid and lime (Meyer, Z., 82, p. 324).
*Dolomite, calcined (Loginov, C., 1934, p. 2503).
*Magnesium calcium oxide hydrate (Stewart, Z., 62, p. 1166).
*Magnesium carbonate (Hood and Clark, Z., 71, p. 844).
*Magnesium bicarbonate (Andrlik and Kohn, Z. B., II, 3, p. 404).
Magnesium polysulphide (Hlavati, Z., 60, p. 415).
Asbestos (Blomley, C. Z., 1922, p. 1033).
Magnesium acetate (Ishida, C. Z., 1925, p. 1607).

VI. METALS AND METALLIC COMPOUNDS

*Aluminium alloys with copper or zinc dust (Devos, Chz., 37, R., p. 107).
Aluminium chloride with calcium phosphate and magnesia (Hood and Clark, Z., 75, p. 942).
Aluminium oxychloride (Bomonti, C., 1936, p. 2646).
Aluminium nitrate (Hood, Z., 71, p. 844).
Basic aluminium carbonate (Hunyadi, C., 1922, p. 274).
Colloidal alumina and barium oxide and hydrate (Zameron, Chz., 1931, R., p. 151).
Colloidal alumina and active carbon (Traube, Z., 86, p. 186).
Aluminium hydrate with calcium sulphate (Kamous, Z. B., II, 5, p. 228).
Aluminium hydrate and sulphurous acid (Singh, Z., 76, p. 690).
*Sodium aluminate with sulphurous acid (Wayne, C., 1931, p. 646; Nicholson and Beale, Z., 77, p. 579; Faith and Sartorius, D. Z., 1936, p. 792).
Sodium aluminate (Wayne, C., 1933, p. 1037).
*Calcium aluminate (Lewitzki, J. fabr., 53, p. 18).
Aluminium sulphate and sodium sulphite (Hazewinkel, D. Z., 1910, p. 336).

TABLE 51a (*Continued*)

Ammonia alum (Brucher about 1860; Viviers, J. fabr., 60, p. 23).
Aluminium tannate (Feng and Wilbrow, S. B., 1930, p. 64).
Aluminium sulphide and polysulphide (Hlavati, Chz., 27, p. 254; Z., 60, p. 415).
Bauxite (Hood, Z., 71, p. 844; Hardy, I. S. J., 1933, p. 64).
*Ferrous sulphate (Oehlrichs, Z., 64, p. 802; Barnet, Z., 73, p. 437).
*Potassium ferrocyanide with sulphur dioxide (Arnal, C., 1927, p. 225; p. 2080).
Ferrous sulphocyanide (Hebden, D. Z., 1926, p. 1044).
*Manganese oxide (Manoury, Z., 64, p. 822).
*Lead acetate (de Villele, C., 1931, p. 374).
Zinc dust with oxalic acid and sinc sulphate (Jamazaki, Z., 74, p. 914).
Zinc dust with alcohol and ammonia (Leuchtenberger, 1887, in Newlands, p. 649).
Colloidal zinc with additions (C. b., 1933, p. 1612).
Zinc sulphate with potassium ferrocyanide (Thompson, 1898, in Newlands, p. 649).
Tin in pieces with coke and wood charcoal (Rambousek, Chz., 33, R., p. 507).
*Tin fluoride (Tiemann, Z., 64, p. 53).
Mercury iodide with potassium iodide (Haddon, Z. B., II, 9, p. 6).
Antimony polysulphide (Hlavati, Z., 60, p. 415).
Phosphomolybdic acid (Pohlmann, C., 1928, p. 195).
Phosphotungstic acid (Pohlmann, *ibid.*).

VII. ORGANIC SUBSTANCES AND THEIR COMPOUNDS; BONEBLACK AND ITS SUBSTITUTES

Tannic acid with iron and titanium (Hebden, D. Z., 51, p. 1044).
*Tannic acid with glue, starch, or albumin (Davis, Z., 18, p. 225).
Oxalic acid with barium carbonate and permanganates (Talamo, N. Z., 29, p. 210).
Oxalic acid with iron and antimony (Tiemann, Z., 63, p. 326).
Tin oxalate (Tiemann, Z., ang., 1913, p. 104).
Tin oxalate and charcoal (Tiemann, Chz., 37, R., p. 573).
Picric acid (Pohlmann, C., 1928, p. 195).
Formaldehyde with kieselguhr and alumina (C., 1933, p. 463).
Formaldehyde sulphonic acid (Straatman, S. C., II, 24, p. 22).
Alcohol with acetic ether and sulphuric acid (Vazquez, C., 1933, p. 522).
*Alcohol with sulphurous acid (Larrondo, S. ind., 84, p. 18).
Glycol (Bloch, C., 1893, p. 1445).
Glycerin with sulphurous acid (Carballeda, Chz., 38, R., p. 162; Barrios, Z., 66, p. 504).
Glycerin with sulphuric acid (Carballeda, Z., 63, p. 268).
Acetone (Andriessen and Schweizer, S. B., 56, p. 12).
Methyl, ethyl, or butyl acetates with acetic acid (C., 1927, p. 648).
Invert sugar (Tiemann, Z., ang., 1914, p. 500).
*Glucose and its salts (Wolvekamp, Z., 61, p. 103).
Benzine, naphtha, and petroleum ether (Seo, C., 1927, p. 240).
Petroleum distillate, bitumen, asphalt, ozokerite Meiro, Z., 84, p. 556).
Solid mineral oil (Nowakowski, C. Z., 17, p. 266).
*Hydrocarbons as petroleum (Meiro, C., 1932, p. 2104).
Lime and lead soaps (Prins, C. Z., 35, p. 885).
Nitrated rosin oils (Bauer, D. Z., 42, p. 26).
Colophony and balsam nitrate (Bauer, Z., 66, p. 634).
Paraffin oil with carbon tetrachloride (Hapgood, C. Z., 32, p. 1059).
Asphalt oils, sulphonates (Staegemann, C., 1931, p. 2520).
*Casein (Fleshman, C., 1936, p. 1735).
Gelatin and animal glue (Pajot, C., 1934, p. 2144).
Gelatin, pectate, and starch (Fleshman, C., 1936, p. 1735).
Pepsin with hydrochloric acid (Schreiber, C., 1926, p. 666).
Pepsin and bromelin (Fleshman, Z., 1935, p. 3449).
Alginic acid from algae (Allenet, Chz., 106, R., p. 180).
Straw (Lenze, D. Z., 33, p. 937).
Maize stalks (Redfern, 1890, in Newlands, p. 657).
Humus substances (Wellensieck, D. Z., 36, p. 832).

TABLE 51*a* (*Continued*)

Humus-containing earths, as black Russian soil (Kochan and Wostrukow, C. b., 1933 p. 3591).
*Wood charcoal (Thompson, 1888, in Newlands, p. 656).
Plant charcoal treated with alkali and acid (Lotz, Z., 62, p. 1140).
Plant charcoal treated with chlorine (Molenda, 62, p. 379).
Carbonized bagasse (Clacher, S. C., 16, p. 64).
Carbonized sawdust (Ck., C. Z., 44, p. 663).
Alumina carbonized with petroleum residues (Chaseborough, 1898, in Newlands, p. 656).
Colloidal graphite (Hugel, Z., 1936, p. 4087).
*Natural zeolite (Urban, Z., 76, p. 690).
Carbonation scums with soda or lime (Z., 59, p. 700).

VIII. ELECTROLYTIC SUBSTANCES

*Aluminium (Johnson, 1890, in Newlands, p. 647).
*Iron (Horsin-Déon, Bl. Ass., 16, p. 729).
Iron and zinc (Chubbin, Z., ang., 1914, p. 498).
Magnetic oxide of iron (Blyth, in Newlands, p. 654).
Air containing ozone (Consonno and Crespi, C. B., p. 2462).
Sulphurous acid and calcium hypochlorite (Wallace and Ventre, *ibid.*, p. 3005).
Aluminium sulphate and iron vitriol (Fr. P., Chz., 60, R., p. 175).
Sodium and aluminate (Faith and Sartorius, *ibid.*, p. 3605).
Aluminium and silicon hydrated gels (Fr. P., Chz., 60, R., p. 175).
Aluminium and silicon hydrated gels and formaldehyde (Fr. P., D. Z., 61, p. 750).
Kieselguhr (Herles, Z. B., 18, p. 94).
"Collactivit," or sawdust carbonized by sulphuric acid (D. Z., 61, p. 973; C. Z., 44, p. 663).

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