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CANE SUGAR AND ITS MANUFACTURE

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AND ITS
MANUFACTURE

BY

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PREFACE

To the First Edition

THE aim of the present work is to compile in one book everything that is known about the chemistry and the technology of the sugar cane and cane sugar manufacture.

In doing this, the discussion of technicalities relating to machinery has been carefully avoided, because this subject is already sufficiently dealt with in Noël Deerr's work, " Sugar and the Sugar Cane." Only those points are mentioned here which are indispensable for the proper understanding of the chemical and technical questions investigated in this work.

Although the author has chiefly made use of his own investigations and researches carried on during his 17 years' continuous stay in Java at the head of the West Java Sugar Experiment Station, he has also gladly and gratefully referred to the results of work done by his colleagues in other parts of the world. In every case, where reference is made to the work of others, this has been acknowledged in a foot-note.

The enumeration of the chemical and physical characteristics of the three kinds of sugar is chiefly borrowed from Prof. Dr. E. O. von Lippmann's excellent handbook, " Die Chemie der Zuckerarten."

In agreement with that distinguished scientist, the author has used in his book Fischer's nomenclature for the different kinds of sugar, because the terms *dextrose* and *levulose* are fast becoming obsolete in scientific literature and are apt to cause confusion.

In the present work the different terms have the following signification :—

SUGAR : The commercial product.

SUCROSE : The chemical body, the principal constituent of the commercial product.

GLUCOSE : The chemical body, also called dextrose.

FRUCTOSE : The chemical body, also called levulose.

INVERT SUGAR : The mixture of exactly equal proportions of glucose and fructose.

REDUCING SUGAR : Mixtures of uneven proportions of glucose and fructose.

The author has pleasure in acknowledging his great indebtedness to two gentlemen, Mr. James P. Ogilvie, Associate Editor of the *International Sugar Journal*, and Mr. T. H. P. Heriot—especially the latter—for a painstaking and laborious revision of the English text.

H. C. PRINSEN GEERLIGS.

AMSTERDAM, *May*, 1909.

PREFACE

To the Second Edition

WHEN it was found necessary to compile a second edition of this work, it was at once obvious that a simple revision or amplification of the first edition would be entirely insufficient.

Since 1911, when the first edition was issued, both the theory and the practice of the work done in the province of sugar manufacture have made such great strides that almost every chapter of the book has had to be re-modelled and re-written in order to be brought up to the present standard of the work.

Especially have the sections on JUICE EXTRACTION, CLARIFICATION, FILTRATION, PRESERVATION OF SUGAR AND MOLASSES, undergone radical changes and been brought more in line with modern views and their practical application.

The general trend of the book, however, which has hitherto been found practical and useful, has remained as it was put forward in the Preface to the First Edition.

I gratefully avail myself of this opportunity to offer my sincere thanks to my son, Mr. R. J. Prinsen Geerligs, for his valuable assistance in the compilation of the numerous data scattered throughout the literature of the subject, and their arrangement in this volume; and to Mr. Norman Rodger and Mr. James P. Ogilvie, F.I.C., for their thorough and painstaking editing of the manuscript, and for their careful reading of the proofs.

H. C. PRINSEN GEERLIGS, PH.D.

AMSTERDAM,
30th September, 1924.

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FIRST PART

THE RAW MATERIAL

CHAPTER I

THE CONSTITUENTS OF THE SUGAR CANE

I.—Cane Sugar or Sucrose

Occurrence in Nature.—Sucrose is found in a great many plants, dissolved in the sap. Although it may be considered as one of the most universal constituents of plants, only a few of them contain it in such a degree and so sparingly mixed with other bodies as to allow of its profitable extraction. Such are: the sugar cane; the beetroot; the sorghum; the coco, date, palmyra and nipah palms; and the maple tree. Then bees extract it from the flowers of many plants, but in this case it is soon transformed into invert sugar.

Crystalline Form.—Sucrose appears in the form of anhydrous, bright, monoclinic crystals, having a pure, sweet taste, and possessing a specific gravity of 1·585 at 15° C. Large crystals, candy for instance, which have crystallized out slowly from hot solutions, are formed by the combination of parallelic disposed lamellæ, which generally include parts of the mother-liquor in their interstices. Such a crystal can be split like a diamond along its axis by a sharp, heavy blow with a hammer. The occluded mother-liquor can cause coloration of the candy crystals; moreover, owing to its presence, candy, which externally is perfectly dry, on pulverization often yields a moist powder. When a broken crystal of sucrose is placed in a supersaturated solution of the same substance and allowed to grow there, the damaged portion will gradually become healed, until the original form of the crystal is restored; after that crystalline growth proceeds regularly, provided it is allowed to do so in every direction, as, for example, when the crystal is suspended by means of a thread in a supersaturated sucrose solution, which is thickened with isinglass or agar-agar, or when it is kept in regular and constant motion in a supersaturated solution.

Sucrose crystallizes from its aqueous solution in crystals, which are larger in proportion as the liquid in which they are formed contains a less amount of bodies other than sucrose and as the crystallization is slower. Very rapid crystallization or crystallization from syrups containing a large amount of foreign substances is apt to yield small sucrose crystals.

Further particulars concerning the crystallization of sucrose will be found in the chapter on *Boiling*.

Strongly supersaturated sucrose solutions when agitated will suddenly deposit very minute so-called "mealy" crystals, whilst steady agitation of moderately supersaturated sucrose solutions causes the crystals to grow as slowly and continuously as when at rest. In this case they become even more regular, because, owing to the constant motion, they are free in every direction and are able to move throughout the whole of the supersaturated liquid, which gives them a more regular crystalline form than if they had remained deposited on the bottom of the crystallization vessel.

Sucrose, even when it is in a pure state, sometimes crystallizes in an abnormal form from solutions containing certain foreign bodies, such as raffinose, zinc salts, some calcium salts, or products resulting from the overheating of sugar. The influence of these substances is, however, not always perceptible, as sucrose will sometimes crystallize in its usual form from solutions containing a large proportion of raffinose. The abnormality of the crystalline form of sucrose consists in the stretching of one of the axes of the crystal, which causes it to assume the shape of a needle. This becomes very obvious when the crystals combine into bundles.

Solubility in Water.—Sucrose is freely soluble in water and its solubility increases as the temperature rises. The values for the solubility of sucrose at different temperatures are recorded in the following table compiled by Herzfeld* :—

Temperature.	Per cent. of Sucrose.	Temperature.	Per cent. of Sucrose.	Temperature.	Per cent. of Sucrose.	Temperature.	Per cent. of Sucrose.	Temperature.	Per cent. of Sucrose.
°C.		°C.		°C.		°C.		°C.	
0	64·18	14	66·18	28	68·37	42	70·78	56	73·39
1	64·31	15	66·33	29	68·53	43	70·96	57	73·58
2	64·45	16	66·48	30	68·70	44	71·14	58	73·78
3	64·59	17	66·63	31	68·87	45	71·32	59	73·98
4	64·73	18	66·78	32	69·04	46	71·50	60	74·18
5	64·87	19	66·93	33	69·21	47	71·68	61	74·38
6	65·01	20	67·09	34	69·38	48	71·87	62	74·58
7	65·15	21	67·25	35	69·55	49	72·06	63	74·78
8	65·29	22	67·41	36	69·72	50	72·25	64	74·98
9	65·43	23	67·57	37	69·89	51	72·44	65	75·18
10	65·58	24	67·73	38	70·06	52	72·63	66	75·38
11	65·73	25	67·89	39	70·24	53	72·82	67	75·59
12	65·88	26	68·05	40	70·42	54	73·01	68	75·80
13	66·03	27	68·21	41	70·60	55	73·20	69	76·01

* "Zeitschr. Rübenzuckerind." 1892, 181

Temperature.	Per cent. of Sucrose.	Temperature.	Per cent. of Sucrose.	Temperature.	Per cent. of Sucrose.	Temperature.	Per cent. of Sucrose.	Temperature.	Per cent. of Sucrose.
°C.		°C.		°C.		°C.		°C.	
70	76·22	77	77·70	84	79·24	91	80·84	98	82·49
71	76·43	78	77·92	85	79·46	92	81·07	99	82·73
72	76·64	79	78·14	86	76·69	93	81·30	100	82·79
73	76·85	80	78·36	87	79·92	94	81·53	—	—
74	77·06	81	78·58	88	80·15	95	81·77	—	—
75	77·27	82	78·80	89	80·38	96	82·01	—	—
76	77·48	83	79·02	90	80·61	97	82·25	—	—

General formula for the solubility: $y = 64{\cdot}1835 + 0{\cdot}13477\,x + 0{\cdot}0005307x^2$. y = per cent. of sugar, and x = temp. in degrees C.

Contraction on being dissolved in Water.—When sucrose is dissolved in water a considerable contraction takes place; this reaches its maximum at 56 per cent., as may be seen from the following table* :—

Sucrose, Grms.	Water, Grms.	Volume, c.c.	Sucrose, Grms.	Water, Grms.	Volume, c.c.
0	100	100·0000	55·0	45·0	99·0059
10	90	99·7218	55·9	44·1	99·0055
20	80	99·4732	56·0	44·0	99·0042
30	70	99·2822	56·1	43·9	99·0055
40	60	99·1103	57·0	43·0	99·0059
50	50	99·0219	—	—	—
60	40	99·0121	—	—	—
70	30	99·0921	—	—	—
80	20	99·2756	—	—	—
90	10	99·5745	—	—	—
100	0	100·0000	—	—	—

* E. O. von Lippmann "Chemie der Zuckerarten" II 1081.

Sp. Gr. of Sucrose Solutions.—According to Brix tables the specific gravity of sucrose solutions is at a temperature of 15° C. as follows:—

Per Cent.	Specific Gravity.	Per Cent.	Specific Gravity.	Per Cent.	Specific Gravity.
1	1·00388	23	1·09686	45	1·20565
2	1·00779	24	1·10145	46	1·21100
3	1·01173	25	1·10607	47	1·21639
4	1·01570	26	1·11072	48	1·22182
5	1·01970	27	1·11541	49	1·22728
6	1·02373	28	1·12013	50	1·23278
7	1·02779	29	1·12488	51	1·23832
8	1·03187	30	1·12967	52	1·24390
9	1·03599	31	1·13449	53	1·24951
10	1·04014	32	1·13934	54	1·25571
11	1·04431	33	1·14423	55	1·26086
12	1·04852	34	1·14915	56	1·26658
13	1·05276	35	1·15411	57	1·27235
14	1·05703	36	1·15911	58	1·27816
15	1·06133	37	1·16413	59	1·28400
16	1·06566	38	1·16920	60	1·28989
17	1·07002	39	1·17430	61	1·29581
18	1·07441	40	1·17934	62	1·30177
19	1·07884	41	1·18460	63	1·30777
20	1·08329	42	1·18981	64	1·31381
21	1·08778	43	1·19505	65	1·31989
22	1·09231	44	1·20033	66	1·32601

Gerlach calculated the following formula for the specific gravity of x per cent. of sucrose dissolved at 17·5° C.:—

$$y = 1 + 0{\cdot}00386571327\,x + 0{\cdot}00001414091916\,x^2 + 0{\cdot}0000003287946571 76\,x^3$$

from which values Scheibler computed the following interpolation formulæ:—

$t = 0°, y = 1 + 0{\cdot}003976844\,x + 0{\cdot}0000142764\,x^2 + 0{\cdot}000000029120\,x^3$
$t = 10°, y = 1 + 0{\cdot}003915138\,x + 0{\cdot}0000139524\,x^2 + 0{\cdot}000000032728\,x^3$
$t = 15°, y = 1 + 0{\cdot}003884496\,x + 0{\cdot}0000139399\,x^2 + 0{\cdot}000000033806\,x^3$
$t = 20°, y = 1 + 0{\cdot}003844136\,x + 0{\cdot}0000144092\,x^2 + 0{\cdot}000000030912\,x^3$
$t = 30°, y = 1 + 0{\cdot}003796428\,x + 0{\cdot}0000145456\,x^2 + 0{\cdot}000000030664\,x^3$
$t = 40°, y = 1 + 0{\cdot}003764028\,x + 0{\cdot}0000143700\,x^2 + 0{\cdot}000000035192\,x^3$
$t = 50°, y = 1 + 0{\cdot}003722992\,x + 0{\cdot}0000148080\,x^2 + 0{\cdot}000000032440\,x^3$
$t = 60°, y = 1 + 0{\cdot}003683112\,x + 0{\cdot}0000155904\,x^2 + 0{\cdot}000000026368\,x^3$

Although yet other formulæ have been suggested, those mentioned here are quite sufficient for ordinary purposes.

Influence of Temperature on the Sp. Gr.—The specific gravity of sucrose solutions diminishes as the temperature rises, because the rise in temperature increases their volume. Gerlach determined the following values for the specific gravity of solutions of different concentrations, at various temperatures, in which water at 17·5° C. = 1.

°C.	0 per cent. Sucrose.	15 per cent. Sucrose.	30 per cent. Sucrose.	45 per cent. Sucrose.	60 per cent. Sucrose.	75 per cent. Sucrose.
0	1·0007	1·0636	1·1337	1·2113	1·2972	1·3916
20	0·9996	1·0606	1·1288	1·2046	1·2889	1·3822
40	0·9942	1·0504	1·1212	1·1958	1·2794	1·3722
60	0·9857	1·0448	1·0881	1·1851	1·2683	1·3610
80	0·9745	1·0336	1·0768	1·1729	1·2562	1·3488
100	0·9621	1·0202	1·0634	1·1597	1·2424	1·3356

At the 6th Session of the International Commission for the Unification of Methods of Sugar Analysis held in London, on May 31st, 1909, it was resolved to adopt universally in sugar analytical methods the tables for the specific gravity of sucrose solutions prepared by the Imperial German Normal Standardization Commission, in which that of water at 4° C. was taken as unity, and the sucrose solutions are tested at 20° C. (See pages 8 and 9).

For calculating the percentage of sucrose in solutions tested at temperatures deviating from 20° C., the table on page 10 may be used, calculated by Lange from the figures found by the Imperial German Normal Standardization Commission.

Boiling Point of Aqueous Sucrose Solutions.—According to Gerlach's researches, the boiling point of aqueous sucrose solutions of different concentration is at the ordinary atmospheric pressure:—

Per cent. Sucrose	10	20	30	40	50	60	70	80	90·8
Temperature	100·4	100·6	101·0	101·5	102·0	103·0	106·5	112·0	130·0° C.

Solubility of Sucrose in Presence of Invert Sugar.—T. van der Linden* gives a table for the solubility of sucrose in water when invert sugar is present simultaneously at temperatures of 30° and 50° C, of which the averages are reproduced on page 11.

* "Archief voor de Java Suikerindustrie," 1919, 594.

TABLE FOR FINDING THE CONTENT OF PURE SUCROSE FROM THE TRUE DENSITY AT 20° C. WITH WATER AT 4° C. AS UNITY.

Per-cent Sucrose.	0·0	0·1	0·2	0·3	0·4	0·5	0·6	0·7	0·8	0·9
	True density at 20° C. of the units at the left-hand and the tenths above.									
0	0·998234	0·998962	0·999010	0·999398	0·999786	1·000174	1·000563	1·000952	1·001342	1·001731
1	1·002120	1·002509	1·002897	1·003286	1·003675	1·004064	1·004453	1·004844	1·005234	1·005624
2	1·006015	1·006405	1·006796	1·007188	1·007580	1·007972	1·008363	1·008755	1·009148	1·009541
3	1·009934	1·010327	1·010721	1·011115	1·011510	1·011904	1·012298	1·012694	1·013089	1·013485
4	1·013881	1·014277	1·014673	1·015070	1·015467	1·015864	1·016261	1·016659	1·017058	1·017456
5	1·017854	1·018253	1·018652	1·019052	1·019451	1·019851	1·020251	1·020651	1·021053	1·021454
6	1·021855	1·022257	1·022659	1·023061	1·023463	1·023867	1·024270	1·024673	1·025077	1·025481
7	1·025885	1·026289	1·026694	1·027099	1·027504	1·027910	1·028316	1·028722	1·029128	1·029535
8	1·029942	1·030349	1·030757	1·031165	1·031573	1·031982	1·032391	1·032800	1·033209	1·033619
9	1·034029	1·034439	1·034850	1·035260	1·035671	1·036082	1·036494	1·036906	1·037318	1·037730
10	1·038143	1·038556	1·038970	1·039383	1·039797	1·040212	1·040626	1·041041	1·041456	1·041872
11	1·042288	1·042704	1·043121	1·043537	1·043954	1·044370	1·044788	1·045206	1·045625	1·046043
12	1·046462	1·046881	1·047300	1·047720	1·048140	1·048559	1·048980	1·049401	1·049822	1·050243
13	1·050665	1·051087	1·051510	1·051933	1·052356	1·052778	1·053202	1·053626	1·054050	1·054475
14	1·054900	1·055325	1·055751	1·056176	1·056602	1·057029	1·057455	1·057882	1·058310	1·058737
15	1·059165	1·059593	1·060022	1·060651	1·060880	1·061208	1·061738	1·062168	1·062598	1·063029
16	1·063460	1·063892	1·064324	1·064756	1·065188	1·065621	1·066054	1·066487	1·066921	1·067355
17	1·067789	1·068223	1·068658	1·069093	1·069529	1·069964	1·070400	1·070836	1·071273	1·071710
18	1·072147	1·072585	1·073023	1·073461	1·073900	1·074338	1·074777	1·075217	1·075657	1·076097
19	1·076537	1·076978	1·077419	1·077860	1·078302	1·078744	1·079187	1·079629	1·080072	1·080515
20	1·080959	1·081403	1·081848	1·082292	1·082737	1·083182	1·083628	1·084074	1·084520	1·084967
21	1·085414	1·085861	1·086309	1·086757	1·087205	1·087652	1·088101	1·088550	1·089000	1·089450
22	1·089900	1·090351	1·090802	1·091253	1·091704	1·092155	1·092607	1·093060	1·093513	1·093966
23	1·094420	1·094874	1·095328	1·095782	1·096236	1·096691	1·097147	1·097603	1·098058	1·098514
24	1·098971	1·099428	1·099886	1·100344	1·100802	1·101259	1·101718	1·102177	1·102637	1·103097
25	1·103557	1·104017	1·104478	1·104938	1·105400	1·105862	1·106324	1·106786	1·107248	1·107711
26	1·108175	1·108639	1·109103	1·109568	1·110033	1·110497	1·110963	1·111429	1·111895	1·112361
27	1·112828	1·113295	1·113863	1·114229	1·114697	1·115166	1·115635	1·116104	1·116572	1·117042
28	1·117512	1·117982	1·118453	1·118923	1·119395	1·119867	1·120339	1·120812	1·121284	1·121757
29	1·122231	1·122705	1·123179	1·123653	1·124128	1·124603	1·125079	1·125555	1·126030	1·126507
30	1·126984	1·127461	1·127939	1·128417	1·128896	1·129374	1·129853	1·130332	1·130812	1·131292
31	1·131773	1·132254	1·132735	1·133216	1·133698	1·134180	1·134663	1·135146	1·135628	1·136112
32	1·136596	1·137080	1·137565	1·138049	1·138534	1·139020	1·139506	1·139993	1·140479	1·140966
33	1·141453	1·141941	1·142429	1·142916	1·143405	1·143894	1·144384	1·144874	1·145363	1·145854
34	1·146345	1·146836	1·147328	1·147820	1·148313	1·148805	1·149298	1·149792	1·150286	1·150780
35	1·151275	1·151770	1·152265	1·152700	1·153256	1·153752	1·154249	1·154746	1·155242	1·155740
36	1·156238	1·156736	1·157235	1·157733	1·158233	1·158733	1·159233	1·159733	1·160233	1·160734
37	1·161236	1·161738	1·162240	1·162742	1·163245	1·163748	1·164252	1·164756	1·165259	1·165764
38	1·166269	1·166775	1·167281	1·167786	1·168293	1·168800	1·169307	1·169815	1·170322	1·170831
39	1·171340	1·171849	1·172359	1·172869	1·173379	1·173889	1·174400	1·174911	1·175423	1·175935
40	1·176447	1·176960	1·177473	1·177987	1·178501	1·179014	1·179527	1·180044	1·180560	1·181076
41	1·181592	1·182108	1·182625	1·183142	1·183660	1·184178	1·184696	1·185215	1·185734	1·186253
42	1·186773	1·187293	1·187814	1·188335	1·188856	1·189379	1·189901	1·190423	1·190946	1·191469
43	1·191993	1·192517	1·193041	1·193565	1·194090	1·194616	1·195141	1·195667	1·196193	1·196720
44	1·197247	1·197775	1·198303	1·198832	1·198360	1·199890	1·200420	1·200950	1·201480	1·202010
45	1·202540	1·203071	1·203603	1·204136	1·204668	1·205200	1·205733	1·206266	1·206801	1·207335
46	1·207870	1·208405	1·280940	1·209477	1·210013	1·210549	1·211086	1·211623	1·212162	1·212700
47	1·213238	1·213777	1·214317	1·214856	1·215395	1·215936	1·216476	1·217017	1·217559	1·218101
48	1·218643	1·219185	1·219729	1·220272	1·220815	1·221360	1·221904	1·222449	1·222995	1·223540

Per cent Sucrose.	0·0	0·1	0·2	0·3	0·4	0·5	0·6	0·7	0·8	0·9
	True density at 20° C. of the units at the left-hand and the tenths above.									
49	1·224086	1·224632	1·225180	1·225727	1·226274	1·226823	1·227371	1·227919	1·228469	1·229018
50	1·229567	1·230117	1·230668	1·231219	1·231770	1·232322	1·232874	1·233426	1·233979	1·234532
51	1·235085	1·235639	1·236194	1·236748	1·237303	1·237859	1·238414	1·238970	1·239527	1·240084
52	1·240641	1·241198	1·241757	1·242315	1·242873	1·243433	1·243992	1·244552	1·245113	1·245673
53	1·246234	1·246795	1·247358	1·247920	1·248482	1·249046	1·249609	1·250172	1·250737	1·251301
54	1·251866	1·252431	1·252997	1·253563	1·254129	1·254697	1·255264	1·255831	1·256400	1·256967
55	1·257535	1·258104	1·258674	1·259244	1·259815	1·260385	1·260955	1·261527	1·262099	1·262671
56	1·263243	1·263816	1·264390	1·264963	1·265537	1·266112	1·266686	1·267261	1·267837	1·268413
57	1·268989	1·269565	1·270143	1·270720	1·271299	1·271877	1·272455	1·273035	1·273614	1·274194
58	1·274774	1·275354	1·275936	1·276517	1·277098	1·277680	1·278262	1·278844	1·279428	1·280011
59	1·280595	1·281179	1·281764	1·282349	1·282935	1·283521	1·284107	1·284694	1·285281	1·285869
60	1·286456	1·287044	1·287633	1·288222	1·288811	1·289401	1·289991	1·290581	1·291172	1·291763
61	1·292354	1·292946	1·293539	1·294131	1·294725	1·295318	1·295911	1·296506	1·297100	1·297696
62	1·298291	1·298886	1·299483	1·300079	1·300677	1·301274	1·301871	1·302470	1·303068	1·303668
63	1·304267	1·304867	1·305467	1·306068	1·306669	1·307271	1·307872	1·308475	1·309077	1·309680
64	1·310282	1·310855	1·311489	1·312093	1·312699	1·313304	1·313909	1·314515	1·315121	1·315728
65	1·316334	1·316941	1·317549	1·318157	1·318766	1·319374	1·319983	1·320593	1·321203	1·321814
66	1·322425	1·323036	1·323648	1·324259	1·324872	1·325484	1·326097	1·326711	1·327325	1·327940
67	1·328554	1·329170	1·329785	1·330401	1·331017	1·331633	1·332250	1·332868	1·333485	1·334103
68	1·334722	1·335342	1·335961	1·336581	1·338200	1·337821	1·338441	1·339063	1·339684	1·340306
69	1·340928	1·341551	1·342174	1·342798	1·343421	1·344046	1·344671	1·345296	1·345922	1·346547
70	1·347174	1·347801	1·348427	1·349055	1·349682	1·350311	1·350939	1·351568	1·352197	1·352827
71	1·353456	1·355087	1·354717	1·355349	1·355980	1·356612	1·357245	1·357877	1·358511	1·359144
72	1·359978	1·360413	1·361047	1·361682	1·362317	1·362953	1·363590	1·364226	1·364864	1·365501
73	1·366139	1·366777	1·367415	1·368054	1·368693	1·369333	1·369973	1·370613	1·371254	1·371894
74	1·372536	1·373178	1·373820	1·374463	1·375105	1·375749	1·376392	1·377036	1·377680	1·378326
75	1·378971	1·379617	1·380262	1·380909	1·381555	1·382203	1·382851	1·383499	1·384148	1·384796
76	1·385446	1·386096	1·386745	1·387396	1·388045	1·388696	1·389347	1·389999	1·390651	1·391303
77	1·391956	1·392610	1·393263	1·393917	1·394571	1·395226	1·395881	1·396536	1·397192	1·397848
78	1·398505	1·399162	1·399819	1·400477	1·401134	1·401793	1·402452	1·403111	1·403771	1·404430
79	1·405091	1·405752	1·406412	1·407074	1·407735	1·408398	1·409061	1·409723	1·410387	1·411051
80	1·411715	1·412380	1·413044	1·413709	1·414374	1·415040	1·415706	1·416373	1·417039	1·417707
81	1·418374	1·419043	1·429711	1·420380	1·421049	1·421719	1·422390	1·423059	1·423730	1·424400
82	1·425072	1·425744	1·426416	1·427089	1·427761	1·428435	1·429109	1·429782	1·430457	1·431131
83	1·431807	1·432483	1·433158	1·433835	1·434511	1·435188	1·435866	1·436543	1·437222	1·437900
84	1·438579	1·439259	1·439938	1·440619	1·441299	1·441980	1·442661	1·443342	1·444024	1·444705
85	1·445388	1·446071	1·446754	1·447438	1·448121	1·448806	1·449491	1·450175	1·450860	1·451545
86	1·452232	1·452919	1·453605	1·454292	1·454980	1·455668	1·456357	1·457045	1·457735	1·458424
87	1·459114	1·459805	1·460495	1·461186	1·461877	1·462568	1·463260	1·463953	1·464645	1·465338
88	1·466032	1·466726	1·467420	1·468115	1·468810	1·469504	1·470200	1·470896	1·471592	1·472289
89	1·472986	1·473684	1·474381	1·475080	1·475779	1·476477	1·477176	1·477876	1·478575	1·479275
90	1·479976	1·480677	1·481378	1·482080	1·482782	1·483484	1·484187	1·484890	1·485593	1·486297
91	1·487002	1·487707	1·488411	1·489117	1·489823	1·490528	1·491234	1·491941	1·492647	1·493355
92	1·494063	1·494771	1·495479	1·496188	1·496897	1·497606	1·498316	1·499026	1·499736	1·500447
93	1·501158	1·501870	1·502582	1·503293	1·504006	1·504719	1·505432	1·506146	1·506859	1·507574
94	1·508289	1·509004	1·509720	1·510435	1·511151	1·511868	1·512585	1·513302	1·514019	1·514737
95	1·515455	1·516174	1·516893	1·517612	1·518332	1·519051	1·519871	1·520492	1·521212	1·521934
96	1·522656	1·523378	1·524100	1·524823	1·525546	1·526269	1·526993	1·527717	1·528441	1·529166
97	1·529891	1·530616	1·531342	1·532068	1·532794	1·533521	1·534248	1·534976	1·535704	1·536432
98	1·537161	1·537889	1·538618	1·539347	1·540076	1·540806	1·541536	1·542267	1·542998	1·543730
99	1·544462	1·545194	1·545926	1·546659	1·547292	1·548127	1·548861	1·549595	1·550329	1·551064
100	1·551800									

Temperature in °C.	Degrees Brix.														
	0	5	10	15	20	25	30	35	40	45	50	55	60	65	70
	Subtract from the Readings.														
0	0·28	0·47	0·63	0·76	0·87	0·97	1·06	1·15	1·24	1·30	1·36	1·40	1·43	1·45	1·48
5	0·35	0·45	0·55	0·64	0·72	0·79	0·85	0·91	0·97	1·01	1·04	1·07	1·09	1·11	1·13
10	0·32	0·37	0·42	0·47	0·51	0·56	0·60	0·64	0·67	0·69	0·71	0·73	0·74	0·75	0·76
15	0·20	0·21	0·24	0·26	0·28	0·30	0·31	0·33	0·35	0·36	0·37	0·37	0·38	0·38	0·39
16	0·16	0·18	0·19	0·21	0·23	0·24	0·25	0·27	0·28	0·29	0·29	0·30	0·30	0·31	0·31
17	0·12	0·13	0·15	0·16	0·17	0·18	0·19	0·20	0·21	0·22	0·22	0·22	0·23	0·23	0·23
18	0·08	0·09	0·10	0·11	0·12	0·12	0·13	0·13	0·14	0·15	0·15	0·15	0·15	0·15	0·16
19	0·04	0·05	0·05	0·06	0·06	0·06	0·07	0·07	0·07	0·08	0·08	0·08	0·08	0·08	0·08
	Add to the Readings.														
21	0·05	0·05	0·06	0·06	0·06	0·06	0·07	0·07	0·07	0·07	0·07	0·08	0·08	0·08	0·08
22	0·10	0·11	0·12	0·12	0·13	0·13	0·14	0·14	0·14	0·15	0·15	0·15	0·16	0·16	0·16
23	0·15	0·16	0·17	0·18	0·19	0·20	0·21	0·22	0·22	0·22	0·23	0·23	0·23	0·24	0·24
24	0·21	0·22	0·23	0·24	0·26	0·26	0·28	0·29	0·30	0·30	0·30	0·31	0·31	0·32	0·32
25	0·27	0·28	0·30	0·31	0·32	0·33	0·35	0·36	0·37	0·38	0·38	0·39	0·39	0·40	0·39
26	0·33	0·34	0·36	0·38	0·39	0·41	0·42	0·44	0·45	0·45	0·46	0·47	0·47	0·48	0·47
27	0·40	0·40	0·42	0·44	0·46	0·48	0·50	0·51	0·53	0·53	0·54	0·55	0·55	0·56	0·56
28	0·46	0·47	0·49	0·51	0·54	0·55	0·58	0·59	0·61	0·61	0·62	0·63	0·64	0·64	0·64
29	0·53	0·54	0·56	0·58	0·61	0·63	0·65	0·67	0·69	0·70	0·70	0·71	0·72	0·72	0·72
30	0·60	0·61	0·63	0·66	0·68	0·71	0·73	0·75	0·77	0·78	0·79	0·80	0·80	0·80	0·80
31	0·68	0·68	0·70	0·73	0·76	0·79	0·81	0·83	0·85	0·86	0·87	0·88	0·88	0·89	0·89
32	0·75	0·76	0·78	0·81	0·84	0·87	0·90	0·92	0·94	0·95	0·95	0·96	0·97	0·97	0·97
33	0·83	0·83	0·86	0·89	0·92	0·95	0·98	1·00	1·02	1·03	1·04	1·05	1·05	1·05	—
34	0·91	0·91	0·94	0·97	1·01	1·04	1·07	1·09	1·10	1·11	1·12	1·13	1·14	1·14	—
35	0·99	0·99	1·02	1·06	1·09	1·13	1·16	1·17	1·19	1·20	1·21	1·22	1·22	1·22	—
36	1·07	1·08	1·10	1·14	1·18	1·21	1·24	1·26	1·28	1·29	1·29	1·30	1·31	1·30	—
37	1·15	1·16	1·19	1·23	1·27	1·30	1·33	1·35	1·36	1·37	1·38	1·39	1·39	1·39	—
38	1·24	1·25	1·28	1·32	1·36	1·39	1·42	1·44	1·45	1·46	1·47	1·48	1·48	1·47	—
39	1·32	1·34	1·37	1·41	1·45	1·48	1·51	1·52	1·54	1·55	1·55	1·56	1·56	1·56	—
40	1·41	1·43	1·46	1·50	1·54	1·57	1·60	1·61	1·63	1·64	1·64	1·65	1·65	1·65	—
42	1·60	1·62	1·66	1·69	1·73	1·76	1·78	1·80	1·81	1·82	1·82	1·83	1·83	1·82	—
44	1·80	1·82	1·85	1·89	1·93	1·95	1·97	1·99	2·00	2·00	2·01	2·01	2·01	2·00	—
46	2·01	2·03	2·06	2·10	2·13	2·15	2·17	2·18	2·19	2·19	2·19	2·19	2·19	2·17	—
48	2·23	2·25	2·27	2·31	2·34	2·36	2·37	2·38	2·38	2·38	2·38	2·38	2·37	2·35	—
50	2·46	2·47	2·50	2·53	2·55	2·57	2·58	2·58	2·58	2·57	2·57	2·57	2·55	2·54	—
55	3·05	3·07	3·08	3·11	3·12	3·12	3·12	3·10	3·08	3·07	3·06	3·05	3·02	3·00	—
60	3·69	3·71	3·73	3·73	3·72	3·70	3·67	3·64	3·61	3·59	3·57	3·54	3·50	3·46	—

Composition, Per Cent.			Sucrose, per 100 Parts of Water.
Sucrose.	Invert Sugar.	Water.	
	Temperature 30° C.		
66·68	1·70	31·04	214·85
65·43	3·22	30·90	211·75
61·57	8·02	30·07	204·75
55·73	15·71	27·92	199·60
47·40	26·92	25·84	183·45
45·25	30·39	24·88	181·85
39·88	36·90	23·39	170·50
36·44	41·99	22·24	163·85
29·21	51·45	19·38	150·70
24·92	56·62	18·22	136·80
20·22	63·87	15·91	127·10
	Temperature 50° C.		
62·21	13·39	24·54	253·45
57·60	18·26	23·85	241·45
48·35	30·45	21·05	229·70
43·46	37·90	19·05	228·15
37·16	45·58	17·25	215·40
32·65	51·15	16·20	201·55

Solubility of Sucrose in Alcohol, etc.—Sucrose is very sparingly soluble in absolute alcohol, viz., only one part sucrose dissolves in 80 parts of absolute alcohol at the boiling point; but dilute alcohol dissolves it much more readily, and this in proportion as the alcohol is more dilute and the temperature higher, as is shown in the following table compiled by Pellet.* The first column represents the c.c. of absolute alcohol present in 100 c.c. of the solution, and the other columns the grms. of sucrose dissolved in 100 c.c. of the mixture at temperatures up to 40° C.

In each case less sucrose is dissolved in a mixture of alcohol and water than could be dissolved at the same temperature in the amount of water present in the mixture, if it had not been mixed with alcohol.

Sucrose also dissolves in dilute methyl alcohol, in hot glacial acetic acid, but not in chloroform, ether and similar solvents; and it is practically insoluble in anhydrous glycerine.

* "Bull. Assoc. Chim. Sucr. et Dist.," 15, 631

c.c. Absolute Alcohol.	Grms. Sucrose in 100 c.c. at				
	0°	10°	20°	30°	40°
0	85·5	87·3	88·3	91·8	94·2
10	80·7	82·5	85·0	87·0	90·0
20	74·2	76·5	79·0	82·0	84·0
30	65·5	68·0	71·0	74·0	77·5
40	56·7	59·0	62·0	65·5	69·0
50	45·9	48·5	52·0	55·6	60·0
60	32·9	35·5	39·0	42·5	47·5
70	18·2	20·0	22·5	26·0	31·0
80	6·4	7·2	8·1	10·1	14·0
90	0·7	0·8	1·1	1·6	2·3
97·4	0·08	0·15	0·25	0·36	0·50

Action of Electric Current on Sucrose Solutions.—In aqueous solutions, sucrose is a non-conductor of electricity, but after a prolonged action of the current a slight conductivity is apparent. This, however, is not due to the sucrose itself but either to impurities in the water used for solution or from decomposition products of the sucrose, as the electric current, provided it is of sufficient power, will attack sucrose, inverting it with formation of acids.

Rotatory Power.—Aqueous sucrose solutions deflect the plane of the polarized light ray to the right with a specific rotatory power which is only insignificantly influenced by the concentration of the solution. Bates and Jackson* found the specific rotation of sucrose, at a concentration of 26 grms. per 100 c.c. and a temperature of 20° C. to be $\frac{a}{D} = 66{\cdot}529°$, while Kraisy and Traegl† found it to be 66·516°. According to Landolt, the specific rotatory power of sucrose is, for concentrations coming into consideration in sugar-house work (which never exceed 26 per cent‡), expressed in arc degrees :—

$$a\frac{20}{D} = 66{\cdot}435 + 0{\cdot}00870\,c - 0{\cdot}000235\,c^2$$

in which formula c stands for concentration.

* "Scientific Papers of the Bureau of Standards," No. 268

† "Zeitschr. Rübenzuckerind." 1924, 204.

‡ $\frac{26.048}{1.1} = 23.68$

Influence of Concentration on Rotatory Power.—By this formula, the figures for the rotation of sucrose for the different concentrations are as follows :

c.	Rotation.	*c.*	Rotation.	*c.*	Rotation.
1	66·443	25	66·506	50	66·283
5	66·473	30	66·485	55	66·203
10	66·499	35	66·452	60	66·111
15	66·513	40	66·407	65	66·007
20	66·515	45	66·351		

At low concentrations the rotatory power increases considerably and amounts, according to Nasini and Villavechia, to :—

c	1·2588	1·2378	1·2083	1·0129	0·8255	0·6631	0·5985	0·5880	0·3350
$a\frac{20}{D}$	66·604	66·716	66·855	67·096	67·250	67·370	67·562	67·983	68·241

At higher concentrations, the differences in the rotatory power are so small that within the limits of concentration met with in all sugar-house polarization work they do not affect the results, whilst at lower concentrations, where they are more considerable, the figure which they influence is itself so small, that even there the difference is insignificant. In the well-known Schmitz tables for the calculation of the sucrose content from the polarimeter readings, the influence of the concentration is taken into consideration.

Influence of Temperatures.—The rotatory power of sucrose diminishes as the temperature rises to an extent, which for the simple rotatory power itself—quite apart from changes in the instrument brought about by differences of the temperature—can according to Schönrock's calculation be expressed by the formula :—

$$a\frac{t}{D} = a\frac{20}{D} - a\frac{20}{D} \times 0{\cdot}000217\ (t^\circ - 20).$$

In practical working this figure is also influenced by the alterations in the rotatory power of the quartz wedges of the polarimeter, by the expansion of the polarization tubes, etc., which cause the correction for the Ventzke scale with glass polarimeter tubes to become

$$\text{readings at } t^\circ = \text{readings at } 20^\circ\ [1 - 0{\cdot}0003\ (t^\circ - 20)\ ]$$

which means that for every degree Ventzke the rotation is found to be 0·0003° V. too low for every degree Centigrade above 20° C. (the temperature at which the polarimeter is standardized), and the same amount too high for every degree Centigrade below that temperature of 20° C.

Influence of Foreign Bodies.—When sucrose is dissolved in water containing alcohol, its rotatory power is slightly higher than when pure water is used; thus, for a concentration of 10 per cent. of sucrose in a mixture of 1 part of water and 3 parts of alcohol it becomes at 20° C. 66·827 instead of 66·499. Alkalis, alkali carbonates and alkaline earths reduce the specific rotation of sucrose, probably owing to the formation of saccharates, as neutralization with acid restores the original rotation.

The reduction of the rotatory power by alkaline substances is much more considerable in concentrated than in dilute solutions, and amounts, according to Pellet, for the alkalis, their carbonates, and the alkaline earths, to the following values:—

At concentrations $c = 5{\cdot}4$ and $c = 17{\cdot}3$, 1 grm. of the undermentioned substances marks the rotation of the stated quantities of sucrose in grammes.

	$c = 5{\cdot}4$	$c = 17{\cdot}3$
1 grm. of caustic potash	0·170 ..	0·500
1 ,, ,, soda	0·140 ..	0·450
1 ,, potassium carbonate ..	0·044 ..	0·065
1 ,, sodium carbonate	0·040 ..	0·132
1 ,, ammonia	0·073 ..	0·085
1 ,, ammonium carbonate ..	0·040 ..	0·067
1 ,, lime	0·700 ..	1·000
1 ,, baryta	0·190 ..	0·430

Influence of Basic Lead Acetate.—Basic lead acetate, though also an alkaline substance, does not affect the rotation of sucrose to such an extent.

Bates and Blake* stated the influence of basic lead acetate solution of 1·25 specific gravity on the rotation of a solution of 26·048 grms. sucrose in 100 c.c. to be in degrees Ventzke:—

c.c. Basic Lead Acetate Solution.	Difference.	c.c. Basic Lead Acetate Solution.	Difference.
0·5 ..	0·09	10·0 ..	+0·19
1·0 ..	0·13	12·0 ..	+0·29
1·5 ..	0·10	15·0 ..	+0·29
2·0 ..	0·13	18·0 ..	+0·45
2·5 ..	0·06	20·0 ..	+0·45
3·0 ..	0·08	25·0 ..	+0·58
4·0 ..	0·06	30·0 ..	+0·62
5·0 ..	0·03	35·0 ..	+0·77
6·0 ..	0·00	40·0 ..	+0·77
7·0 ..	+0·05	63·0 ..	+0·95
8·0 ..	+0·09		

* "Jl. Amer. Chem. Soc.," 1907, 286.

The addition of small quantities of basic acetate solution therefore causes a progressive reduction in the polarimeter reading; with larger quantities of the reagent the reduction becomes less; the solution to which 6 c.c. of basic lead solution has been added gives the same polarimeter reading as one without lead, while still larger quantities cause a progressive increase of the rotation so long as reducing sugars are absent. As in sugar-house work the excess of basic lead never goes beyond a few drops, we may, in ordinary practice, safely neglect the influence of this reagent on the polariscopic reading.

Neutral Salts.—Alkali chlorides, sulphates, and phosphates, as well as the chlorides of the alkaline earths, reduce the rotation of sucrose solutions, as is shown by the following table of Bodenbender and Steffens.*

	Sucrose.	Salt.	Water.	Polarization.	Difference.
Potassium chloride	5	1	94	4·987	0·013
	10	2	88	9·856	0·144
	20	4	76	19·869	0·131
Sodium chloride	5	1	94	4·969	0·131
	10	2	88	9·853	0·147
	20	4	76	19·586	0·414
Barium chloride	5	1	94	4·952	0·048
	10	2	88	9·944	0·056
	20	4	76	19·402	0·598
Magnesium sulphate	5	1	94	4·995	0·005
	10	2	88	9·890	0·109
	20	4	76	19·880	0·120
Sodium phosphate	5	1	94	4·958	0·042
	10	2	88	9·933	0·067
	20	4	76	19·689	0·311

In the presence of small amounts of inorganic salts such as are met with in ordinary sugar-house routine work, the influence of the salt is imperceptible. With proportions of 4 parts of the inorganic salt to 20 parts of sucrose the product itself is so impure that the absolutely certain determination of the sucrose by polarization is rendered impossible by the large volume of the precipitate occasioned by the clarification and also by the dark coloration of the liquid. Besides, clarification with basic lead acetate converts all the salts into acetates, the action of which is less than that of the original salts, so that in practical working the effect of these inorganic salts on the rotatory power of sucrose may safely be neglected.

* "Zeitschr. Rübenzuckerind." 31, 808.

Refractive Index of Sucrose Solutions.—O. Schönrock* has determined the refractive index for sucrose solutions in water for concentrations between 0 and 66 per cent., and at temperatures between 10° and 35° C.

Table I below gives the results of measurements of N at a water content of the solution W; that is to say, for the number of grms. of water in 100 grms. of the sucrose solution.

Table II shows the difference in the refractive index N in units to the fifth decimal between 20° C. and the temperature t for the different water contents W. It is to be observed that the value for N falls as that of t rises.

TABLE I

W	N^{20}	W	N^{20}	W	N^{20}
100	1·3330	78	1·3672	56	1·4076
99	1·3344	77	1·3689	55	1·4096
98	1·3359	76	1·3706	54	1·4117
97	1·3374	75	1·3723	53	1·4137
96	1·3388	74	1·3740	52	1·4158
95	1·3403	73	1·3758	51	1·4179
94	1·3418	72	1·3775	50	1·4200
93	1·3433	71	1·3793	49	1·4221
92	1·3448	70	1·3811	48	1·4242
91	1·3464	69	1·3829	47	1·4264
90	1·3479	68	1·3847	46	1·4285
89	1·3494	67	1·3865	45	1·4307
88	1·3510	66	1·3883	44	1·4329
87	1·3526	65	1·3902	43	1·4351
86	1·3541	64	1·3920	42	1·4373
85	1·3557	63	1·3939	41	1·4396
84	1·3573	62	1·3958	40	1·4418
83	1·3590	61	1·3978	39	1·4441
82	1·3606	60	1·3997	38	1·4464
81	1·3622	59	1·4016	37	1·4486
80	1·3639	58	1·4036	36	1·4509
79	1·3655	57	1·4056	35	1·4532
				34	1·4555

* "Zeitschr. Rübenzuckerind." 1911, 421.

TABLE II

	Water Content equals														
t	100	95	90	85	80	75	70	65	60	55	50	45	40	35	30
10	72	80	89	97	105	114	122	131	139	147	156	164	172	180	189
12	60	67	73	80	86	93	100	106	113	119	126	132	138	145	151
14	47	52	57	62	66	71	76	81	86	90	95	100	104	109	114
16	32	36	39	42	45	48	51	55	58	61	64	67	70	73	76
18	17	19	20	22	23	25	26	28	29	31	32	34	35	37	38
20	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
22	18	20	21	23	24	26	27	28	30	31	33	34	36	37	38
24	38	41	43	46	49	52	55	58	60	63	66	69	72	74	77
26	50	63	67	71	75	80	84	88	92	96	100	104	108	112	116
28	80	86	92	97	102	108	113	119	124	129	134	140	145	150	155
30	104	111	118	124	131	137	144	150	163	163	169	176	182	188	195
32	129	137	144	152	160	167	175	182	190	197	205	212	220	227	235
34	155	164	172	181	190	198	207	215	224	232	241	249	258	266	275
36	182	192	202	211	221	230	240	249	259	268	278	287	296	306	315

Entirely independent of one another, Tolman and Smith* in the United States, and Prinsen Geerligs and van West† in Java, had made tables of comparison between refractive index and sucrose content. The former was established at 20° C, the latter at 28° C. Taking into consideration the correction for the temperature, these tables both fully agree with those of Schönrock.

Viscosity.—The viscosity of sucrose solutions increases as the concentration rises and decreases as the temperature rises. This is shown by the following three tables, compiled by Claassen‡. In these the number of seconds which

Sucrose on 100 Water.	Rate of Flow. Time in Seconds.	Viscosity. Saturated Solution = 100.
100	22	13
160	47	29
188	88	54
219	164	100
224	187	114
230	217	132
236	248	151
242	280	171
248	318	194
256	373	227
270	464	283

* "Jl. Amer. Chem. Soc.," 1906, 1480. † "Int. Sugar Journal," 1908, 68.
‡ "Zeitschr. Rübenzuckerind.· 48, 535.

100 c.c. of a sucrose solution of different concentrations require to flow out from an Engler viscosimeter is recorded as rate of flow. V (viscosity) means the ratio of these figures calculated on 100, being the rate of flow of the saturated sucrose solution at the temperature of 30° C. of 219 parts of sucrose on 100 parts of water.

The second table shows the rate of flow of a sucrose solution saturated at 30° C. when examined at different temperatures.

Temperature °C.	Rate of Flow. Time in Seconds.	Temperature. °C.	Rate of Flow. Time in Seconds.
16	476	42	86
21	330	51	54
25	246	61	39
30	163	71	30
37	114	—	—

The third table gives the rate of flow of sucrose solutions of increasing concentrations at increasing temperatures, and shows that the decrease in viscosity by the rise in temperature is much more considerable than its increase by the higher concentration.

Sucrose per 100 of Water.	Per Cent. Water.	Per Cent. Sucrose.	Temperature in °C.	Rate of Flow in Seconds.
201	33·3	66·7	17	180
206	32·7	67·3	22	161
229	30·4	69·6	35	110
273	26·8	73·2	55	63
294	25·4	74·6	63	55
323	23·6	76·4	71	51

The values for the viscosity of impure sucrose solutions, e.g., molasses, which generally contain as much foreign substance as sucrose, differ much from those given above, a point that will be dealt with later on in the chapter on molasses.

Behaviour of Sucrose at High Temperature.—Perfectly dry sucrose may be heated for a long time at temperatures exceeding 100° C., and even melted

at 160° C*., without undergoing any decomposition, but in presence of even traces of water it is decomposed at temperatures above 100° C.; this is shown by the dark colour it assumes, and the property it acquires of reducing Fehling's solution.

Sucrose, when heated for some time at its melting point, changes without any loss of weight into a mixture of glucose and fructosan (levulosan). Thus :—

$$C_{12}H_{22}O_{11} = C_6H_{12}O_6 + C_6H_{10}O_5$$

On heating sucrose at a still higher temperature it loses water, and leaves behind a dark-coloured body called caramel; if the temperature is further raised this is decomposed into gaseous products such as carbon dioxide, carbon monoxide, formic acid, acetone, etc., and a glossy coke which, on combustion in contact with air, burns entirely away and leaves no residue, provided the sugar used in the experiment was pure.

Calorific Value.—On combustion in a bomb calorimeter, sucrose gives a calorific value of 3955·2 kg/kg calories or 7119·4 B.T.U.

Decomposition of Sucrose Solutions on Boiling.—On continuously heating sucrose solutions at their boiling point for a long time, the sucrose combines with water even at the ordinary atmospheric pressure, and gradually changes into a mixture of equal parts of glucose and fructose.

The material composing the vessel in which the solution is boiled exerts a considerable influence on the rapidity of the transformation; copper has the greatest accelerating action in this respect, then silver, next platinum, whilst with glass vessels the rate of hydrolysis is least of all, especially when a water-bath, and not a naked flame, is used as the source of heat. In such determinations inconvenience is caused by the formation of small quantities of acid, so that the values thereby found do not represent the amount of hydrolysis due to water alone. In order to remove this source of error, Herzfeld†, in his researches on the decomposition of sucrose at high temperatures with prolonged action, rendered his solutions slightly alkaline at the commencement of the experiment. The results he obtained on heating sucrose solutions having an initial alkalinity of 0·01-0·05 per cent. are summarized in the following table. The solutions contained in metal vessels were heated in a water or oil bath at the temperature given in the first column of the table. The figures in the first table refer to the quantity of sucrose decomposed per hour per 100 parts of solution, and those in the second give the amount of sucrose decomposed during the same period of time per 100 parts of sucrose originally present.

* Burne ("Chem. News," 1914, 110) found the fusion point of sucrose to be 185° C.
† "Zeitschr. Rübenzuckerind." 43, 745

°C.	10 Per Cent.	30 Per Cent.	50 Per Cent.
80	0·0044	0·0047	0·0100
90	0·0079	0·0087	0·0196
100	0·0114	0·0127	0·0292
110	0·0163	0·0167	0·0388
120	0·0282	0·0577	0·1399
130	0·2055	0·2600	0·5900
140	0·5100	—	—

°C.	10 per cent.	15 per cent.	20 per cent.	25 per cent.	30 per cent.	35 per cent.	40 per cent.	45 per cent.	50 per cent
80	0·0444	0·0373	0·0301	0·0229	0·0157	0·0168	0·0179	0·0190	0·0200
90	0·0790	0·0667	0·0541	0·0418	0·0290	0·0317	0·0344	0·0371	0·0392
100	0·1140	0·0961	0·0781	0·0602	0·0423	0·0466	0·0508	0·0551	0·0584
110	0·1630	0·1362	0·1093	0·0825	0·0557	0·0612	0·0667	0·0721	0·0766
120	0·2823	0·2582	0·2341	0·2098	0·1857	0·2063	0·2669	0·2474	0·2678
130	2·0553	1·7582	1·4610	1·1638	0·8667	0·9451	1·0235	1·0119	1·1800
140	5·1000	—	—	—	—	—	—	—	—

Under a high pressure, coincident with a higher boiling point, this reaction takes place more rapidly, so that a solution is completely inverted after six hours' heating at 150° C., which if otherwise boiled at 100° C. would be totally transformed at the end of 24 hours.

Sucrose solutions, when boiled with live steam at 130° C., become very rapidly decomposed, and in the case of higher concentrations than those recorded in Herzfeld's tables, the loss by this means is undoubtedly considerable.

As a result of this decomposition, besides glucose and fructose, carbon dioxide, formic acid, acetic acid, and many other more complex organic acids are formed. The temperature at which the decomposition reaches a maximum lies between 110° and 120° C., so that these temperatures ought to be avoided as much as possible in the course of manufacture.

The action of superheated steam also breaks up sucrose; at 160° C., a temperature which can occur in boilers, humic acid, carbonic acid, and formic acid are formed. At 280° sucrose is split up into carbonic acid and carbon with the formation of various empyreumatic products.

Decomposition by Light.—Berthelot and Gaudechon* showed that sucrose solutions under the influence of ultraviolet light rays separate gas, and this in proportion to the amount of invert sugar corresponding to their concentration. Light inverts sucrose solutions and forms gas from the invert sugar, viz., carbon monoxide from fructose, and carbon monoxide and hydrogen from glucose.

Decomposition of Sucrose by Acids.—Dilute acids exert a hydrolysing power on sucrose, slowly at the ordinary temperature but quickly on heating. Thereby one molecule of sucrose combines with one molecule of water to form one molecule of fructose and one molecule of glucose. Thus :—

$$C_{12}H_{22}O_{11} + H_2O = C_6H_{12}O_6 + C_6H_{12}O_6$$

Inversion.—The sugar thus transformed now deflects the plane of polarization to the left instead of to the right, hence the newly-formed body bears the name of invert sugar, and the phenomenon itself that of inversion. During this transformation, 95 parts of sucrose combine with 5 parts of water to form 100 parts of invert sugar.

Inversion Velocity.—The velocity of the inversion depends on different laws, which have been formulated by Ostwald as follows :—

1. On inversion by a strong acid, sulphuric or hydrochloric acid for instance, in every unit of time a constant part of the sucrose present is inverted ; the value of the constant depends only on the acid used. Each acid has, therefore, its inversion constant, which is expressed by the formula $C\frac{1}{t} \times log \frac{A}{A-x}$, in which A represents the quantity of sucrose initially present, and x that of sucrose inverted, so that this constant is inversely proportional to the time in which the inversion is completed.

2. The velocity of inversion by equal quantities of the acid acting on different quantities of sucrose in the same volume is always the same. At the same concentration of the acid the constant is not influenced by the quantity of sucrose, so that even the most concentrated sucrose solutions can be inverted by minimum quantities of acid.

3. The action of the acid is in direct proportion to its concentration, though the inversion velocity of strong mineral acids increases more rapidly with progressive inversion than that of feeble ones.

4. The inversion velocity increases rapidly with the temperature.

* "Comptes rendus de l'Académie des Sciences," 1912, 2-12.

Inversion Constants of some Acids.—Ostwald determined the inversion constants of a number of acids by mixing 10 c.c. of a 40-50 per cent. sucrose solution at 25° C. with 10 c.c. of a normal solution of the acid, and ascertaining after a certain time the amount of invert sugar formed.

These constants, and the same values calculated on hydrochloric acid as a starting point with the figure 100, are shown in the table underneath.

	Constant.	HCl = 100		Constant.	HCl = 100
Hydrobromic acid	24·38	111·4	Tartaric acid ..	0·674	3·08
Hydrochloric acid	21·87	100·0	Citric acid ..	0·377	1·72
Nitric acid ..	21·87	100·0	Formic acid ..	0·335	1·53
Sulphuric acid ..	11·72	53·6	Malic acid ..	0·278	1·27
Sulphurous acid ..	6·63	30·4	Lactic acid ..	0·233	1·07
Oxalic acid ..	4·00	18·57	Succinic acid ..	0·119	0·545
Phosphoric acid ..	1·357	6·21	Acetic acid ..	0·088	0·400

It is true that these comparative figures are not of great practical value as they are based on the temperature of 25° C. and the inversion velocity of the acids rises very irregularly and unequally at higher temperatures. Thus it increases for sulphuric and sulphurous acid very rapidly between 30° and 40°, for oxalic acid at 40°, for phosphoric acid between 40° and 50°, and for acetic acid between 70° and 80° C. The inversion constant of some acids is at 40° eight times, and at 55° as much as forty-eight times as great as at 25° C.

The inversion constant of weak acids is measurable only at temperatures at which inversion by strong acids is so rapid that accurate determinations cannot be made, and it is for this reason that the compilation of trustworthy tables of comparison for higher temperatures is impossible. Hence Ostwald's table is of little value in the sugar-house, where but very slight amounts of weak acids are met with and high temperatures have to be considered.

On the other hand we reap much more benefit from Noël Deerr's determinations* of the inversion velocity, which were made at 97° C. with concentrations of 1/1000 normal acid. He poured 25 c.c. of a 20 per cent. sucrose solution into a 50 c.c. flask, added 5 c.c. of 1/1000 normal acid, filled up to the mark with water and kept the mixture during 30 minutes in a boiling water bath, of which the temperature was about 97° to 98° C. After that, the solution was rapidly cooled and polarized.

* "Hawaiian Expt. Station Bulletin," No. 35.

We give below the constants found thereby and their relation to hydro chloric acid, taken as a basis with the figure 100

	Constant.	Hydrochloric Acid. =100
Hydrochloric acid	181	100·0
Nitric acid	180	99·9
Sulphuric acid	132	72·9
Sulphurous acid	88	48·6
Oxalic acid	77	42·7
Phosphoric acid	34	18·7
Acetic acid	12	6·4

The presence of foreign bodies exerts a remarkable influence on the velocity of inversion by acids, there being an increase with neutral salts, such as sodium chloride, magnesium chloride, etc., when these occur in higher concentrations than 0·5 *N*, but a decrease when the concentration is lower. Sulphates on the contrary produce a decrease in velocity, both when in low and also when they occur in high concentrations. Glucose increases the velocity.

Inverting Power of Salts.—Besides acids, many salts, especially those of inorganic acids, possess the power of inverting sucrose at the boiling point of its solutions, which power does not increase in proportion to their concentration. This must be ascribed to the fact that these salts occur in the solution in a partially dissociated state and that the part of the acid which is practically uncombined exerts its inverting power like a free acid. Such are, in fact, the salts of heavy metals as the sulphates of zinc, iron, and copper, which possess an acid reaction in solution.

When glucose, however, is present, salts having a neutral reaction also show this property, and especially those which are very liable to dissociation, like ammonium salts or salts with feeble bases. Salts with a strong base, or salts of organic acids (which even when free are but feeble hydrolysts), accordingly possess also a very slight inverting power or none at all, or can even neutralize by their mere presence the action of the other salts. Calcium carbonate too will prevent this action, so that we may safely conclude that the glucose acts here as a feeble acid which has the effect of liberating extremely small quantities of acid from the salts.

These seeming highly intricate and embarrassing phenomena can, however, very clearly be explained by the new theory of the dissociation of water into hydrogen and hydroxyl ions.*

According to that theory it is supposed that in pure water at 25° C. one ten-millionth grammolecule of free hydrogen ions is present. This for convenience' sake is written as 1×10^{-7} or pH=7 (pH standing for hydrogen potential).

If an acid is dissolved in water, the concentration of the hydrogen ions increases, and the negative logarithm or the value of pH decreases; while, on the other hand, an addition of alkali to the water causes a decrease in the hydrogen ions and an increase in the value for pH. All values for pH or hydrogen potential over 7 point to an alkaline reaction; 7 is just neutral, and values under 7 indicate an acid reaction.

It has been proved that in aqueous solutions having a lower pH than 7, the free hydrogen ions exert an invertive action on sucrose, the rate of which is more rapid the higher the temperature and the stronger the concentration of the hydrogen ions. The determination of the hydrogen potential of an aqueous solution therefore furnishes at the same time a measure of its invertive power. Sucrose solutions possess a potential just under 7, while the values for solutions of glucose and fructose are lower still. This explains why a solution of pure sucrose in water becomes slightly inverted after a prolonged ebullition, and why this inversion proceeds more rapidly as soon as reducing sugars are also present in the solution. Free acids, acid salts, as well as mixtures having a pH of under 7 therefore possess an invertive power, and as such is the case in many of the instances enumerated above, it is now clear why a mixture of sodium chloride and glucose in aqueous solution is apt to invert sucrose, and also why the simultaneous presence of calcium carbonate can annihilate that property.

A list of the hydrogen potentials of some acids is given here for comparison with the invertive action determined in the empirical way by Ostwald and by Noël Deerr, and a good concordance between them can be perceived, insomuch that the acids having the highest invertive power are at the same time the most strongly dissociated and show the highest concentration of hydrogen ions. Complete conformity could not be expected, since the concentration of the hydrogen ions is a variable value at the different temperatures and concentrations met within the different experiments.

The concentrations of the hydrogen ions and the hydrogen potential for a few acids at 20° C. and in 0·1 molar solution are as follows :—

* W. Mansfield Clark, "The Determination of Hydrogen Ions."

Acid.	Concentration.	Potential.
Hydrobromic acid	$9{\cdot}3 \times 10^{-2}$	1·03
Hydrochloric acid	$9{\cdot}3 \times 10^{-2}$	1·03
Nitric acid	$9{\cdot}3 \times 10^{-2}$	1·03
Sulphuric acid	$1{\cdot}12 \times 10^{-1}$	0·95
Sulphurous acid	$3{\cdot}35 \times 10^{-2}$	1·47
Oxalic acid	$6{\cdot}55 \times 10^{-2}$	1·18
Phosphoric acid	$3{\cdot}04 \times 10^{-2}$	1·52
Malonic acid	$3{\cdot}31 \times 10^{-2}$	1·48
Citric acid	$7{\cdot}45 \times 10^{-3}$	2·13
Formic acid	$4{\cdot}4 \times 10^{-3}$	2·36
Malic acid	$6{\cdot}1 \times 10^{-3}$	2·22
Lactic acid	$3{\cdot}8 \times 10^{-3}$	2·42
Succinic acid	$2{\cdot}55 \times 10^{-3}$	2·39
Acetic acid	$1{\cdot}35 \times 10^{-3}$	2·87

Since, obviously, the invertive power of aqueous solutions finds its expression in the figure indicating the concentration of the hydrogen ions, it is clear that the determination of that factor, together with that of the total acidity, is of the greatest importance in the analysis of juices, syrups and molasses in sugar-house laboratories.

As organic acids occur largely in cane juice, and as therefore in neutralized juices and massecuites the amount of organic salts is large in proportion to the inorganic ones, and further because the percentage of reducing sugars is, as far as first products go, very low when compared with that of the sucrose, there will be very little danger of inversion by neutral salts in the practice of cane sugar manufacture from the milling to the after-products stage. But in the after-products and molasses, which generally possess an acid reaction and in which the reducing sugars have become concentrated, inversion at high temperatures is often observed; this is doubtless largely due to the presence of free acids, but not altogether, for it is possible that the action of inorganic salts may also account for it in some degree.

Action of Concentrated Acids.—Mineral acids of high concentration decompose sucrose. Gaseous hydrochloric acid transforms it into a mixture of ulmic acid and caramel; concentrated hydrochloric acid chars it; whilst a sucrose solution when boiled with dilute hydrochloric acid yields a series of decomposition products, which have been investigated by Berthelot and André. The principal products of this reaction are formic acid, levulinic acid, and humic acid, along with small quantities of carbonic acid, carbon monoxide, and furfural. Our own researches in this province will be dealt with in the

discussion on glucose and fructose. Glucose forms chiefly levulinic acid, whilst humic acid is the principal decomposition product of fructose.

Action of Alkalis and Alkaline Earths.—Alkaline bodies, such as potash, soda, lime, etc., only attack sucrose solutions when they are heated with them in a concentrated state. On boiling a sucrose solution with a small percentage of potash or soda, it remains unchanged, but with higher concentrations of the alkali the sucrose becomes decomposed with the formation of formic acid, lactic acid, and humic bodies. Lime also attacks sucrose very slowly and one may even introduce small pieces of quicklime into a 10 per cent. sucrose solution and there allow them to slake without fear of decomposition occurring; but on boiling a sucrose solution with lime for a long time, the sugar is attacked, and yields acetic and lactic acids which of course remain in solution as lime salts.

Saccharates.—Moderately strong concentrations of solutions of alkalis and alkaline earths combine with sucrose and form soluble alkaline compounds called saccharates, which require just as much acid for their neutralization as the quantity of base in the compound.

Alkali Saccharates.—The formula of such a saccharate is, for instance, $C_{12}H_{21}O_{11}K$; after addition of carbonic acid to the solution of a saccharate it breaks up into sucrose and the alkali carbonate, but in aqueous solutions, even when very dilute, it is stable and cannot be decomposed by dialysis.

Calcium Saccharates.—The alkaline earths form a whole series of saccharates, of which some are soluble and others insoluble, and which, owing to their property of being readily decomposed by carbonic acid, may render good service in the separation of sucrose from molasses or from impure solutions in general.

The monobasic calcium saccharate $C_{12}H_{22}O_{11}CaO$ is soluble in water, but by boiling its solution it is transformed into tribasic saccharate and sucrose.

$$3C_{12}H_{22}O_{11}CaO = C_{12}H_{22}O_{11}\,3\,CaO + 2C_{12}H_{22}O_{11}$$

The bisaccharate is also soluble in water and its solutions are likewise transformed into tribasic saccharate and sucrose on boiling.

On passing carbonic acid gas into a solution of calcium saccharate it is at first entirely absorbed, but soon the mass becomes viscous and gelatinous and absorbs the carbonic acid much less readily. Finally, when the gas has been completely absorbed the liquid becomes thin again, calcium carbonate is precipitated and sucrose is found in solution. The gelatinous character of the first-formed compound of carbonic acid and calcium saccharate, $C_{12}H_{22}O_{11}3CaCO_3 2Ca(OH)_2$, causes the difficult absorption of the carbonic

acid, which penetrates in large bubbles through the viscous liquid and for the greater part passes out unabsorbed; but when later on the liquid becomes fluid again by the gradual decomposition of the compound referred to above, the rate of the carbonic acid absorption is restored, and finally the saccharate becomes quantitatively split up into calcium carbonate and sucrose.

Strontium Saccharate.—Strontium bisaccharate is formed by dissolving strontium hydrate in a boiling 15 per cent. sucrose solution; the separation of the saccharate sets in as soon as more than two molecules of strontium hydrate are added to each molecule of sucrose present, and on the addition of further quantities of strontium hydrate almost all of the sucrose is precipitated as saccharate when about three molecules of strontium hydrate are present. The insolubility of strontium saccharate in alcohol is utilized in the separation of small amounts of sucrose from plant extracts.

To this end the plants are extracted with hot 90 per cent. alcohol, and the extract is filtered. To the hot filtrate so much strontium hydrate solution is added, that for each part of sucrose more than three parts of the hydrate are present. The mixture is boiled for half an hour, filtered, and the precipitate washed with hot alcohol. The liquid is boiled again with strontium hydrate for half an hour, and filtered through a hot water funnel. The precipitates are pressed as dry as possible, suspended in water, and decomposed by a current of carbonic acid. Strontium carbonate is thrown down, whilst the filtered sucrose solution can be evaporated and allowed to crystallize.

Barium Saccharate.—Barium saccharate crystallizes out in the shape of a solid cake by carefully mixing at an elevated temperature a sucrose solution with a quantity of hot and saturated solution of barytes sufficient to form the compound: $C_{12}H_{22}O_{11}BaO$.

This body is fairly well insoluble in barytes solutions, and it is technically utilized in beet sugar factories to desaccharify molasses.

It is somewhat soluble in water, and is decomposed into barium carbonate and sucrose by carbonic acid.

Ferric Saccharate.—Ferric saccharate is formed by allowing iron to dissolve slowly in sucrose solution in presence of oxygen. The ferric saccharates of the Pharmacopœia are not true saccharates, since all of them contain alkali; a part of their iron content is, however, present in the form of saccharate. Ferric saccharate may be obtained by precipitating ferric chloride dissolved in a sucrose solution with sodium hydrate, filtering the precipitate off, washing it, and suspending it again in a sucrose solution which will gradually dissolve it. The ferric oxide in the saccharate compound is not liable to precipitation by ammonia or alkalis, nor to coloration by tannin, or potassium ferrocyanide, or potassium sulphocyanide. In fact it no longer shows the usual ferric reactions.

Ammonium sulphide, on the contrary, gives a precipitate of iron sulphide from such a solution, whilst acids decompose the compound and restore the property of the ferric oxide to give blue colorations with ferrocyanides and red ones with sulphocyanides.

Lead Saccharate.—On precipitation of a sucrose solution with an ammoniacal basic lead acetate solution, a precipitate of tribasic lead saccharate is obtained. This may also be prepared by stirring sucrose with water and plumbic oxide; if the proportions are well chosen, all the sucrose is deposited as an insoluble tribasic lead saccharate. On suspending this in water, through which a current of carbonic acid is passed, the sucrose can again be liberated.

Influence on Solubility of Saccharates.—Owing to the formation of saccharate a great many substances which are sparingly soluble in water, or even not at all, dissolve easily in sucrose solutions. Lime is for that reason much more soluble in a sucrose solution than in water, and further the calcium saccharate formed thereby possesses the property of dissolving other bodies which do not dissolve in water or in sucrose solutions; among these we may specially mention calcium oxalate, phosphate, and carbonate, and oxides of the heavy metals; when the saccharate is broken up they of course become insoluble again. Sucrose prevents the precipitation of iron, lead, and copper hydroxides, etc., by soda, as those hydroxides immediately pass over into soluble saccharates.

Apart from this formation of saccharates, sucrose solutions dissolve certain other bodies to a greater extent than water; especially towards lime salts of organic acids is this property exhibited in a marked degree, but towards those of sulphuric, sulphurous and carbonic acids to a much less extent. In the case of the simultaneous presence of either alkali or acid, however, their solubility greatly increases, as will be clearly shown in the chapter on *Concentration of the Juice.*

Double Combinations with Salts.—Sucrose forms double compounds with the salts of alkali metals; those with sodium chloride, sodium iodide, as well as those with potassium chloride, potassium nitrate, and sodium nitrate, are obtained in a crystallized form. Their composition can be represented by the formula

$$C_{12}H_{22}O_{11}NaCl + 2\,H_2O$$

and they can be prepared by allowing a mixture of the sucrose solution with the salt to evaporate slowly at the ordinary temperature in a desiccator over sulphuric acid. These compounds are dissociated in solution, and can be separated into their constituents by dialysis. Besides the few crystallizable double compounds mentioned above, there still exist many others which as yet have not been induced to crystallize and are obtained as viscous syrupy fluids. These combinations, principally those of the organic salts, are of very

great importance in sugar manufacture, and we therefore prefer to discuss them in greater detail in a later chapter on *Molasses*.

Decomposition of Sucrose by Oxidizing Agents.—Powerful oxidizing agents readily attack sucrose. Nitric acid oxidizes it with much evolution of gas into saccharic acid, and further into tartaric acid, and oxalic acid.

Fehling's solution attacks sucrose only very slowly and after prolonged boiling, that is to say, when it has already become somewhat inverted; pure sucrose is however not attacked. Nor does ammoniacal silver nitrate solution oxidize sucrose solution at the ordinary temperature; on heating, however, silver is deposited as a consequence of the oxidation of the sucrose. Free oxygen does not attack sucrose; in the case, however, of the solution being acid or containing invert sugar it absorbs oxygen with the formation of formic acid. In presence of catalytic substances as spongy platinum, it is oxidized into carbonic acid and water. Finely granulated boneblack washed with hot water and treated when still hot with a concentrated sucrose solution at 85°-90° C. oxidizes the sucrose in presence of atmospheric oxygen so rapidly that it becomes caramelized with the liberation of much steam, which reaction is so violent that it sometimes becomes explosive. Ozone, though not attacking neutral sucrose solutions, will oxidize acid or alkaline ones. Hydrogen peroxide causes rapid inversion, especially in presence of iron salts, and after that oxidizes the newly-formed invert sugar.

Decomposition of Sucrose by Ferments.—Sucrose is very liable to be broken up by all kinds of ferments. Almost every species of yeast contains a ferment (invertase) that transforms sucrose into invert sugar, which latter afterwards becomes changed by the influence of the fermentative enzymes of the yeast cells into alcohol, carbonic acid, and certain by-products, thus causing the sucrose as such to totally disappear.

Alcoholic Fermentation.—Only a very few yeasts do not contain invertase and cannot transform sucrose either directly or indirectly. It has often been suggested to make use of these for removing by fermentation the glucose from syrups or molasses in order to purify them, and thus improve the extraction of the still intact sucrose; but this has not yet been introduced in practice.

Lactic and Butyric Acid Fermentation.—The lactic acid bacteria transform sucrose, especially in presence of lime, into lactic acid, by which process carbonic acid and hydrogen are also formed; this is often followed by the butyric acid fermentation, forming the unpleasant smelling butyric acid from the lactic acid—a phenomenon sometimes met with in practice.

Dextran Fermentation.—Sometimes we find infection by another lower organism, *Leuconostoc mesenterioides*, in cane sugar factories. This rapidly

forms a gelatinous body called dextran, as well as much lactic and acetic acids. The dextran is part of the body of the *Leuconostoc* itself, and is principally formed in an alkaline or neutral solution, so that the *Leuconostoc* by its own decomposition products renders the conditions of its development even more troublesome. Heating to the boiling point as well as disinfection with a 1 per cent. ammonium fluoride solution kills the *Leuconostoc*; therefore such a fluid is an excellent remedy against this infection in sugar-houses.

The chief constituent of the gelatinous mass is the dextran, $C_6H_{10}O_5$, which in a pure state appears as a neutral white mass. It does not dissolve in water but swells in it to a thick jelly, which is again precipitated by alcohol. It dissolves in dilute alkalis and then possesses a rotatory power of $\alpha D = +230°$. It does not reduce Fehling's solution, and is precipitated from its pseudo-solution in water by basic lead acetate. Acids hydrolyse it under pressure first into unfermentable dextrin and afterwards quantitatively into glucose.

The *Leuconostoc* organism forms more dextran from sucrose than from either glucose or fructose; so that its action does not require a preliminary inversion of the sucrose. Since the acid formed by the fungus during its action has a retarding influence on the latter, and a neutral reaction is much more favourable to the formation of dextran, the fungus in encountered in sugar-house practice in such places where juices are kept constantly neutral or faintly alkaline by the addition of lime.

Levan Fermentation.—Greig Smith and Steel* describe an organism called by them *Bacillus levaniformans*, which they found in cane juice, in which it formed a gummy substance named by them levan. At the same time succinic and acetic acids were formed. The pure levan dissolves in a small proportion of water to form a mucilage resembling gum arabic, and on the addition of more water the solution becomes white and opalescent. A solution of 1 grm. of crude gum in 100 c.c. of water when observed in a 100 mm. tube in a Laurent polarimeter with monochromatic sodium light gives a reading which is equivalent to a specific rotation of about $\alpha D = -40°$. On inversion according to the Clerget method, the levan gum is transformed into fructose; after the hydrolysis the specific rotation becomes $-92{\cdot}5°$. After oxidation with nitric acid of 1·24 specific gravity at 60° C. only oxalic acid is obtained, and neither mucic nor saccharic acid is formed. On treatment with dilute acids in the cold or more quickly on warming, the gum is readily and completely hydrolysed, the sole product being fructose, which is produced in practically the theoretical quantity required by the formula:

$$C_6H_{10}O_5 + H_2O = C_6H_{12}O_6.$$

W. L. Owen† found that other bacteria also are able to form levan from sucrose, among which he enumerates *Bacterium gummosum*, *Bacterium*

* "Int. Sugar Journal," 1903, 448. † "Louisiana Planter," 1923, II, 331, 353, 373.

vermiforme, *Bacillus vulgatus* and *Bacillus liadermos*. He found that those bacteria produce the levan from the uninverted sucrose, and that the simultaneous presence of reducing sugars retards this formation. The optimum value for pH for the formation of levan is between 6·7 and 7, or just within the acid range.

Ethyl Acetate Fermentation.—The fungus *Thielaviopsis æthaceticus, Went*, which causes the pineapple disease or black rot of the sugar cane, forms, besides alcohol, ethyl acetate from sucrose.

Detection of Sucrose.—When it is required to ascertain beyond doubt that a solution really contains sucrose, the isolation of this sugar can be effected by means of strontium hydrate by following the rules laid down on page 27. The crystals obtained can be identified by their crystalline form, and by the behaviour of their solution towards polarized light and Fehling's solution before and after hydrolysis by dilute acids.

Colour Tests.—In the course of time a great many colour tests for sucrose have been suggested, but they all have the disadvantage of not only detecting sucrose but also a number of other kinds of sugar, as they do not apply to the sucrose itself but to its decomposition products, either furfural or humin substances, which, according to the conditions of the reaction, arise from the sugars.

Among these may be mentioned the α-naphthol reactions. In alcoholic solution α-naphthol when mixed with a trace of sucrose and carefully poured into concentrated sulphuric acid produces a pink-coloured ring where the two liquids meet.

By mixing alcoholic α-naphthol solutions with a trace of sucrose and much concentrated sulphuric acid and heating the mixture on a water-bath, it very quickly assumes a reddish-pink coloration, which, however, likewise appears in the presence of fructose, sorbose, or raffinose. Under similar circumstances β-naphthol yields a reddish-brown and resorcinol a red coloration which, however, is also common to the other already mentioned sugars. As yet there does not exist a characteristic colour reaction for sucrose, so that to actually confirm its presence we are restricted to the characteristic reactions of the substance after its isolation in a pure form.

II.—Glucose (Dextrose)

Occurrence.—Glucose, besides occurring along with fructose as a product of decomposition due to inversion in over-ripe canes, is a normal constituent of both ripe and unripe cane, and is therefore to be found in all cane juices to a greater or less extent. It is also a very widely distributed constituent of plants in general, and is manufactured commercially in large quantities by the action of mineral acid on starch.

Crystalline Form.—Anhydrous glucose, crystallized from its concentrated solution in water, appears in the form of hard columnar crystals, melting at 146° C., while the more common hydrate, $C_6H_{12}O_6+H_2O$, occurs in opaque crystal crusts or in big transparent crystals with a melting point of about 86° C. It is impossible to state a fixed figure for its melting point, since at high temperatures the hydrate passes into the anhydride and the varying proportions of these two of course influence the temperature at which it melts.

Solubility in Water and in other Solvents.—Glucose readily dissolves in water; at 15° C. 100 parts of water dissolve 81·68 parts of the anhydrous compound or 97·85 parts of the hydrate. Glucose is also soluble in alcohol, and the more so in proportion as it is hotter and more dilute. It is very soluble in methyl alcohol, but not in ether nor in acetone.

Specific Gravity of Solutions in Water.—According to Salomon* the specific gravity of glucose solutions in water for the different concentrations is at a temperature of 17·5° C. (where water is 17·5° C = 1), as in Table opposite.

Rotatory Power.—The real value for the specific gravity is not obtained at once when the anhydrous glucose is dissolved in water, but one which is a little lower; it increases only after some time, probably owing to the slow transformation into the hydrate. Solutions of the hydrate, however, when freshly made show immediately the same specific gravity as that given by solutions which have stood for some time. Like sucrose, glucose deflects the plane of polarization to the right, though to a lesser degree. At high concentrations the specific rotatory power is somewhat greater than at low ones, but at the concentration in use in sugar-house practice it is fairly constant and may be stated as $\alpha\ D = +52{\cdot}85$ for the anhydrous glucose in a concentration of 14 grms. in 100 c.c.

Influence of Concentration and Temperature.—Taking the rotation of sucrose as 100, then that of the anhydrous glucose is 79·7, and that of the hydrate 72·5. As glucose can be determined by the polarimeter in the same way as sucrose, and as every degree Ventzke equals 1 per cent. sucrose when 26·048 grms. of the saccharine matter under observation are dissolved in

* "Berliner Berichte," 121, 2711.

100 c.c. at 17·5° C. (26 grms. in 100 true c.c. at 4° C.) and read in a 200 mm. tube, so under the same circumstances every degree Ventzke is equal to 1·25 per cent. of anhydrous glucose. Variation in temperature does not influence this rotatory power within those limits which are not exceeded in ordinary laboratory practice.

Influence of Alkalis and Salts.—Alkalis, and salts with an alkaline reaction, however, influence the rotation because these bodies either attack glucose or

Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.
1	1·00375	21	1·0800	41	1·1530
2	1·0075	22	1·0838	42	1·1568
3	1·0115	23	1·0876	43	1·1605
4	1·0153	24	1·0910	44	1·1643
5	1·0192	25	1·0946	45	1·1680
6	1·0230	26	1·0985	46	1·1716
7	1·0267	27	1·1020	47	1·1753
8	1·0305	28	1·1058	48	1·1790
9	1·0342	29	1·1095	49	1·1825
10	1·0381	30	1·1130	50	1·1863
11	1·0420	31	1·1170	51	1·1900
12	1·0457	32	1·1205	52	1·1935
13	1·0495	33	1·1240	53	1·1968
14	1·0533	34	1·1275	54	1·2005
15	1·0571	35	1·1310	55	1·2040
16	1·0610	36	1·1348	56	1·2075
17	1·0649	37	1·1383	57	1·2100
18	1·0687	38	1·1420	58	1·2148
19	1·0725	39	1·1456	59	1·2183
20	1·0762	40	1·1494	60	1·2218

combine with it. Neutral salts when present in considerable quantities reduce it, but in ordinary work no salt concentrations occur to such a degree as to exert any appreciable influence, and they can therefore be safely neglected. Neither acids nor sucrose affect the specific rotation of glucose.

Bi-rotation or Muta-rotation.—The above figures do not apply in the case of freshly made, cold glucose solutions, as in this case the glucose has a much greater rotatory power, which may even become twice the value just mentioned. After standing for some time at the ordinary temperature, or sooner on being heated, the bi-rotation (also termed muta-rotation) disappears, and the rotatory power reverts to the constant figure of 52·85°.

Two glucose solutions, containing respectively 1·8194 grms. and 1·1051 grms. of glucose in 20 c.c. of water showed subsequently the following values for the rotation*:—

Time after being dissolved.	Specific rotatory power observed.	Time after being dissolved.	Specific rotatory power observed.
5½ minutes	+ 105·16°	7 minutes	+ 104·26°
10 ,,	+ 101·55°	15 ,,	+ 98·63°
15 ,,	+ 96·99°	30 ,,	+ 88·61°
25 ,,	+ 87·86°	60 ,,	+ 73·58°
50 ,,	+ 72·26°	420 ,,	+ 52·60°
70 ,,	+ 63·33°		
90 ,,	+ 59·71°		
360 ,,	+ 52·94°		

Glucose, when dissolved in water containing ammonia, shows a constant rotation immediately without any sign of bi-rotation. A concentration of 0·1 per cent. of ammonia is quite sufficient for this purpose, nor is it advisable to use a greater one, as strong ammonia will attack the glucose and affect its rotation.

Refraction of Light.—The refraction of light caused by glucose solutions is shown in Stolle's table† on refraction given here. The figures refer to observations, of which one series is made 10 minutes and the other 24 hours after the preparation of the solutions. It is seen from these figures that the refractive indices of glucose solutions differ only very slightly from those of sucrose solutions; this has since been confirmed by a large number of other investigators.

Concentration of the Glucose.		Specific Gravity $\frac{17·5}{4}$		Refractive Index.	
0·9967	0·9967	1·00215	1·00215	1·33465	1·33473
2·0013	2·0013	1·00624	1·00624	1·33588	1·33605
4·0021	4·0022	1·01383	1·01383	1·33873	1·33890
8·0058	8·0059	1·02879	1·02881	1·34448	1·34456
12·0252	12·0257	1·04413	1·04417	1·35017	1·35026
15·9976	15·9990	1·05882	1·05891	1·35572	1·35581
19·9896	19·9915	1·07384	1·07394	1·36128	1·36164
25·0168	25·0193	1·09259	1·09270	1·36825	1·36863

* Parcus and Tollens, "Zeitschr. Rübenzuckerind." 40, 841. † "Zeitschr. Rübenzuckerind." 51, 335

Action of Heat.—Glucose when heated above 100° C. assumes a brown coloration; at 170° C. water passes off, and the coloured residue contains, amongst other products, dextrosan or glucosan, $C_6H_{10}O_5$, while at a still higher temperature the substance swells, blackens, and gives off a large quantity of gaseous products, leaving a carbonaceous mass, containing caramel, which when further heated in contact with the air, undergoes total combustion.

Calorific Value.—The calorific value of glucose as determined in the bomb calorimeter amounts to 3742·6 kg./kg. calories or 6736·7 B.T.U.

Action of Acids.—Dilute acids exercise no influence on glucose, but more concentrated strong acids, e.g., a 5 per cent. sulphuric acid solution, break it up just like sucrose, causing formation of black humin compounds, a little levulinic and formic acid and a little carbonic acid and carbon monoxide.

Action of Alkalis.—Dilute alkalis convert glucose, especially at a high temperature, into a mixture of glucose, fructose, and mannose. This process goes on gradually until the glucose and the fructose are represented in approximately equal parts, while the quantity of mannose is very small. Even very weak concentrations of alkalis or alkaline earths have this effect.

Converting Power.—A quantity of 5 grms. of lime when added to 100 grms. of glucose dissolved in 400 grms. of water is able at a temperature of 70° C. to convert the dextrose into the above-mentioned mixture within a few hours. On dissolving 20 grms. of anhydrous glucose and 10 c.c. of N/1 potassium hydroxide solution in water to a volume of 500 c.c., and heating the mixture to 60° C., the rotation decreases after 10, 20, 30, 40, 50, 85, and 135 minutes to + 5° 30′, 4° 20′, 3° 10′, 2° 20′, 1° 50′, 0° 43′, and 0° 10′ respectively. The specific rotation thus decreases to about 0, while at this temperature comparatively little glucose is decomposed by the action of the dilute alkali. Basic lead acetate, being an alkaline substance, also possesses this power, and so do, at a high temperature, the potassium and sodium salts of organic acids; but here the small amount of dissociated base contained in solution is able to effect the transformation without destroying the glucose.

Formation of Glucosates.—Apart from this reaction, alkalis and alkaline earths combine with glucose and form glucosates, compounds of a character analogous to the saccharates already mentioned. Potassium and sodium glucosates are formed by the precipitation of alcoholic glucose solutions with alcoholic potash or soda solutions; their composition is expressed by the formula $C_6H_{11}O_6K$; they are not crystallizable and are unstable both in the dry state and in solution. Calcium glucosate can be obtained by the precipitation of a solution containing glucose and lime with alcohol; this

compound when exposed to light is also unstable and is broken up at 50°C. with the formation of colour. Neither neutral nor basic lead acetate throws down glucose from its solution in water, but it is entirely precipitated by ammoniacal lead acetate (i.e., basic lead acetate to which ammonia is added till a precipitate is just about to form).

Destructive Action of Alkali.—These glucosates and those of the heavy metals are without special interest to the sugar manufacturer; but a third action of alkali on glucose is the destructive one, which takes place to some extent in the first-mentioned action in which the glucose is transformed. Together with this a part of the glucose is decomposed and if the quantity of alkali is sufficient and the action is continued long enough, all of the glucose is gradually destroyed and transformed, chiefly into organic acids.

Decomposition Products.—The products of the decomposition formed by this reaction differ according to the concentration, the temperature, and the nature of the alkali. On heating a glucose solution under 70° C. with potash or soda, the alkaline reaction disappears, the liquid assumes a brown coloration, and it is found to contain the salts of two acids, which Peligot called glucinic and saccharumic acid. These names now have become obsolete, since the real chemical character of those acids cannot be ascertained any longer with full accuracy. The former of these two is very unstable and decomposes spontaneously, immediately at high temperature, and gradually when cold, into humic, acetic, formic, and carbonic acids, while also some apoglucinic acid is formed. The saccharumic acid is a more stable one and forms readily crystallizable salts; when uncombined it however soon becomes coloured and decomposes into a number of products the nature of which is as yet uninvestigated. This spontaneous decomposition of the acids formed by the action of alkaline bodies on glucose accounts for the fact that cane syrups and molasses which at the outset are neutral subsequently always become acid.

At very high temperatures, at boiling point for instance, potash attacks glucose solutions with the formation of alcohol, acetone, acetic and formic acids, and dark-coloured humic substances.

At ordinary temperatures alkalis convert glucose without any considerable coloration, by simple molecular disengagement, into lactic acid, a very stable body which is not liable to spontaneous decomposition. At temperatures under 55° C. lime also forms lactic acid from glucose, but at higher temperatures the already mentioned acids are formed, which are apt to undergo spontaneous decomposition and become acid. This is the reason why during the process of carbonating cane juice, where much lime is brought into contact with glucose, the temperature of the limed juice must be maintained under 55° C. so long as the excess of lime is not saturated by carbonic acid.

Baryta forms products analogous to those obtained with lime, but as some of the primary decomposition products possess insoluble barium salts they are

thrown down and thus escape further decomposition, so that when working with baryta there is less chance of acid liquids than with lime.

Double Compounds.—Glucose forms double compounds with salts of a similar character as sucrose. Those with potassium and sodium chloride and with sodium bromide crystallize, but there are still many others which have not yet been obtained in the crystalline form.

Action of Oxidizing Agents.—Glucose is very liable to oxidation by various reagents. Nitric acid converts it into saccharic acid while at the same time oxalic and tartaric acids are formed. Free oxygen oxidizes an alkaline glucose solution; ozone leaves glucose intact when in neutral solution, but in an alkaline liquid oxidizes it completely into formic and carbonic acid and water. Hydrogen peroxide causes, especially in presence of a trace of an iron salt, a violent reaction, which takes place with development of heat, formic, acetic and tartronic acids and some other compounds being simultaneously formed.

Reduction of Cupric Oxide.—Glucose, especially in alkaline solution, readily absorbs oxygen, both free and combined, hence the salts of many metallic oxides, such as gold chloride, silver nitrate, as well as platinum chloride, mercuric chloride, and bismuth nitrate become reduced by an alkaline glucose solution. Cupric oxide is reduced to cuprous oxide, and this in proportion to the quantity of glucose, provided the conditions are always similar. This property is made use of in the determination of glucose with Fehling's solution, an alkaline solution of cupric hydroxide. In this process the content of a glucose solution is ascertained by the quantity which is required to transform all the cupric oxide contained in a known quantity of the standard copper solution into cuprous oxide, or by weighing the quantity of cuprous oxide which has been precipitated on boiling a known quantity of the glucose solution with an excess of Fehling's solution. During the oxidation the glucose is converted into organic acids, the character of which is not yet fully known.

Iodine dissolved in borax solution slowly oxidizes glucose into gluconic acid, while this reagent does not attack ketoses. Romijn has based a method of determining glucose in the presence of fructose on this reaction.*

Glucosides.—On introducing gaseous hydrochloric acid into a solution of glucose in methyl alcohol, methyl glucoside is formed and water split off.

$$C_6H_{12}O_6 + CH_3OH = C_6H_{11}O_5O.CH_3 + H_2O.$$

This glucoside can more easily be prepared by boiling 1 part of glucose for half-an-hour to an hour in a reflux condenser with 4 parts of acetone-free methyl alcohol which has previously been dehydrated by redistillation over quicklime and then heated with 0·25 per cent. of gaseous dry hydrochloric acid. After that the liquid is heated in a sealed tube in a water-bath for 50 hours, evaporated to one-third of its volume, and allowed to crystallize. Besides methyl glucoside,

* "Zeitschr. anal. Chem." 1897, 349.

also ethyl, propyl, isopropyl, amyl, allyl and other glucosides have been prepared. All these share with the natural glucosides extracted from plants the properties of not reducing Fehling's solution, of being stable towards alkalis, and of becoming hydrolysed by acids with the liberation of the glucose. Numerous attempts to form sucrose by these or similar reactions from glucose and fructose have been unsuccessful.

Action of Yeast.—Glucose may be decomposed by yeast, and since it needs no previous inversion the fermentation proceeds more rapidly than with the sucrose. The organisms of the lactic and butyric acid fermentations as well as *Leuconostoc* attack it in the same way as with sucrose.

Detection of Glucose.—When it is necessary to state without doubt that a sugar is actually glucose, we can make use of its property of oxidizing into saccharic acid, which is only formed from glucose or glucose-containing groups, e.g., sucrose. To this end 5 grms. of the sugar are evaporated on the water-bath with 30 c.c. of nitric acid of 1·15 spec. gravity to a syrupy consistency and the excess of acid expelled by adding water and evaporating again. The residue is dissolved in 20 c.c. of water, carefully neutralized with potassium carbonate while still hot, and a few drops of acetic acid added ; this addition is repeated after again evaporating. If glucose has originally been present, the potassium salt of saccharic acid crystallizes, and is drained off on a porcelain plate, re-crystallized from as little water as possible, re-dissolved, neutralized with ammonia, and precipitated by silver nitrate; 5 grms. of glucose yield 2 grms. of the silver salt dried in a desiccator over sulphuric acid. It must, however, not be forgotten that the simultaneous presence of other readily oxidizable sugars can cause glucose to pass over into oxalic acid along with them.

Osazone.—Another characteristic test for glucose is its osazone, which is prepared in the following way :—

One grm. of anhydrous glucose if dissolved in 100 c.c. of water and mixed with 5 c.c. of a solution of 40 grms. of phenylhydrazine and 40 grms. of glacial acetic acid, and the volume made up to 100 c.c., will yield, after being heated for one hour at 100° C., a crystalline precipitate of glucosazone, the weight of which is, after cooling, filtering, washing and drying, 0·32 grms. Its melting point is 205°, provided that care is taken in the determination to heat the substance rapidly, for if carried out otherwise too high a value is obtained.

Colour Reactions.—A number of colour tests are yielded by glucose, such as a red coloration with a freshly prepared sulphuric acid solution of α-naphthol, but none of these is characteristic of glucose alone, since such colour reactions, as has already been pointed out when dealing with sucrose, are common to other sugars also.

III.—Fructose (Levulose)

Occurrence.—In the sap of perfectly ripe sugar cane little or no fructose seems to occur, but it may be found in unripe or overripe canes. In unripe cane it is principally present in the parts still growing, from which it disappears as the cane ripens. In dead canes, where the sucrose has already undergone partial inversion, fructose occurs, of course, as one of the products of this inversion.

Crystalline Forms.—Fructose appears in the form of fine hygroscopic needles or of compact crusts of transparent prisms, which are not hygroscopic, and possess a specific gravity of 1·669 at 17·5° C.

Specific Gravity of Solutions.—Fructose easily dissolves in water; its solutions possess the following specific gravity values at 17·5° C.

Per Cent. Fructose.	Specific Gravity.	Per Cent. Fructose.	Specific Gravity.
6	1·02150	16	1·06503
7	1·02575	17	1·06950
8	1·03012	18	1·07380
9	1·03447	19	1·07825
10	1·03870	20	1·08253
11	1·04303	21	1·08700
12	1·04747	22	1·09137
13	1·05175	23	1·09588
14	1·05620	24	1·10030
15	1·06053	25	1·10488

It is slightly soluble in cold absolute alcohol, but very soluble in hot and in dilute alcohol, and also in a mixture of alcohol and ether. It is also soluble in methyl alcohol, in glycerin, and in acetone.

Rotatory Power.—It deflects the plane of polarization to the left, and its levo-rotatory power exceeds the dextro-rotatory power of glucose and sucrose; the rotation is, however, much influenced by temperature, and diminishes when the latter rises.

Influence of Concentration and Temperature.—In concentrations up to 20 per cent. the specific rotation of fructose diminishes by 0·67° for every degree C. rise in temperature. For a concentration of 10 per cent. the specific

rotation is according to Jungfleisch and Grimbert at 20° C. — 90·18 arc degrees or — 134·1 degrees Ventzke. At higher concentrations these values become greater, so the specific rotation at the same temperature for a concentration equal to 5 per cent. is — 95·22 arc degrees or — 143·3 degrees Ventzke.

Bi-rotation.—Fructose shows birotation but not to such an extent as glucose. Jungfleisch and Grimbert have shown that at 7° C., and for a concentration of 9·75 per cent., values for αD = — 97·33°, — 96·11°, — 95·11°, and — 94·77°, are given 35, 55, 75, and 105 minutes respectively after the moment of the preparation of the solution, and that afterwards the rotation remains unchanged.

Influence of Foreign Bodies.—The rotatory power of fructose is considerably influenced by all kinds of foreign bodies; it is for instance reduced by alcohol and by hydrochloric acid. Alkalis diminish it too, but this can be ascribed to the formation of fructosates, which causes the amount of uncombined fructose to decrease.

Optical Refraction.—The values for the refractive indices of fructose solutions are given in the following table by Stolle. The figures in the first column apply to solutions which were obtained 10 minutes after the preparation of the solution, those in the second after six hours, and those of the third after 24 hours.

Concentration of the anhydrous Fructose.			Specific Gravity $\frac{17·5}{4}$			Refractive Index.		
1·0090	1·0091	1·0091	1·00236	1·00245	1·00244	1·33448	1·33448	1·33448
2·0100	2·0100	2·0105	1·00610	1·00610	1·00635	1·33596	1·33558	1·33597
4·0110	4·0016	4·0124	1·01406	1·01420	1·01441	1·33872	1·33854	1·33882
8·0073	8·0074	8·0091	1·02932	1·02933	1·02955	1·34447	1·34421	1·34456
12·0250	12·0263	12·0275	1·04513	1·04524	1·04534	1·35008	1·34991	1·35036
15·9990	15·9999	16·0055	1·06112	1·06017	1·06050	1·35572	1·35545	1·35591
25·0160	25·0190	25·0187	1·09466	1·09479	1·09478	1·36825	1·36846	1·36814

Behaviour of Fructose on Heating.—On heating dry fructose for some time above its melting point, it becomes partially decomposed and forms an amorphous yellowish mass. On heating it *in vacuo* at 140° to 160° C., water splits off and fructosan (levulosan) remains to the amount of about 90 per cent. of the weight of the fructose used.

Moist fructose is decomposed even at the lower temperatures and gives off water and carbonic acid. It is therefore very necessary when drying substances containing fructose for determining the moisture in cane sugar-house products, to carefully avoid decomposing this sugar, otherwise too high a value for the moisture will result. It is advisable to dry such substances first at 60° C. and afterwards to drive off the last traces of water at 100° C. On further heating, fructose gives off much vapour and leaves behind a carbonaceous mass, which on continued heating in presence of the oxygen of the air undergoes total combustion.

Calorific Value.—The calorific value of fructose as determined in the bomb calorimeter is 3755·0 kg./kg. calories, or 6759·0 B.T.U.

Decomposition on boiling Aqueous Solutions.—On boiling fructose with water it is very rapidly decomposed: 4 grms. of fructose boiled in a platinum vessel attached to a reflux condenser for 60 hours with 50 c.c. of water yield a brown-coloured liquid, having an acid reaction. The rotatory and the reducing powers are thereby diminished by 10 per cent. and about 1 per cent. of the sugar becomes unfermentable. If the fructose solution is heated to temperatures above 100° C., the decomposition is much quicker and more intense, and gives rise to the formation of a number of acids.

Action of Acids.—Very dilute acids heated for a long time with fructose solution change its rotatory power, whilst more concentrated mineral acids (e.g., 5 per cent. hydrochloric or sulphuric acid) split it up into formic, levulinic, and humic acids with the simultaneous formation of furfural and carbonic acid.

Action of Alkalis.—Dilute alkalis, as well as potassium and sodium salts of organic acids, exert an action on fructose analogous to that on glucose, and the final products are identical. Especially at high temperatures, fructose is on partial decomposition converted into fructose, glucose, and mannose, the composition of which mixture is the same as that obtained in the transformation of glucose. The strong levo-rotation of the fructose hereby gradually diminishes till the mixture shows no further rotation. During this transformation more acids are formed by decomposition than in the case of glucose, while decomposition is always more rapid with fructose than with the former sugar. In practical work, however, much difference cannot be observed in the products of decomposition of glucose and fructose, as both their character and their mutual proportion are very much alike.

Alkali Fructosates.—The fructosates of potassium and sodium can be prepared in exactly the same way as the corresponding glucosates; they

are extremely hygroscopic and soon become decomposed with the formation of colour. Their general formula is:

$$C_6H_{11}O_6Na$$

Calcium Fructosate.—Calcium fructosate is chiefly employed for isolating fructose from its solutions. To this end the solution is cooled down to a few degrees below freezing point and mixed with 6 parts of fresh and very finely powdered quicklime to every 5 parts of fructose present. After that the mixture is stirred and allowed to crystallize. After 24 hours' standing the calcium fructosate is found to crystallize in the form of fine needles, which are centrifugalled, suspended in ice water, and decomposed with carbonic acid, which liberates the fructose. All these operations must be performed in the cold, as the fructosate is easily decomposed by heat.

Lead Fructosate.—Neither neutral nor basic lead acetate throws down fructose from its solution in pure water, but the last-mentioned reagent precipitates this sugar in part, so long as salts occur in the same solution with which basic lead acetate forms insoluble compounds. When in this case a precipitate of a lead salt is formed, it carries down a part of the fructose as a plumbic salt. As in the clarification of cane sugar products the addition of basic lead acetate is only to form an insoluble precipitate with compounds in the solutions, this reagent will also eliminate a part of the fructose from the product. For this reason clarification with lead subacetate or basic lead acetate is not applicable when it is necessary to determine the content of reducing sugars in the filtrate. Such solutions are better clarified with neutral lead acetate.

Fructose is quantitatively precipitated by ammonical lead subacetate (i.e., basic acetate of lead to which ammonia is added till a precipitate is just about to form). The precipitate is white at first but becomes pink after some days owing to partial decomposition.

Double Compounds.—As in the case of sucrose and glucose, fructose combines with salts to form double compounds; of these the following have already been obtained in the crystallized form by evaporating their concentrated aqueous solutions over sulphuric acid: calcium chloride, bromide, and iodide; strontium chloride, bromide, and iodide, and barium iodide. Their formulæ are respectively:—

$C_6H_{12}O_6 . Ca Cl_2 + 2 H_2O$; $C_6H_{12}O_6 . Ca Br_2 + 4 H_2O$; $C_6H_{12}O_6 . CaI_2 + 2 H_2O$; $C_6H_{12}O_6 . Sr Cl_2 + 3 H_2O$; $C_6H_{12}O_6 . Sr Br_2 + 3 H_2O$; $C_6H_{12}O_6 . Sr I_2 + 4 H_2O$; and $C_6H_{12}O_6 . Ba I_2 + 2 H_2O$.

These double compounds readily dissolve in water but they are dissociated in aqueous solution, so that their solutions exert a rotation equal to that of

the fructose which they contain. Compounds with other salts have not yet been obtained in the crystalline form, but they undoubtedly exist as will be shown in the chapter on *Molasses*.

Action of Oxidizing Agents.—Nitric acid oxidizes fructose into formic, oxalic, tartaric acid, &c., without the formation of saccharic acid. Hydrogen peroxide yields, especially in presence of a trace of an iron compound, acetic, formic and tartronic acid. The oxidation products of fructose by the action of metallic oxides are identical with those of glucose, the only difference in the decomposition being that fructose is more quickly oxidized than glucose. Iodine dissolved in borax solution does not oxidize fructose, and by means of this reaction it may be differentiated from glucose.

Fructosides.—A substance analogous to methyl glucoside is obtained from fructose by dissolving one part of crystallized fructose in nine parts of anhydrous methyl alcohol and adding so much solution of gaseous hydrochloric acid in methyl alcohol that the mixture contains about 0·5 per cent. of hydrochloric acid. The mixture is allowed to stand for 48 hours at 35° C., it is then neutralized with silver carbonate and evaporated. The fructoside does not crystallize nor does it reduce Fehling's solution, but it becomes hydrolysed by boiling with dilute acid with the re-formation of fructose.

Action of Yeast.—Fructose is liable to the action of all the fermentative agents which attack glucose; it ferments with wine or beer yeast, though more slowly than glucose, but finally yields the same products in the same proportion; it is also attacked by the lactic and butyric acid ferments.

Detection of Fructose.—The strong levo-rotatory power and the property of forming an insoluble compound with lime are characteristic of fructose, as well as the reaction of yielding neither mucic nor saccharic acid on oxidation with nitric acid. But besides these the quantity of osazone which it yields with phenylhydrazine is a good confirmatory test. On heating 1 grm. of anhydrous fructose dissolved in 100 c.c. of water for an hour with 5 c.c. of the phenylhydrazine and glacial acetic acid mixture mentioned on page 38, the same glucosazone, as in the case of glucose, is precipitated, but its quantity is 0·70 grms., or almost twice as much as in the former case. The melting point of the glucosazone is of course also 205° C.

Methylphenyl Glucosazone.—Another good characteristic is the methylphenylosazone, for this compound does not form from aldoses at all, and from the ketoses only of fructose in a crystallized form. In order to obtain the crystallized precipitate a solution of 1·8 grms. of fructose in 10 c.c. of water is mixed with 4 grms. of methylphenylhydrazine and so much alcohol that a limpid solution is obtained. Now 4 c.c. of 50 per cent. acetic acid are added,

the mixture is heated from 5 to (at a maximum) 10 minutes in a water-bath and allowed to cool in a beaker covered with a glass plate. After some time, not exceeding two hours, reddish crystals amounting to 80 per cent. of the fructose used are formed, these may be recrystallized from hot 10 per cent. alcohol and should have a melting point of 153° C.

Colour Reactions.—Fructose, even in very dilute solution, may be detected with great ease by a great number of colour reactions, but here also most of these tests do not apply solely to fructose but likewise to other sugars, for instance sucrose, which in most of the reactions is hydrolysed first into glucose and fructose, the latter then giving the reaction as though originally present. Thus the colour reactions with the α- and β-naphthol and with resorcinol and sulphuric acid apply as well to fructose as to raffinose, sucrose, and the other ketose, sorbose.

The colour reaction with ammonium molybdate and acetic acid is the only one which can be claimed to be characteristic for fructose. It should be noted that, except acetic acid, no other organic or inorganic free acid must be present in the solution. On adding to 10 c.c. of a 4 per cent. ammonium molybdate solution, 10 c.c. water, 0·2 grms. of glacial acetic acid and a trace of fructose, and heating the mixture in a water bath at 95°-98° C., after three minutes a deep blue coloration appears, owing to the reduction of the molybdic acid. Other sugars produce this coloration less rapidly and less intensely.

This reaction does not occur in the presence of free acids other than acetic acid; these may be removed by adding lead acetate, precipitating the excess of lead with sodium carbonate, and acidifying the filtrate with acetic acid.

IV.—Invert Sugar

Although invert sugar is not a simple sugar but consists of a mixture of equal parts of glucose and fructose, it occurs so largely that some particulars of it may be mentioned under this separate heading.

Preparation.—Invert sugar is prepared commercially by heating a sucrose solution with a very small quantity of acid until the solution, when examined by the polarimeter, shows the levo-rotation corresponding with its concentration.

Herzfeld calculated the following table for the levo-rotation of a completely hydrolysed sucrose solution for different concentrations at the temperature of 20° C.

Per Cent. Sucrose.	Per Cent. Invert Sugar Obtained.	Levo-rotation.		α D 20.
		In ° Ventzke.	In Arc degrees.	
10	10·53	12·2	4·22	— 20·04
11	11·58	13·5	4·65	— 20·08
12	12·64	14·7	5·08	— 20·11
13	13·68	16·0	5·52	— 20·15
14	14 74	17·2	5·95	— 20·19
15	15 79	18·5	6·39	— 20·23
16	16·84	19·9	6·83	— 20·27
17	17·90	21·0	7·27	— 20·30
18	18 95	22 4	7·71	— 20·34
19	20·00	23·6	8·15	— 20·38
20	21·05	24·9	8·60	— 20·42
21	22·10	26·2	9·04	— 20·46
22	23·16	27·5	9·49	— 20·49
23	24·21	28·8	9·94	— 20·53
24	25 26	30·1	10·39	— 20·57
25	26 32	31·4	10·58	— 20·61

An invert sugar solution for analytical purposes is prepared, according to Soxhlet's method, by dissolving 9·5 grms. of sucrose in 700 c.c. of hot water, heating it with 100 c.c. of N/5 solution of hydrochloric acid for half-an-hour on a water-bath at 100° C., cooling rapidly to 20°, neutralizing it with the required amount of N/5 sodium hydroxide solution, and making it up with water to 1000 c.c., by which operation a solution of exactly 1 per cent. of invert sugar is obtained.

Rotatory Power.—The specific rotation of invert sugar is equal to the difference in those of the two components, and is consequently much affected both by differences in concentration and in temperature.

Influence of Temperature on Rotation.—Lippmann* found these figures for concentrations of 17·12 invert sugar (equal to 16·38 sucrose).

Degrees C.	0	10	20	30	40	50	60	70	80
α D	−27·9	−24·5	−21·4	−18·0	−15·2	−12·0	−8·5	−5·8	−2·0

which agree very well with those calculated by Tuchschmid's formula :—

$$\alpha \frac{t}{D} = -(27{\cdot}9 - 0{\cdot}32\,t).$$

* "Berliner Berichte," 13, 1823

According to this formula there exists a temperature, viz., 87° C., at which the rotation of invert sugar is zero ; of this property use is made in sugar analysis, although at so high a temperature invert sugar is liable to be decomposed, and the figures for the rotation thus obtained will therefore not be entirely reliable.

Influence of Concentration on Rotation.—At higher concentrations the levo-rotation of invert sugar increases as will be shown by Borntraeger's figures for a temperature of 20° C. (Concentrations being in grams per 100 c.c.).

Conc. =	5	10	15	20	25	30	40	50	60
$a\frac{20}{D}$ =	−19·75	−20·04	−20·32	−20·58	−20·84	−21·08	−21·53	−21·94	−22·30

In such experiments it should be remembered that by considerably diluting the hydrolysed solution, the specific rotatory power is at first further to the left than after standing some time. In these determinations one must therefore take the precaution to dilute the sucrose solution, previous to the inversion, to about the same concentration as that at which it will be polarized after that operation.

Unlike the polarization of the simple sugars, in which the commonly occurring quantities of salts or acids do not affect the rotatory power to any noticeable extent, that of the invert sugar is usually much influenced by acids or salts, since it is mixed for the purpose of the inversion with large percentages of hydrochloric acid.

Gubbe* represents the influence of hydrochloric acid on the rotation of invert sugar at 20° C. at a concentration of hydrochloric acid of from 0·3 per cent. (S) by the following formula :—

$$a\frac{20}{D} = -(19{\cdot}55 + 0{\cdot}32621\ S).$$

Herzfeld's directions † prescribe 5 c.c. of hydrochloric acid of 1·188 sp. gr., corresponding to 6 grms. of 38 per cent. acid or to 2·2 grms. of hydrochloric gas, which is dissolved in a volume of 100 c.c. This concentration will therefore, according to the above formula, increase the rotatory power of the invert sugar by $0{\cdot}32621 \times 2{\cdot}2 = 0{\cdot}72°$.

Langguth Steuerwald‡ uses in his method of inversion 30 c.c. hydrochloric acid of 1·10 sp. gr., or 6·6 grms. of HCl, which renders the influence of that acid still stronger than in Herzfeld's prescription.

The influence which is exerted by salts is still more considerable than that of the free acid. Since therefore the presence of hydrochloric acid, the concentration of the solution, and the temperature, are factors which have a consider-

* "Zeitschr. Rübenzuckerind" 1884, 1355. † "Zeitschr. Rübenzuckerind." 40, 194, 208, 443.
‡ "Int. Sugar Journal," 1913, 489.

able influence on the rotation of invert sugar, great care should be taken when polarizing sugar products after inversion, to adhere exactly to the prescribed directions as to weight, concentration, and temperature, and to use the formula which is adapted to these conditions.

The action of acids, bases, &c., on invert sugar is of course the same as has been described for glucose and fructose.

Nomenclature.—In this work we adopt the system of grouping the glucose, the fructose, and the mixture of both these sugars known as invert sugar, under the common name of " reducing sugars." The amount of these reducing sugars present is determined from the quantity of copper oxide reduced, without making any distinction between the individual sugars in the mixture. Wherever, therefore, in this work the term " reducing sugars " is used, it always means that sugar or mixture of sugars which has the property of reducing Fehling's solution.

The explanation of the reason why we have adopted the terms glucose, fructose, and reducing sugar, instead of the more widely used expressions dextrose, levulose, and glucose, is given in the Preface.

V.—Starch

Starch occurs in the chlorophyll-bearing cells of the leaves, around the bundles in the leaf-sheaths and around those of the top of the stem, whilst in the adult joints little or no starch is found.

It occurs in very minute granules. It produces a blue colour on treatment with a solution of iodine in potassium iodide.

VI.—Fibre

Occurrence.—All the solid substances of the cane are grouped under the name of " cellulose " or " fibre." The first-mentioned term is not correct, as the fibre, properly speaking, only partly consists of cellulose, the other constituents being lignin and a pentosan in proportions which vary according to the species of the cane and its age, as will be seen from the table given overleaf.

By " fibre " is understood that portion of the vegetable tissue which resists boiling with first 1·25 per cent. sulphuric acid for one hour and then with 1·25 per cent. solution of caustic soda for the same period of time.

	Black Java.	Seedling No. 36.	Seedling No. 100.	Seedling No. 103.	Seedling No. 105.	Seedling No. 139.	Seedling No. 247.	Black Java.			
								5	6	9	12
								Age in Months.			
Fibre	55·94	62·57	53·68	49·29	58·39	56·76	56·22	38·36	39·75	41·36	50·3
Pentosan	22·23	23·68	23·32	21·46	22·54	24·08	23·80	25·70	28·50	30·82	31·50
Ash	1·98	1·47	3·49	3·12	2·23	1·68	1·98	3·45	3·35	4·02	3·95
Nitrogenous Substance, etc.	2·0	2·0	2·0	2·0	2·0	2·0	2·0	2·0	2·0	2·0	2·0
Lignin (by difference) ..	17·75	10·28	17·51	24·13	14·84	15·48	16·00	30·49	26·40	21·80	12·25

The pentosans are determined by Tollen's method in which the substance is distilled with hydrochloric acid, the distillate neutralized with sodium carbonate, then acetic acid and a solution of phenylhydrazine acetate added, and the precipitated furfural hydrazone finally weighed.

Properties of Cellulose.—Cellulose may be hydrolysed by sulphuric acid and is thereby converted into glucose. The pentosan which may be isolated from the fibre by extraction with dilute soda solution and precipitation with acetic acid and alcohol is, when freshly precipitated, soluble in hot water, but on cooling the solution becomes opaque and gelatinous. Alcohol does not immediately precipitate the aqueous solution, but only after the addition of a little acetic acid or soda solution. The aqueous opaque solution becomes clear after addition of soda or potash, and these can dissolve still more of the gum, but it is precipitated by excess of lime or baryta and by lead acetate.

After being dried, the gum is only slightly soluble in water, but it completely dissolves, though slowly, in soda solution.

Rotatory Power of the Pentosan.—The solution of 5 grms. of cane gum in 100 c.c. of water obtained by the addition of as little soda as possible is slightly opalescent, but can be polarized after being allowed to subside for some days. It deflects the plane of polarization strongly to the left, and to such an extent that the specific rotation of the organic matter, free from moisture and ash, is about $aD = -80°$.

Composition of the Pentosan.—On hydrolysis with hydrochloric acid the gum yields (calculated to 100 parts of dry and ash-free material) 90 per cent. of sugar, chiefly xylose, accompanied by a small proportion of another sugar possessing a more positive rotatory power, probably arabinose or glucose. On distillation with hydrochloric acid of 1·06 specific gravity the pentosan

yields 46·3 per cent. of furfural which corresponds to 88·3 per cent. of xylan. Oxidation with nitric acid does not produce mucic acid but trioxyglutaric acid, a proof that the cane gum consists of xylan accompanied perhaps by araban. Browne* found 75 per cent. xylan, 24 per cent. araban, and 1 per cent. galactan.

Combinations of Cane Gum.—Cane gum enters into combination with bases. The soda compound is up to now the only one investigated; it dissolves in water to a viscous liquid, but is insoluble in alcohol. The soda content is 10 per cent. which corresponds with the formula $C_5H_8O_4)_2$ NaOH.

In English and American publications we often find the term "gum" used for other bodies which properly speaking do not belong to the class of gummy matters, and might better be tabulated under the name of "organic non-sugar." In the *Hawaiian Planters' Monthly*, 1906, 374, Peck gives the following definition of what he understands as gum:—

"By this term is indicated all the impurities precipitated by subacetate of lead, and includes, naturally, a great many substances other than true gums, such as glucinic, melassinic and succinic acids, and perhaps partially aspartic acid. The precipitate, produced as in the sucrose and glucose determinations, with an exactly sufficient amount of the lead solution, and the addition of a few drops of acetic acid, was thoroughly washed first by decantation and then on the filter, until the washings gave no test for sugars with α-naphthol. The precipitate was then suspended in water and decomposed with sulphuretted hydrogen, filtered, concentrated, and weighed in a platinum dish. This was then ignited, and the weight of the ash deducted."

The question of the removal of cane gum from the juice is discussed in the chapter on *Clarification.*

Incrustating Matter.—The incrustating matter which in cane fibre accompanies the cellulose is, according to Browne and Blouin, lignin, and has the same composition as Lindsey and Tollens ascribe to the lignin from wood, viz., $C_{24}H_{26}(CH_3)_2O_{10}$. The former investigators report the following composition for cane fibre (protein, ash, fat, etc. excluded):—

	Per cent.
Cellulose (including oxycellulose) ..	55
Xylan	20
Araban	4
Lignin	15
Acetic acid	6
	100

* "Archief voor de Java Suikerindustrie." 1896, 1024.

Saccharetin.—Langguth Steuerwald* isolated from cane fibre a constituent occurring therein to the amount of 5 per cent. of the dry substance, to which he gives the name of saccharetin. He treated finely powdered cane fibre for a long spell with a diluted solution of caustic soda and precipitated from the solution thus obtained the equally dissolved cane gum or xylan by means of Fehling's solution. The liquid filtered off from the precipitate gave a precipitate of saccharetin after having been acidified by hydrochloric acid.

Properties.—Saccharetin is a light-yellow, amorphous tasteless body, which in a moist state has an aroma of vanillin, but in a dry state is odourless. It is very soluble in alcohol and in glacial acetic acid, but sparingly so in water, and not at all in ether, benzol, acetone, chloroform or carbon bisulphide. It dissolves in solutions of soda or potash and in ammonia with a yellow colour.

Saccharetin may be precipitated from these alkaline solutions by acids and from those in alcohol and in acetic acid by addition of water. The solution in ammonia gives a precipitate on addition of barium chloride and of calcium chloride. Ferric chloride colours the neutral solution dark, and on addition of alcohol a dark brown precipitate is thrown down, which, in a dry state, only contains 6·3 per cent. of iron.

Lead acetate yields yellow precipitates both in neutral and in alkaline solutions.

On treatment with phloroglucin and hydrochloric acid saccharetin is coloured intensively red and orange red with aniline and sulphuric acid, thus showing the lignin reactions. The alkaline saccharetin solution does not reduce Fehling's test solution, and it does not possess any rotatory power. Neutral and alkaline saccharetin solutions are completely decolorized by bone-char. Elementary analysis gives a molecular formula of $(C_5H_7O_2)_n$. According to Steuerwald, saccharetin belongs to the phlobaphenes, and may be considered the aromatic compound in wood or what is otherwise called lignin.

Saccharetin does not occur in the sap of the cane and is not dissolved in the expressed juice, but is present as a constituent of the fine fibre or " cush-cush " in suspension. The acid or neutral cane juice does not dissolve it from that fibre, but as soon as the juice is limed to an alkaline reaction it gets dissolved to some extent. The same thing occurs if the cane bagasse on the carriers is macerated with alkaline sweet-water from the presses, or with alkaline last-mill juice. As, however, owing to the very close incrustation of cellulose with lignin, the latter substance dissolves only very reluctantly, the amount of that incrustating body which enters into solution is very small after all. But, since common clarification does not remove saccharetin, and since that body assumes a dark colour in contact with iron salts, it is

* " Archief voor de Java Suikerindustrie." 1911, 1543.
" Int. Sugar Journal," 1912, 53.

advisable to get as little saccharetin in the juice as possible; hence the use of sieves with fine meshes is to be recommended in straining the cane juice.

Colloid Water.—The fibre does not occur in an anhydrous state, but contains a certain amount of sugar-free colloid water of hydration, so that in the living organism of the cane stalk the fibrous parts are found sugar-free with their own water content along with the sugar-containing parenchyma cells.

When discussing the milling work we shall find opportunity for referring to this colloid water. The author has concluded from his experiments* that the colloid water amounts to about 20 per cent. of the weight of the dried fibre, while Langguth Steuerwald† found a figure of 22·4 per cent. at ordinary temperature and pressure, which value may considerably decrease at high pressure.

Solubility of Constituents of Cane Fibre in Water.—Cane fibre is not insoluble in water since on prolonged ebullition it gradually decreases in weight. Shorey‡ found that cane fibre from which all the constituents of the juice had been withdrawn by extraction with cold water yielded a soluble substance to boiling water, the amount of which increased the longer the treatment with hot water was continued. A quantity of finely divided cane, which after extraction with cold water contained 10 per cent. of fibre, only contained 9·3 per cent. of that constituent after boiling with water for five minutes, and no more than 8·8 per cent. when the boiling was continued for two hours. So that during that time no less than 12 per cent. of the fibre was dissolved and carried off. Krüger§ boiled 50 grms. of finely shredded cane repeatedly with 200 c.c. of water and dried the residue after 10, 15, and 20 extractions. The following table shows us that after 20 extractions the fibre content was 0·43 per cent. less than after 10, and that hot water therefore continuously dissolves parts of the cane fibre.

No.	10 × *a.*	15 × *b.*	20 × *c.*	Difference, *a*—*c.*	10 × *a.*	15 × *b.*	20 × *c.*	Difference, *a*—*c.*
I.	9·94	9·75	9·46	0·48	10·15	9·92	9·71	0·44
II.	9·98	9·78	9·59	0·39	10·18	9·93	9·74	0·44
III.	10·13	9·92	9·73	0·40	10·04	9·84	9·61	0·43
Average ..	10·01	9·81	9·59	0·42	10·12	9·90	9·69	0·44

* "Sugar Cane," 1898, 129.
† "Archief voor de Java Suikerindustrie," 1912, 1321.
‡ "Sugar Cane," 1898, 129.
§ "Mitteilungen der Versuchs—Station, Kagok" II., 4.

The author has stated that the constituent which hot water dissolves from the fibre has a decided dextro-rotatory power. For this reason the sucrose content of bagasse is found to be too high if the boiling is too prolonged. A period of ten minutes, if 30 grms. of bagasse and 300 c.c. of water are used, is quite sufficient to extract all the sugar, and at the same time does not cause any appreciable quantity of the fibre to pass over into the liquid. Owing to the fact that hot water extracts some of the fibre, the determination of the fibre content in sugar cane and in bagasse by extraction with boiling water is apt to yield lower results for that constituent.

Previous to the presence of the saccharetin (which is soluble in alcohol) being known as a regular constituent of the cane fibre, we recommended the determination of the fibre content in cane and in bagasse by extraction with alcohol. In order to prevent coagulation of albuminous matter, the finely shredded cane or bagasse was washed first with a little hot water and afterwards treated in an extraction apparatus with strong alcohol till all the sugar had been removed. It is specially in the case of young cane that the fibre content found by water extraction differs much from that determined by extraction with alcohol.

	Age of the Cane in Months.			
	5	6	9	12
Fibre by water extraction ..	7·36	7·99	8·38	10·52
Fibre by alcohol extraction ..	8·0	8·65	8·35	10·94
Difference on 100 parts of fibre	8·2	7·5	5·3	3·9

Experiments by Van Welie* showed him that on extraction of cane fibre by means of alcohol yet other bodies than saccharetin pass over from the cane fibre into the solution. Langguth Steuerwald† showed likewise that on extraction of cane fibre with alcohol, matter from the solid fibre was dissolved, so that extraction by alcohol also yields values for the fibre content which are too low. The real figure for the fibre content is to be found in an indirect way by subtracting from the total dry substance of the cane the amount of dry substance belonging to the juice, found by multiplying the sucrose content by 100 and dividing the product by the quotient of purity of the expressed juice.

A cane with 29·65 per cent. of dry substance, a sucrose content of 13·16, and a juice purity of 85·2 thus contains $29{\cdot}65 - \frac{13{\cdot}16 \times 100}{85{\cdot}2} = 14{\cdot}20$ per cent. of fibre.

* "Archief voor de Java Suikerindustrie," 1913, 458.
† "Int. Sugar Journal," 1913, 542.

Calorific Value.—As cane fibre forms the principal source of fuel for the cane sugar industry, the determination of its calorific value is of considerable importance. This value is given here for the fibre of seven different varieties.

Fibre of the Varieties.	I	II	III.	IV	V.	VI.	VII.
	Moisture	Ash.	Calorific Value in B.T.U.				
			On the moist fibre.	Calculated to dry fibre from I & III.	Calculated to dry organic substance from II & IV.	On dried fibre.	Calculated on dry organic substance from II & III.
Black Java, 1906 ..	9·60	2·05	7587	8388	8559	8172	8370
Seedling No. 247 ..	9·12	1·85	7686	8460	8620	8325	8482
,, ,, 36 ..	8·32	1·42	7695	8492	8449	8171	8341
,, ,, 100 ..	8·46	3·03	7553	8249	8256	7996	8246
,, ,, 105 ..	8·64	1·79	7616	8334	8503	8125	8273
,, ,, 139 ..	9·26	1·42	7540	8329	8442	7862	7974
Black Java, 1907 ..	8·69	1·68	7772	8514	8681	8154	8293

Column V shows the calorific value for the calculated organic substance, ascertained in the moist samples and calculated on 100 parts of dry and ash-free substance. In column VII is given the same value, but this time determined in the previously dried samples, and again calculated to 100 parts of dry and ash-free substance. The figures for these latter values are all a little low, which, as all the water driven off was merely hygroscopic, is difficult to explain, because in that case the figures in columns V and VII should have been identical. All of the figures stated are averages of three determinations in which very concordant results were obtained, and because in every one of the cases the difference is on the same side, we may safely conclude to their having one common cause. Whatever this may be is not known yet, but as the fuel is used in the moist and not in the anhydrous condition, the figures of column V apply more to the conditions met with in practical working. Therefore we suggest using the figures of column V when it is necessary to have a base for the calculation of heat produced, consumed, and lost in a sugar factory, or for similar calculations, and then we can take the average figure of 4751 kg./kg. calories or 8552 B.T.U.

Coates* found for the calorific value of Louisiana cane 8325 B.T.U. or about

* "Louisiana Planter," 1906, 236.

the same as found in Java. Kerr and Percy* found for the fibre of Louisiana cane 8368 B.T.U. and of Cuba cane 8435.

When making such calculations it must not be forgotten that these figures represent the highest values, as the determinations were made at the common temperature in the calorific bomb, where the latent condensation heat of the water, both that already existing and that formed by the combustion, is included in the figure for the calorific value. This heat escapes, however, in practical working, when the vapour is driven off into the chimney and thus carries away that latent heat. In calculations of the real calorific value we therefore subtract from the calculated calorific effect of the sucrose and fibre the figure for the latent heat of all the water, that which already existed in the fuel as well as that which is formed by combustion of the organic substances.

Elementary Constitution of Cane Fibre.—The elementary constitution of a few samples of cane fibre calculated for 100 parts of water and ash-free substance is as follows :—

Fibre of the Varieties.	Carbon.	Hydrogen.	Oxygen.	Nitrogen	Calorific Value.		Ratio H : O.
					V.	VII.	
Seedling No. 36 ..	48·45	6·45	44·94	0·16	8449	8341	6·97
,, ,, 100 ..	47·88	6·55	45·39	0·18	8256	8246	6·93
,, ,, 105 ..	48·14	6·43	45·25	0·18	8503	8273	7·04
,, ,, 139 ..	47·13	6·30	46·43	0·14	8442	7974	7·37
Black Java ..	46·82	6·67	46·33	0·18	8681	8293	6·94

Comparisons between Cane Fibre and Wood.—Cane fibre, therefore, closely resembles wood, as may be seen from the enumeration of the composition of several kinds of wood, to be found in Beilstein's *Organische Chemie*, I, 1078.

Kind of Wood.	C.	H.	N.	O.	O : H.	Ash.
Yoke Elm	49·48	6·08	0·84	43·60	7·17	1·62
Beech	49·89	6·07	0·93	43·11	7·10	1·06
Poplar	50·31	6·32	0·98	42·39	6·71	—
Birch	50·61	6·23	1·12	42·04	6·75	0·85
Oak	50·64	6·03	1·28	42·05	6·97	1·65
Willow	51·75	6·19	0·98	41·08	6·62	2·00

* "Louisiana Expt. Station Bulletin," No. 117.

The resemblance becomes still more pronounced on comparison of the calorific values of wood and cane fibre. Though not properly belonging to this series, the figures for the calorific value of cane trash (dry leaves) are given here to make our list as complete as possible.

Kind of Material.	Moisture.	Calorific Value in B.T.U.	
		Undried.	On 100 Parts of Dry Material.
Beech	13·0	7502	8624
Fir	12·2	7960	9067
Oak	13·3	7182	8283
Pine	11·8	8073	9157
Old Teak	9·58	8114	8955
Young Teak	29·41	5947	8424
Cane Trash (leaves) ..	10·46	6663	7807

Difference in Comparison of Fibre.—In chemical composition the fibres of the various cane varieties do not show considerable differences, but their physical aspects do. The fibre from cane having a high fibre content is much more compact than that of cane possessing only a low fibre content. This is shown by the table given here, containing the weight in grms. of 100 c.c. of loosely shaken, finely chopped, air-dried cane fibre of various kinds having differing fibre contents.

Cane Variety.	Per cent. Fibre in the Cane.	Weights in grms. of 100 c.c. Fibre.	Number of Analyses from which the Average is calculated
Seedling No. 100 ..	9·77	5·45	3
Black Java ..	10·38	6·71	9
White Manila ..	11·80	6·38	3
Seedling No. 247 ..	13·85	6·97	8
,, ,, 139 ..	14·16	7·30	5
,, ,, 33a ..	15·59	7·23	3
,, ,, 36 ..	15·72	4·95	8

Composition of Ash from Cane Fibre.—Apart from the constituents already mentioned, the fibre obtained from cane by extracting all the other substances with alcohol still contains about 1 per cent. of albuminoid matter, and from 1 to 3 per cent. of ash, of which the composition is given here:—

Silica	80·57
Iron and calcium phosphates	6·87
Calcium carbonate	0·86
Potassium carbonate	11·70
	100·00

VII.—Pectin

Occurrence.—Some pectin or pectic substance is always found in cane juice. In that of certain varieties it is present in greater amount than in others, and the composition of the soil also seems to influence its presence.

Origin.—Farnell* found the origin of this pectin present in the juice to be the pectinogen which he encountered in cane fibre to the extent of about one per cent. The milling process allows part of the pectin to pass over into the juice.

Properties.—Pectin is only precipitated on the strong addition of lime as is the case in the carbonatation and sulphitation processes, and even then its removal is not a complete one, and therefore it may be expected in the clarified juice and in the molasses to a greater or less extent. It is soluble in water but insoluble in acidified alcohol; it does not yield sugar on being heated with dilute acids, nor furfural on distillation with hydrochloric acid. Its lime salt is somewhat soluble in water and more so in sugar solution. Lead acetate precipitates pectin from its aqueous solution. On evaporating 5 grms. of pectin from cane juice with 60 c.c. of nitric acid of 1·15 sp. gr., about 1 grm. of mucinic acid is obtained, corresponding to a content of about 30 per cent. of galactane groups in the pectin.

* "Int. Sugar Journal," 1923 630.

VIII.—Organic Acids

The organic acids of the sugar cane are aconitic, glycollic, oxalic, malic, succinic, tannic, and, in dead cane (as a product of decomposition), also acetic acid. Some investigators report having also detected citric and tartaric acids, but their allegations have not as yet been confirmed. In ripe canes the quantity of organic acid is only very small, and does not exceed 0·15 per cent., of which quantity 0·06 per cent. is present as free acid, and the rest combined with potash.

Glycollic Acid.—Glycollic acid occurs among other constituents in unripe grapes, and is also detected in beet juice bottoms after the juice has been heavily limed and has subsided. Its lime salt is soluble; the acid therefore is not removed by clarification, and it may be traced in the molasses in so far as it has not become decomposed during manufacture.

Oxalic Acid.—Oxalic acid is found in a great many plants, among others in beetroot leaves, wood sorrel, &c., whilst it is to be found as calcium oxalate in a number of fruits, tubers, &c. Calcium oxalate is insoluble in water, but dissolves in a sugar solution, especially when this is dilute and alkaline. On concentration it becomes insoluble and is therefore deposited in the last vessels of the evaporation plants in sugar-houses as incrustation, while it is no longer found in the syrup.

Malic Acid.—Malic acid is found in a great many fruits and juices; it is a very stable acid and does not become decomposed in the course of manufacture; its lime salt is soluble in water, so that the acid is not removed by defecation of cane juice with lime.

Succinic Acid.—Succinic acid, like malic acid, is a very stable acid, and like it also possesses a soluble lime salt.

Aconitic Acid.—The lime salt of aconitic acid is soluble, and therefore its presence may be detected in the clarified juices, syrups and molasses.

Acetic Acid.—Acetic acid is only to be found in dead or diseased canes as a product of the decomposition of the sugar. Its lime salt is soluble, and therefore remains in the juices after liming.

Other Acids produced.—The acids mentioned here originate from the cane and pass over into the juice, but during manufacture large quantities of organic acids are produced by the action of lime on the reducing sugars, and on sucrose. Their properties have already been mentioned on page 36 under the heading *Glucose*.

IX.—Cane Wax

Occurrence.—On the outside of the rind of the cane, especially near the nodes, we find a more or less considerable layer of wax, which is more conspicuous in some varieties than in others.

Properties.—This wax melts at 82° C., boils at 146° C., is insoluble in water and in cold alcohol, slightly soluble in cold ether or chloroform, but dissolves easily in hot alcohol, ether, and chloroform, and in benzene.

According to Wynberg* this material is not a single body, but a mixture of varying proportions of a primary alcohol of the formula C_{30} H_{62} O, with a second body, having the formula C_{33} H_{68} O, while possibly yet other alcohols or esters of a high melting point are present in it.

X.—Colouring Matter

Occurrence.—Besides the saccharetin already discussed under the heading of *Fibre*, various colouring matters occur in the cane and its juice. In the peripheric strata of the rind of the sugar cane there are cells containing the colouring matters, causing the differing coloration of the cane.

Chlorophyll.—One of these, chlorophyll, is the regular constituent found in every green plant cell. It is insoluble in water and in sugar solutions. It is in suspension in the expressed cane juice, for which reason it passes into the scums, not interfering any further with the process of manufacture.

Anthocyan.—A second colouring matter present in the rind is anthocyan, a violet-coloured substance which is found in the outer cells of dark-coloured canes, but is almost absent in white or yellow cane varieties. In contrast with chlorophyll, anthocyan is soluble in water and in sugar solutions, which explains why the juice of canes having a dark-coloured rind is so much more coloured than that of green or yellow canes. It also proves why the last-mill juice of coloured canes in which the juice of the hard rind is strongly represented is so much more coloured than the first-mill juice in which chiefly juice from the soft parenchyma cells is present. The purple colour of the anthocyan solution is changed to a dark green by the addition of lime, but the body is not precipitated until a strongly alkaline reaction is obtained by the addition of a great deal of milk-of-lime on clarification.

Sulphurous acid is incapable of bleaching anthocyan solutions; it decolorizes them to some extent, but after exposure to oxygen of the atmosphere the original colour returns.

* "Int. Sugar Journal," 1909, 521.

Polyphenols.—Although properly speaking not colouring matters, yet the class of bodies coming under the denomination of polyphenols or tannins is apt to cause dark colorations in the expressed cane juice either from the action of oxydases or of ferric compounds. According to the investigations of Schneller* the eyes, shoots and tops, *i.e.*, the assimilating parts of the cane contain polyphenols distributed in the vascular bundles. In the leaves they are found in and near the bundles connected with the stomata; in the top joints of the stalk, where sugar is being accumulated in the bundles as well as the parenchyma. These polyphenols assume a dark coloration on exposure to the air in combination with oxydases, but much more strongly so when coming into contact with ferric salts. The acid cane juice dissolves traces of iron from the rollers and from other machinery, and on oxidation to the ferric state these iron compounds form black compounds with the polyphenols. Sulphurous acid bleaches them, as by the reaction the iron is reduced to the ferrous state, but the coloration returns on exposure to the air, so that this bleaching process is only of short duration. Bone-char and similar absorbent agents remove the polyphenols from the juice, and thereby have a permanent decolorizing effect on cane juices and syrups.

Since the tops contain the largest proportion of polyphenols, the easiest way of decreasing the amount of these bodies in the juice is to top cane low, and use the tops for planting instead of grinding them along with the cane.

XI.—Nitrogenous Bodies

Occurrence.—Sugar cane contains very little nitrogenous substance; only the leaves and the green tops contain a rather considerable quantity, but in the ripe parts of the stalk only the buds are provided with it. A perfectly ripe cane, without the leaves, contains an average of 0·05 per cent. nitrogen, and the whole plant, without the roots, up to 0·08 per cent. Juice filtered through cloth was found to contain from 0·018 to 0·062 per cent. of nitrogen, being 0·036 on the average, and of this small amount a considerable portion was not dissolved, but was present in the form of a colloid, the amount of nitrogen decreasing greatly after filtration through filter paper.

* "Louisiana Planter," 1916, II, 236.

Albumin.—Among the nitrogenous bodies occurring in the sugar cane, albumin occupies only an insignificant place. In unripe cane its amount is larger than in fully ripe cane. It is not yet known to what class of albumin that found in the cane belongs.

Amides and Amino-Acids.—Several investigators report having encountered leucine, asparagine, glycocoll, and similar bodies in the cane juice ; but only asparagine with minor quantities of tyrosine and glutamine have been separated with certainty by Zerban. He found no more than 0·05 per cent. of the weight of the juice of these bodies, but it is not improbable that at the outset the amount of asparagine and glutamine was much larger, since both bodies are readily decomposed and may have become so partially during their isolation.

Bases.—Small portions of xanthine bases, especially guanin, appear to have been detected in cane juice.

Composition of Nitrogenous Substances.—According to Brown and Blouin* the various nitrogenous bodies of sugar cane may be stated as follows :—

	Percentage of Cane.
Albumin (coagulable and soluble in pepsin) ..	0·059
Nuclein, &c. (coagulable and insoluble in pepsin)	0·040
Albumoses and peptones (not coagulable) ..	0·033
Amino acids (aspartic acid)	0·145
Amino acid amides (asparagine)	0·232
Ammonia	0·008
Nitric acid	0·071
Total nitrogenous bodies ..	0·588

The author remarks, however, that the above percentages are subject to considerable variations according to the age and variety of the cane, manner of fertilization, and cultivation.

The amount of nitrogenous bodies occurring in exhausted molasses, in which product the impurities from the juice have of course accumulated, so far as they have not been deposited during manufacture, is likewise very small, and the total amount of nitrogen seldom exceeds 0·2 per cent. of the weight of the molasses.

* "Louisiana Exp. Station Bulletin,' No 91, page 5.

XII.—Incombustible Matter or Ash

Occurrence.—Sugar cane is a plant that absorbs very little mineral substance from the soil ; this is very clearly to be deduced from the minute quantity of ash it contains. The constituents vary in quantity between rather wide limits according to the quality of the soil, the manuring, and the variety of the cane, but each of them is found in every cane ash. The figures given below may be regarded as average examples.

	On 100 Cane.	On 100 parts of Ash.				
	I.	II.	III.	IV.	V.	VI.
Potash	0·153	23·0	12·56	25·63	7·66	38·23
Soda	0·010	1·5	5·67	2·26	6·45	1·30
Lime	0·018	2·7	6·50	3·24	12·53	5·19
Magnesia	0·014	2·1	5·08	3·22	6·61	5·76
Iron Oxide	0·003	0·5	—	—	0·56	1·13
Alumina	—	—	—	—	—	0·25
Silica	0·351	52·8	56·76	53·42	43·75	15·70
Sulphuric Acid	0·016	2·4	2·60	0·53	16·53	18·47
Phosphoric Acid	0·098	14·7	10·63	10·78	5·45	5·27
Carbon	—	—	—	—	—	0·98
Chlorine	0·002	0·3	0·20	0·92	0·21	4·52

I. Analysis by van Lookeren Campagne & v. d. Veen.
II. ,, ,, ,, ,, ,,
III. ,, Bonâme.
IV. ,, ,,
V. ,, Popp.
VI. ,, Brown and Blouin.

CHAPTER II

PROPORTION AND DISTRIBUTION OF THE CONSTITUENTS OF SUGAR CANE

The constituents of the cane mentioned in Chapter I occur in very different proportions during the whole vegetating period of the cane, and are not to be found in the same proportion in every part of the stalk. This is not to be regarded as one homogeneous mass, but as a living organism, every part of which has to fulfil its special function, and has therefore a constitution differing from that of any other part.

According to Went's investigations,* which were modified by those of Kamerling,† and of Geerts‡ we may represent the formation, transformation, transposition, and accumulation of the sugars in the cane in the following way.

Formation of Sugar in the Cane.—Under the influence of light the chlorophyll-bearing tissues of the leaves form carbohydrates from water and the carbonic acid of the atmosphere, probably in the first place sucrose, this formation increasing with the intensity of the light. The sucrose is transferred to the stalk, but if more sucrose is formed than can be so transferred (as will occur in the day time), the surplus is deposited in the form of starch. This starch is, however, dissolved again at night time and then transferred to the stems in the form of glucose. The leaves contain also a little fructose, which is formed by inversion of part of the sucrose.

Transfer of the Sugars.—When considering the further behaviour of the sugars one must make a distinction between those which are transported from old leaves and those from young ones. Sugars coming from an old leaf reach the stem in an already adult joint. Here the sucrose does not undergo any change; the reducing sugar is used for respiration, and is thereby converted into water and carbonic acid, so that its amount constantly diminishes without however disappearing entirely.

* "Archief voor de Java Suikerindustrie," 1896, 605.
† *Ibid.*, 1904, 780.
‡ *Ibid.*, 1923, 1159.

Transformation of the formed Sugar.—When the sugars come from a younger leaf, where in general the assimilation is more powerful, and the quantity of newly-formed carbohydrate more considerable, they reach the stem at its top. Here the sucrose becomes inverted, and the more so, the more rapidly the top grows. This inversion increases the isotonic power of the liquid in the cell, which in its turn promotes the growth. The reducing sugar is partly employed in the formation of the fibre, while another portion reaches the top and combines with nitrogenous substances to form albuminoids, or is deposited there as starch for subsequent use.

Disappearance of the Reducing Sugar.—That part of the reducing sugar which remains in the top is gradually consumed during respiration, while part of it is also deposited temporarily as starch in the leaf-sheaths and in the top joints.

Intensity of the Assimilations.—Kamerling in Java found that in the course of a sunny forenoon the dry substance content of powerfully assimilating cane leaves increased by 15 per cent. notwithstanding that carbohydrates had been continuously transferred to the stem during that time. Taking into consideration that forenoon only, and assuming the weight of the assimilating leaves per stalk to be 300 grms., having early in the morning a dry substance content of 20 per cent., then the leaves of one cane stalk produced at least $300 \times 0{\cdot}20 \times 0{\cdot}15 = 9$ grammes of carbohydrates.

Browne and Blouin* in Louisiana made analyses of the leaves of a young cane at night and in the morning, and from different parts of the stalk.

	Evening per cent.	Morning per cent.
Brix	5·77	5·09
Sucrose	0·94	0·54
Glucose	0·63	0·86
Fructose	0·69	0·77
Ash	1·42	1·24
Free acids	0·27	0·27
Combined acids	0·62	0·54
Nitrogenous bodies..	0·15	0·18
Gums	0·30	0·17

We notice in the juice of the leaves a large decrease in the amount of sucrose during the night with a corresponding increase in the amount of reducing sugars. In the top joints we note an accumulation of the reducing sugars; at this point, which is the region of active growth, we have the greatest disparity between sucrose and reducing sugars, the glucose ratio being 462. In the

* "Bulletin of the Louisiana Exp. Station," 12. p. 91

middle joints, where the process of growth is being suspended, we observe that the reducing sugars are in the minority in comparison with the sucrose, which is henceforth stored up in the pith as reserve material. The bottom joints illustrate the same facts, only to a greater degree. Regarding the other constituents, it will be seen that the percentage of ash, free and combined acid, nitrogenous bodies, and gums all decrease from the leaves downwards to the stalk.

Water consumed during Growth.—Maxwell,* in Hawaii, showed that during the whole period of the growth of the cane a supply of 147·8 parts of water is required for the formation of every part of dry substance.

Successive Composition of a Joint.—When we observe a single joint, from the moment at which it is formed to the period of ripeness, it will be seen that at first it contains no carbohydrates except starch. This constituent gradually becomes consumed, probably in the formation of the fibre, and during that time the joint enters into the stage in which the leaf, attached to it, begins to assimilate. Sugars collect; the joint begins to grow, the glucose and the fructose at first remain unchanged, while the sucrose becomes for the greater part inverted. A part of the invert sugar is conveyed to the younger joints in the top and another part is consumed during the growth for the formation of the fibre. At the moment that the joint is full-grown, it thus contains little sucrose and much glucose and fructose. These two sugars are consumed in the respiration process, but in the meantime new sucrose and also glucose and fructose are continuously supplied by the leaf, the former does not now become inverted any further, nor consumed by respiration, so that finally the joint contains chiefly sucrose and only a little reducing sugar.

At last the joint has reached the period in which the leaf to which it belongs has ceased to assimilate and is dying off. Now the quantity of sugars in the joint only increases by the portion which flows to it from the higher parts of the cane and thus is not fixed by the higher joints. As the distance of the joint from the assimilating leaves becomes larger, owing to the death of more and more leaves, the increase in the sugar content grows less until finally we reach the point where the influx of sugar into the joint is totally stopped, but owing to assimilation by the chlorophyll of the rind, the joint gains a little sucrose; however, this profit is more than counterbalanced by the loss of reducing sugar by respiration. This sugar decreases continually until in the riper joints it sinks to about 0·20 per cent. of the weight of the cane but very seldom disappears totally. In such regions where the cane is allowed to ripen fully, some joints contain no reducing sugars at all, but in practical working where some part of the cane stalk is always unripe or overripe, we are sure to find reducing sugar in every cane juice. It is evident that as soon as the point of maturity is passed, the reducing sugar content increases because of the inversion of sucrose.

* "Jl. Amer. Chem. Soc." 1898, 469.

Bottom Joints.—Not all the joints, however, behave in this way. First of all, the bottom joints, bearing the roots, are exceptions. The roots require reducing sugar for their growth, which is conveyed to their top for the building of the tissues and the formation of albuminoids, while part of it is deposited in the root-cap as starch. This sugar is withdrawn from the joint bearing the roots, where it is formed by inversion of the sucrose. During the development of these joints the increase in sucrose, though notable, is consequently not so considerable as in the higher joints which do not have to provide for the roots. As the former become older, all the sugar which enters into them is used for the formation of roots, so that the sugar content does not further increase.

Joints growing rapidly.—A second exception may be found when cane which in the beginning has grown slowly, and therefore already contains much sugar, suddenly shoots up into a rapid growth at a time when the sky is clouded and thus assimilation by the leaves is relatively insignificant. In this case sugar can be withdrawn from the lower tops to assist the growth of upper ones.

Place of Maximum Sucrose Content in Cane.—As a result of the above-mentioned facts the maximum sucrose content of a cane will for a long time be found in that joint which is on the same level with the soil, or just above the youngest root-bearing joints, and this maximum will be found in higher joints after every banking which covers new joints with moist earth and causes development of roots and consequently consumption of sucrose. At the age of six or seven months the maximum sucrose content will be found a little higher up the stalk, and as the time of maturity approaches will rise until rather near the top. This upward movement can still go on when older joints are already overripe and decrease in sucrose content.

Composition of the different Joints.—After having ascertained that canes from the same field were of similar composition, Went analysed canes from a given field joint by joint, at different periods, and tabulated the results obtained.

The Roman figures at the headings have the following signification :—

I. Number of the joints, starting at the bottom.
II. Length of the joints in centimetres.
III. Weight of the joints in grms.
IV. Per cent. sucrose in the joint.
V. Per cent. reducing sugar in the joint.
VI. Per cent. dry substance apart from sugars in the joint.
VII. Sucrose on 100 parts of the figure from VI.
VIII. Reducing sugar on 100 parts of the figure from VI.
IX. Sucrose on 100 juice.
X. Reducing sugar on 100 juice.
XI. Sum of the figures from IX. and X.

Young Cane.—1 Stalk, 6 Months of Age, 1—11 Bottom Part, 22—30 White Top.

I.	II.	III.	IV.	V.	VI.	VII.	VIII.	IX.	X.	XI.
1—10	8·0	24·5	6·7	0·26	25·5	26·3	1·0	9·4	0·4	9·8
11—15	9·0	36·0	9·7	0·4	16·0	60·6	2·5	11·6	0·5	12·1
16—18	10·0	39·5	9·0	1·0	13·0	69·2	7·7	10·4	1·2	11·6
19—20	8·5	35·5	6·9	1·9	12·8	53·9	14·8	7·9	2·2	10·1
21—30	13·5	39·0	2·5	2·3	12·3	20·3	18·7	2·8	2·6	5·4
Total ..	49·0	174·5	6·94	1·25	—	—	—	—	—	—

1 Stalk, 7 Months of Age, 1—16 Bottom Part, 34—40 White Top.

I.	II.	III.	IV.	V.	VI.	VII.	VIII.	IX.	X.	XI.
1—10	9·0	22·5	5·9	0·14	30·7	19·1	0·4	8·5	0·2	8·7
11—16	8·0	35·0	8·8	0·16	22·0	44·0	0·7	11·3	0·2	11·5
17—18	5·5	30·0	12·1	0·21	17·7	68·3	1·2	14·7	0·2	14·9
19—20	6·0	41·0	13·3	0·24	15·0	88·9	1·6	15·7	0·3	16·0
21—22	6·5	33·0	12·6	0·25	14·4	87·5	1·7	14·7	0·3	15·0
23—24	8·0	53·5	12·0	0·35	13·8	87·0	2·5	13·9	0·4	14·3
25—26	6·0	41·0	10·0	0·7	13·3	75·2	5·3	11·5	0·8	12·3
27—28	7·0	48·0	8·2	1·1	13·1	62·6	8·4	9·4	1·3	10·7
29—30	6·5	45·5	6·1	1·5	13·0	47·0	11·5	7·0	1·7	8·7
31—32	7·5	53·0	3·2	2·2	10·6	30·2	20·7	3·6	2·4	6·0
33	5·5	33·0	1·5	2·6	11·0	13·6	23·6	1·7	3·0	4·7
34—40	13·0	40·0	0·8	2·0	10·9	7·3	18·3	0·9	2·2	3·1
Total ..	87·5	475·5	7·86	1·02	—	—	—	—	—	—

1 Stalk, 8 Months ot Age, 1—18 Bottom Part, 34—41 White Top.

I.	II.	III.	IV.	V.	VI.	VII.	VIII.	IX.	X.	XI.
1—13	10·0	31·0	6·8	0·21	23·6	28·8	0·9	8·9	0·3	9·2
14—16	8·5	41·0	11·7	0·4	16·6	70·5	2·4	14·0	0·5	14·5
17	5·5	29·5	11·8	0·6	15·3	77·1	3·9	14·4	0·7	15·1
18	6·5	38·5	11·6	0·7	14·6	79·4	4·8	13·6	0·9	14·5
19	6·0	36·5	11·2	0·8	14·6	76·7	5·5	13·0	1·0	14·0
20	5·5	36·0	11·2	0·9	14·0	80·0	6·4	13·0	1·0	14·0
21—22	7·5	51·0	11·1	0·9	13·2	84·1	6·8	12·8	1·0	13·8
23	7·0	48·0	10·8	1·4	12·5	86·4	11·2	12·4	1·6	14·0
24	8·0	58·0	10·8	1·5	12·1	89·3	12·4	12·3	1·7	14·0
25	8·0	57·0	10·7	1·6	11·6	92·2	13·8	12·1	1·8	13·9
26	10·0	70·0	10·6	1·9	10·7	99·1	17·7	11·9	2·1	14·0
27	11·5	80·0	9·8	2·0	(11·0*)	(89·1)	(18·2)	(11·0)	(2·2)	(13·2)
28	10·5	77·0	9·2	2·1	11·2	83·0	18·7	10·4	2·4	12·8
29	11·0	72·5	7·9	2·2	11·8	67·0	18·7	9·0	2·4	11·4
30	11·0	72·5	6·9	2·3	10·6	65·1	21·7	7·7	2·5	10·2
31	12·0	74·5	5·5	2·4	11·1	49·5	21·6	6·2	2·7	8·9
32	12·0	71·5	3·8	2·6	10·6	35·8	24·5	4·3	2·9	7·2
33	10·5	59·0	2·3	2·6	9·9	23·2	26·4	2·6	2·9	5·5
34	10·0	42·0	1·3	2·5	10·2	12·7	24·5	1·5	2·8	4·3
35—41	17·5	40·0	0·8	1·8	9·7	8·2	18·6	0·9	2·0	2·9
Total ..	188·5	1085·5	8·17	1·75	—	—	—	—	—	—

The bottom part of the cane is woody and contains little sugar; the sugar content rises rapidly in the joints above ground and there possesses the maximum sucrose content of the whole cane. When, by the operation of banking, these joints are covered with moist earth, the roots sprout and withdraw sugar from them, so that the maximum sugar content is now to be found a little higher up. Then we find quite a series of joints with high sucrose content, and after these a decrease upwards, which is the more marked when the growth is vigorous. The maximum reducing sugar content is to be found in the upper joints, rising in a cane of slow growth and falling somewhat lower in the case of a

* The figures in brackets are not found by direct analysis but calculated by interpolation

rapid growth. Generally speaking, one may conclude that a joint contains its maximum reducing sugar content at the period of its most vigorous growth or a short time after. This reducing sugar has come from inversion of sucrose, which is clearly shown by the relative proportions of the three sugars—sucrose, glucose, fructose, which in slowly growing tops are as 4 : 2 : 1, and in rapidly growing ones as 0·8 : 1 : 1.

1 Stalk, 9 Months of Age, 1—20 Bottom Part, 39—47 White Top

I.	II.	III.	IV.	V.	VI.	VII.	VIII.	IX.	X.	XI.
1—13	12·0	50·0	9·0	0·21	19·2	46·8	1·1	11·1	0·3	11·4
14—15	9·5	49·0	12·6	0·20	16·9	74·5	1·2	15·2	0·2	15·4
16	7·0	35·0	13·0	0·32	16·6	78·3	1·9	15·6	0·4	16·0
17	7·5	40·0	13·1	0·4	16·9	77·5	3·0	15·8	0·5	16·3
18	7·0	37·0	13·5	0·5	14·1	95·7	3·5	15·6	0·6	16·2
19	7·5	45·5	13·5	0·6	13·4	100·7	4·5	15·6	0·7	16·3
20	6·5	42·0	13·5	0·6	13·8	97·8	4·3	15·6	0·7	16·3
21—22	9·5	59·0	13·5	0·5	14·0	97·4	3·6	15·6	0·6	16·2
23	7·5	52·5	13·5	0·8	12·3	109·0	6·5	15·4	0·9	16·3
24	9·0	66·5	13·5	1·1	11·7	115·4	9·4	15·3	1·2	16·5
25	10·0	76·0	13·5	1·2	10·6	127·3	11·3	15·1	1·3	16·4
26	10·0	76·5	13·5	1·2	10·7	126·2	11·2	15·1	1·3	16·4
27	11·5	86·5	13·7	1·2	10·5	130·5	11·4	15·3	1·3	16·6
28	11·0	83·0	13·4	1·3	10·1	132·7	12·9	14·9	1·4	16·3
29	12·0	88·0	13·0	1·4	10·3	126·2	13·6	14·5	1·6	16·1
30	10·5	78·5	12·4	1·5	10·6	117·0	14·1	13·9	1·7	15·6
31	11·0	75·5	11·9	1·5	12·2	97·5	12·3	13·5	1·7	15·2
32	12·0	77·0	11·3	1·5	11·9	95·0	12·6	12·8	1·7	14·5
33	11·5	71·0	11·4	1·7	10·9	104·6	15·6	12·8	1·9	14·7
34	9·5	61·0	9·8	1·8	11·8	83·0	15·3	11·1	2·0	13·2
35	9·5	56·5	8·9	1·9	12·2	72·8	15·6	10·1	2·2	12·3
36	11·0	64·0	7·0	2·1	11·8	59·3	17·8	7·9	2·4	10·3
37	11·0	63·0	5·0	2·4	10·9	45·9	22·0	5·6	2·7	8·3
38	11·0	57·5	3·4	2·4	10·1	33·7	23·8	3·8	2·7	6·5
39	10·0	43·5	1·9	2·2	10·3	18·4	21·4	2·1	2·4	4·5
40—47	18·0	42·0	0·8	1·9	9·8	8·2	19·4	0·9	2·1	3·0
Total ..	262·5	1576·0	11·01	1·31	—	—	—	—	—	—

Ripe Cane.—The distribution of the constituents in fully ripe canes is given by Went as follows:—

CANE PLANTED IN JULY AND ANALYSED THE NEXT YEAR.

25th April.				23rd May.				29th May.				30th June.				6th July.			
No. of the Joint.	Weight.	Sucrose.	Red. Sugar.	No. of the Joint.	Weight.	Sucrose.	Red. Sugar.	No. of the Joint.	Weight.	Sucrose.	Red. Sugar.	No. of the Joint.	Weight.	Sucrose.	Red. Sugar.	No. of the Joint.	Weight.	Sucrose.	Red. Sugar.
1	91·5	13·7	0·5	1	136·5	12·3	0·5	1	141·5	10·6	0·7	1	13·5	8·2	0·55	1	72·0	12·1	0·6
2	75·0	13·3	0·55	2	87·0	12·8	0·6	2	116·0	11·4	0·7	2	61·5	9·3	0·6	2	91·0	13·0	0·5
3	54·5	13·4	0·55	3	75·0	11·9	0·6	3	118·0	12·3	0·7	3	75·0	9·7	0·6	3	110·0	13·7	0·6
4	67·0	13·1	0·55	4	74·5	13·6	0·7	4	123·0	12·7	0·7	4	80·5	10·5	0·7	4	120·0	14·0	0·5
5	71·5	11·0	0·7	5	81·0	13·7	0·6	5	117·5	12·1	0·5	5	78·0	10·8	0·8	5	118·0	14·8	0·5
6	70·5	11·6	0·6	6	80·5	13·9	0·7	6	109·0	13·6	0·6	6	83·5	10·6	0·8	6	114·5	14·7	0·45
7	74·0	12·1	0·6	7	80·0	14·2	0·7	7	102·5	13·0	0·8	7	78·5	11·6	0·8	7	104·5	15·2	0·4
8	72·0	13·1	0·6	8	70·0	14·5	0·55	8	118·0	14·4	0·6	8	81·0	11·5	0·8	8	102·0	15·4	0·4
9	73·5	13·1	0·55	9	69·5	14·6	0·6	9	115·0	13·1	0·7	9	78·5	12·0	0·6	9	81·5	15·8	0·33
10	67·0	13·0	0·55	10	75·5	14·7	0·6	10	120·5	14·4	0·5	10	80·5	12·0	0·5	10	73·0	16·3	0·33
11	73·0	13·3	0·8	11	80·0	15·0	0·6	11	119·0	15·0	0·5	11	69·5	13·0	0·4	11	84·5	16·2	0·35
12	70·5	13·4	0·8	12	79·0	15·1	0·5	12	110·5	15·3	0·4	12	70·0	13·0	0·33	12	81·5	16·5	0·34
13	71·0	13·1	0·8	13	81·0	16·0	0·5	13	96·5	15·6	0·35	13	74·5	13·0	0·4	13	82·0	16·4	0·30
14	62·0	13·2	0·9	14	83·0	15·9	0·5	14	95·5	16·2	0·4	14	77·0	14·0	0·26	14	76·0	17·1	0·29
15	67·5	13·2	0·8	15	84·5	15·3	0·6	15	96·0	16·6	0·35	15	69·0	14·0	0·29	15	82·5	17·2	0·29
16	64·5	13·4	0·8	16	80·5	15·4	0·55	16	87·5	16·2	0·34	16	73·5	14·0	0·32	16	84·5	17·2	0·24
17	62·5	13·9	0·9	17	75·0	15·5	0·4	17	82·5	16·6	0·32	17	72·0	15·1	0·27	17	78·5	17·3	0·25
18	58·5	13·1	0·8	18	68·0	15·6	0·6	18	76·5	16·7	0·4	18	79·0	15·3	0·19	18	74·0	17·5	0·26
19	56·5	13·2	0·8	19	65·5	15·4	0·6	19	70·0	16·4	0·5	19	76·0	15·7	0·20	19	65·5	17·4	0·27
20	59·0	12·8	0·9	20	62·5	14·9	0·6	20	63·5	15·5	0·45	20	73·0	16·6	0·24	20	61·0	17·8	0·26
21	60·5	12·7	1·1	21	62·0	13·6	0·9	21	69·5	17·0	0·32	21	70·5	16·9	0·20	21	62·5	17·4	0·24
22	59·0	12·3	1·1	22	58·5	12·8	0·7	22	64·0	17·0	0·4	22	67·5	16·7	0·21	22	58·0	17·0	0·23
23	54·5	12·0	1·2	23	44·5	13·0	0·8	23	56·5	16·4	0·4	23	65·0	17·1	0·23	23	53·5	17·1	0·24
24	51·0	11·1	1·3	24	33·5	12·3	0·9	24	50·0	16·7	0·34	24	57·5	17·4	0·16	24	43·0	16·8	0·28
25	47·0	10·2	1·5	25	27·0	9·9	1·1	25	49·5	16·1	0·34	25	58·0	16·7	0·21	25 26	64·0	15·7	0·29
26	41·5	6·8	2·1	26	24·5	8·8	1·1	26	45·0	15·3	0·35	25	53·5	—	—	27–28	44·0	13·5	0·27
27	40·0	6·2	2·4	27	21·5	7·5	1·4	27	43·5	14·9	0·45	27	47·5	16·1	0·4	29–30	37·5	13·0	0·29
28	35·5	4·0	2·9	28–30	57·0	4·5	2·1	28	35·5	14·5	0·5	28	41·5	15·6	0·19	31–33	43·5	11·6	0·4
29–34	54·5	2·2	3·9	31–36	30·5	2·0	1·3	29–30	57·0	12·8	0·5	29–30	63·5	14·5	0·17	34–36	37·0	9·9	0·6
								31–32	39·0	10·3	0·8	31–32	48·0	13·5	0·32	37–45	43·5	5·7	0·8
	1805·0	12·00	0·96		1947·5	13·39	0·70	33–34	33·5	7·4	1·0	33–34	38·5	12·5	0·34				
								35–41	39·5	4·8	0·8	35–36	34 0	10·7	0·5		2243·0	15·31	0·38
												37–38	33·0	10·2	0·6				
									2661·0	14·67	0·55	39–40	34·5	8·0	1·0				
												41–46	39·5	5·0	1·1				
													2236·0	13·51	0·44				

Overripe Cane.—When cane is allowed to stand over in the fields a long time after it has reached its maximum sucrose content, the latter decreases irregularly. Although the normal distribution of sucrose in the cane is still faintly visible, the changes are unmistakable; we notice little sucrose in the bottom joints, a rapid rise in the higher joints, an irregular percentage in the

intermediate joints, and a maximum near the top. On the whole the figures are lower; only the top has increased its sucrose content. The reducing sugar has also increased to over 0·50 per cent. in many joints of the cane; the top joints, having ripened, do not contain so much. But, on the whole, sucrose has become inverted in many parts of the cane.

1.				2.				3.				4.			
No. of the joint.	Weight.	Sucrose.	Red. Sugar.	No. of the joint.	Weight.	Sucrose.	Red. Sugar.	No. of the joint.	Weight.	Sucrose.	Red. Sugar.	No. of the joint.	Weight.	Sucrose.	Red. Sugar.
1— 3	100·5	11·6	1·9	1—10	50·0	6·2	1·4	1—5	305·0	8·6	3·9	1—6	225·5	9·3	3·5
4	44·5	13·3	1·0	11—14	176·0	11·4	1·6	6	119·5	12·4	3·5	7	79·5	6·9	3·5
5	44·0	13·9	0·9	15—16	129·0	14·5	0·6	7	132·5	13·2	3·2	8	108·5	7·8	3·5
6	40·5	13·5	1·3	17—18	135·5	13·9	0·9	8	136·5	13·5	3·0	9	118·5	10·6	3·9
7	37·5	13·2	1·6	19—20	129·5	14·3	0·9	9	133·0	13·4	3·2	10	132·5	11·1	4·4
8— 9	62·0	13·6	1·6	21—23	141·5	14·8	0·7	10	132·0	13·7	3·0	11	134·0	12·5	3·8
10—11	49·0	12·6	1·6	24—26	121·5	14·9	0·9	11	122·0	14·4	3·0	12	133·5	11·8	3·5
12—13	40·0	11·0	1·6	27—29	101·5	14·3	0·7	12	112·5	14·6	2·7	13	116·5	11·6	4·8
14—16	53·0	12·8	1·6	30—32	122·5	14·2	0·7	13	107·0	15·5	2·3	14	123·5	12·5	4·4
17—19	68·0	13·1	1·5	33—35	134·5	13·8	0·9	14	99·5	15·6	2·3	15	129·0	12·8	3·9
20—21	64·0	14·4	1·4	36—38	139·0	14·1	0·7	15	98·5	15·6	2·3	16	128·0	12·9	4·6
22—23	72·0	14·6	1·6	39—41	122·5	15·7	0·55	16	99·0	15·3	2·4	17	131·5	13·3	4·1
24—25	52·0	14·1	1·5	42—44	84·5	16·2	0·20	17	95·5	14·8	2·5	18	135·5	12·8	3·9
26—27	44·0	14·0	1·4	45—47	96·0	16·6	0·21	18	88·5	15·2	2·7	19	122·0	12·2	4·0
28—31	81·5	14·8	1·3	48—52	63·0	13·3	0·24	19	83·5	15·2	2·5	20	117·0	12·5	3·9
32—34	59·5	14·6	1·1	53—65	53·5	11·7	0·27	20	81·5	15·5	2·5	21	111·0	12·9	3·7
35—38	81·0	13·2	1·3					21	79·0	15·9	2·3	22	108·5	12·2	3·7
39—41	36·5	9·4	1·6		1800·0	14·00	0·77	22	80·0	15·6	2·0	23	98·0	11·6	4·0
42—48	37·5	9·1	1·6					23	67·0	15·1	2·4	24	99·5	12·0	3·9
	1067·0	13·15	1·46					24—26	161·0	15·1	2·4	25	97·5	12·1	3·7
								27—29	162·5	15·0	2·5	26	85·0	12·1	3·9
								30—32	131·0	14·3	2·3	27	80·0	11·9	3·8
								33—35	77·5	12·6	2·7	28	77·5	11·2	3·9
								36—38	59·5	12·1	2·7	29	66·5	12·1	4·8
								39—49	189·5	10·5	3·5	30	67·5	12·6	3·9
												31—33	132·0	12·9	3·9
									2973·0	13·60	2·72	34—36	103·0	12·9	3·2
												37—39	69·0	12·4	3·0
												40—57	128·5	10·6	2·7
													3258·5	11·70	3·95

Canes attacked by Disease or Insect Pests.—Canes attacked by infectious diseases or by insects do not show such a regular distribution of the constituents as sound ones. Attacks of the cane borer (*Diatræa striatalis*) cause the sucrose content to fall, while that of reducing sugar remains unchanged; the same effect being brought about by attacks from woodpeckers (*Dendrocopus analis*). Attacks by the top borer (*Scirpophaga intacta*) do not seem to affect the sucrose content, but the joints attacked may become infected with the pineapple disease or black rot, causing an increase of reducing sugars.

We give here the composition of different joints of cane attacked in some places by Borers and the Red Smut disease (caused by *Colletotrichum falcatum*).

No. of the Joint.	I.			II.		
	Sucrose.	Reduc'g Sugar.	Observations.	Sucrose.	Reduc'g Sugar.	Observations.
1 2 3	13·4	1·74		15·6	1·70	
4 5	12·8	2·0		15·9	1·33	
6 7	12·2	1·74		15·4	1·67	
8 9	12·7	1·90		14·8	2·0	
10 11	12·6	1·67	Borer.	12·0	3·20	Red Smut.
12 13	7·2	1·90	Red Smut.	11·8	3·07	
14 15	11·0	2·67		14·4	2·67	
16 17	13·2	2·10		12·5	3·33	Red Smut.
18 19	12·3	2·50		16·2	2·10	Borer.
20 21	11·8	2·67		13·6	2·28	Red Smut.
22 23	11·1	2·67		14·2	2·40	
24 25 26	9·8	3·08	Top.			

Influence of Arrowing.—Arrowing is disadvantageous, inasmuch as, vegetation having ceased, the cane is more liable to die off than if it had not arrowed. Arrowing does not, however, affect the composition of the cane to any extent, as may be seen from the analyses on next page of two canes from the same stool, one of which had arrowed and the other not.

These results fully agree with those of some other experiments, in which some thousands of canes which had arrowed were cut from a field simultaneously with an equal number of canes which had not. These experiments were made on three estates, on two of which the white tops of the non-arrowing canes were cut for planting previous to grinding, while on the third the top was crushed along with the canes. Of every parcel 200 canes were measured and weighed, and then crushed in a triple crushing mill without maceration (see bottom page 72).

No. of the Joints.	Weight in grms.	Length in cm.	Per cent. Sucrose.	Per cent. Reducing Sugar.	Per cent. Dry Substance
I.—Arrowed.					
White top	105	9·7	2·4	3·49	17·8
Adult joints, 1 + 2 } from	67·5	11	8·2	2·84	22·6
,, ,, 3 + 4 } the	83·5	12·5	10·0	2·10	24·1
,, ,, 5 + 6 } top.	102	15	11·9	1·67	26·0
Not Arrowed.					
White top	90	8·7	4·2	3·07	17·8
Adult joints, 1 + 2 } from	101·5	12	5·6	3·75	18·8
,, ,, 3 + 4 } the	112	12	8·0	2·84	20·7
,, ,, 5 + 6 } top.	123	14	10·0	2·07	22·3
II.—Arrowed.					
From arrow to joint No. 5	31·7	16·5	2·4	2·13	14·3
Joints 6 + 7 ..	41·8	10	4·6	2·56	20·8
,, 8 + 9 ..	71·7	12	6·2	2·39	22·1
,, 10 + 11 ..	76·8	12	9·4	2·07	24·0
,, 12 + 13 ..	88·1	13	12·2	1·71	26·2
Not Arrowed.					
From vegetation point to Joint 5	18·8	8	2·5	2·25	16·25
Joints 6 + 7 ..	40·6	9	4·6	3·66	18·95
,, 8 + 9 ..	59·0	11	8·6	2·95	23·6
,, 10 + 11 ..	80·3	13	10·8	2·02	25·4
,, 12 + 13 ..	94·3	14·5	13	1·60	26·7

	I.		II.		III.	
	Arrowed.	Not Arrowed.	Arrowed.	Not Arrowed.	Arrowed.	Not Arrowed.
Length of the cane in metres	—	—	—	—	2·56 m.	2·60 m.
Weight of the cane with top	1·65 kg.	1·88 kg.	1·17 kg.	1·24 kg.	1·83 kg.	1·87 kg.
,, ,, without top	—	1·73 ,,	—	1·09 ,,	—	1·72 ,,
Polarization of the juice ..	19·3	18·8	19·43	17·93	19·11	19·61
Reducing sugar in the juice	0·42	0·44	—	—	0·57	0·49
Brix of the juice	21·3	20·2	22·0	20·4	21·09	22·0
Purity of the juice ..	90·6	92·8	88·3	87·9	90·57	89·14
Available sugar in the juice	17·3	17·4	16·86	15·46	17·13	17·22
Fibre on 100 cane ..	13·2	13·0	—	—	—	—

The canes which had arrowed were slightly lighter than the corresponding canes which had not, and from which no tops were cut, but heavier than those from which the tops had been removed. The analysis of the juice reveals no great differences, except in case II. where the available sugar in the canes which had arrowed is almost ½ per cent. more than in the canes without arrows.

Jansz* gives the following figures for juice obtained from 156 canes which had arrowed and 163 which had not :—

Arrowed canes ..	19·3 Brix	17·42 sucrose	90·25 purity.
Non-arrowed canes	19·2 ,,	17·40 ,,	90·62 ,,

No difference could therefore be detected.

All these experiments have been made with Black Java cane and for that variety they give fully corresponding results. With other varieties, however, arrowing appears to have a worse effect, as is shown in the following experiments by Van Vloten.†

He cut samples, each consisting of 50 stalks of each of the varieties Nos. 36, 100 and 247, one of cane which had flowered and one which had not. The former were shorter, lighter and yielded less sugar per 100 cane, as is shown in the table underneath.

Description of the sample.	Weight kg.	Length metres	Kg. per metre	Constitution of the juice					Yield of Sugar on 100 Cane
				Brix.	Sucrose	Quot.	Red. Sugar	Gluc. Ratio	
No. 36. Arrowed	0·930	2·26	0·394	17·40	15·42	88·62	0·40	2·59	10·76
,, 36. Non-arrowed	1·386	3·06	0·454	18·40	16·38	89·02	0·29	1·77	11·49
,, 247. Arrowed	1·421	2·09	0·680	18·70	16·80	89·84	0·60	3·57	11·92
,, 247. Non-arrowed	1·816	2·51	0·742	19·40	17·60	90·72	0·47	2·67	12·64
,, 100. Arrowed	1·272	1·86	0·684	21·30	20·15	94·60	0·22	1·09	15·20
,, 100. Non-arrowed	1·599	2·50	0·639	21·60	20·55	95·14	0·21	1·02	15·60

From these figures he derives the conclusion that every 10 per cent. of arrowing canes in a field diminishes the sugar output by 400 pounds per acre.

Fallen Cane.—When cane falls, the sucrose and the quotient of purity decrease considerably as is shown in the following analyses, made at different periods, of a fallen and a standing cane, cut from the same stool in a field of healthy Black Java cane. The joints are numbered from above downwards, after the white tops had been removed.

* "Archief voor de Java Suikerindustrie," 1896 636.
† *Ibid.*, 1910, 280.

24TH MARCH.

Fallen Cane						Standing Cane.					
No. of the Joints.	Length in Centimetres.	Weight in Grms.	Sucrose.	Reducing Sugar.	Dry Substance.	No. of the Joints.	Length in Centimetres.	Weight in Grms.	Sucrose.	Reducing Sugar.	Dry Substance.
1—2	10	40·4	1·0	2·56	10·6	1—2	9	52·5	3·2	1·83	14·7
3—4	12	77·7	1·6	2·74	12·2	3—4	14	106·5	4·1	2·32	15·7
5	6	56·8	1·6	3·19	13·3	5—6	18·5	144·5	5·8	2·32	18·2
6	9	77·5	2·3	3·49	15·2	7—8	19	161·5	6·9	2·19	19·2
7	10	90·8	3·2	3·49	15·9	9—10	19·5	169·5	7·8	2·13	19·6
8	11	100·3	3·9	2·95	16·9	11—12	18·5	161·2	8·9	2·02	20·6
9	10	103·0	4·5	2·95	15·8	13—14	18	145·5	9·1	2·02	21·8
10	9	104·1	4·5	2·95	16·7	15—16	17	139·7	9·6	1·47	21·8
11	9	103·6	5·1	2·74	17·1	17—18	16	141·5	9·8	1·47	21·8
12	9	103·2	5·4	2·64	16·7	19—20	18	155·2	10·0	1·53	22·4
13—14	18	207·0	6	2·39	16·5	21—22	19	163·2	10·2	1·11	22·8
15—16	19	207·4	7	2·25	17·5	23—24	16	154·7	10·2	1·28	23·3
17—18	16	177·0	7·7	2·25	18·5	Rest	61	600			
19—20	18	196·0	7·9	1·74	18·3						
21—22	18·5	191·3	7·9	1·74	19·3		205·5	2346			
23—24	14·5	156·8	8·4	1·28	21·8						
25	17	173·3	8·1	1·67	19·0						
26	8	78·5	8·5	1·67	20·3						
27	7	65·8	8·7	1·53	20·5						
28	6·5	60·3	8·4	1·47	20·5						
29	9	80·0	8·4	1·42	21·0						
30	11	121·8	8·4	1·27	21·3						
	225·0	2573									

The fallen canes are heavier and longer than the standing ones from the same stools. On the other hand, their sucrose and fibre contents are inferior, and their water and reducing sugar contents higher. These differences point to unripeness in the fallen canes, which grow more rapidly, but ripen more slowly.

H. Atherton Lee and F. M. Clara* measured the losses in actual sugar occurring during the interval between the cutting of cane tops for seed and the actual milling of the cane. The result of this investigation was that the complete harvesting of the cane and the milling on the same day showed a polarization of 16·74 per cent., with a purity of 89·1. Allowing the same cane to stand one day after topping and then cutting, the polarization was found

* "Louisiana Planter," 1923, I, 182.

19TH APRIL.

FALLEN CANE.						STANDING CANE.					
No. of the Joints.	Length in cm.	Weight in grms.	Sucrose.	Reducing Sugars.	Dry Substance.	No. of the Joints.	Length in cm.	Weight in grms.	Sucrose.	Reducing Sugars.	Dry Substance.
1—3	11	47·5	1·5	2·56	12·2	1—3	10	36·5	1·7	2·02	14·5
4—5	8·5	60·1	1·6	3·33	13·6	4—5	8	46·7	2·3	2·84	14·5
6—7	11·5	111·0	1·8	3·66	13·9	6—7	10	75·7	3·4	2·74	15·8
8—9	14	137·3	3·9	3·66	15·9	8—9	11	91·8	5·3	2·47	17·8
10—11	11·5	108·8	5·3	3·33	18·2	10—11	14·5	137·3	7·5	2·32	18·5
12	9	75·1	8·2	2·74	20·3	12—13	20	185·5	8·1	2·02	18·9
13	12	95·5	8·5	2·39	22·0	14—15	19	190·0	8·5	2·02	19·7
14	11	95·0	8·9	2·25	21·3	16—17	18·5	179·3	9·0	1·74	20·2
15	10·5	104·1	8·7	2·25	20·2	18—19	18	178·6	9·8	1·67	23·1
16	12	123·5	9	2·25	20·0	20—21	17·5	180·6	10·4	1·37	23·0
17	13	126·6	9·1	2·19	20·3	22—23	19·0	185·7	10·5	1·37	23·3
18	12	120	9·1	1·83	19·5	24—25	19·5	185·7	11·2	1·12	24·5
19	11	114·1	9·4	1·60	19·5	26—27	19·0	170·5	11·2	1·28	23·3
20	12	114·3	9·6	1·53	20·6	28—29	23	212·0	11·8	1·19	26·2
21	11·5	105·0	9·6	1·42	20·4	30—31	20	210·3	12·6	1·06	25·5
22	12·5	106·6	8·8	1·47	20·1	32	10	109·5	13	0·98	25·0
23	11·5	89·5	8·1	1·47	20·1						
24—25	23	208	9·2	1·83	20·8		254·5	2375·7			
26—27	21	295·5	9·8	1·67	19·8						
28—29	24	214	9·8	1·60	19·7						
30—31	25	242·3	10·1	1·42	20·2						
32	12	122·0	10·2	1·37	21·4						
	294·5	2715·8									

FALLEN CANE.						STANDING CANE.					
1—4	20	108	2·2	3·33	14·0	1—4	9	49·8	3·1	3·49	18·2
5—7	15	121	4·2	3·84	16·8	5—7	8	53·6	5·8	2·95	23·0
8—10	16	134·5	7·0	3·07	20·0	8—10	7	51·5	8·4	1·53	22·3
11—13	12	98	8·45	2·47	20·7	11—13	10	73·7	12·0	0·96	25·5
14—16	12	98·5	10·1	1·87	21·2	14—16	12	98·5	13·9	0·69	26·0
17—18	12	123·5	11·7	1·67	21·6	17—18	10·5	91·5	15·2	0·64	26·8
19—20	12	109	12·0	1·28	22·9	19—20	12	112·0	15·1	0·64	26·3
21—22	17	153·5	12·3	1·10	22·5	21—22	18	174·2	15·1	0·64	26·7
23	12	102·2	12·1	1·10	23·5	23—24	19	196·5	15·0	0·76	25·8
24	11	101·1	12·0	1·10	21·6	25—26	18	178·7	15·2	0·76	26·2
25	11·5	115·8	12·0	1·10	22·3	27—28	19	202·2	15·7	0·88	26·3
26	8·5	87·3	12·3	0·96	21·3	29—30	18	187·0	15·0	0·76	26·0
27—28	16	174·2	12·0	0·93	21·8	31—32	15	141·0	15·0	0·76	26·9
29—30	16	160·7	12·0	0·83	22·9	33—34	16	151·3	15·6	0·69	27·3
31—32	14	133·1	12·4	0·76	21·6	Rest	81	85·9			
33—34	15	122·6	13·2	0·85	23·5						
Rest	93	89·7					272·5	2621·3			
	314·0	2840									

to be 15·8 per cent., with a purity of 84·1 ; cane cut three days after topping showed a polarization of 15·28 per cent. and a purity of 80·4 ; while the figures for the fourth day were 13·91 and 73·2 respectively.

Deterioration of Cut Canes.—Canes which are kept for a long time, either in the field or under shelter, after being cut, deteriorate very rapidly ; the sucrose becomes partially inverted and the reducing sugar content consequently increases, while the weight of the cane diminishes owing to the evaporation of water. Contrary to the view generally held, the acidity of the juice does not augment during the first few days after cutting.

Müller von Czernicky* reports experiments in which a few tons of canes were completely dug out and cleaned from adhering earth, just as is the custom in Java when harvesting cane. These canes were tied into 12 bundles of 24 each which were preserved indoors, and of which two bundles were analysed each day during six consecutive days.

Days after the Cutting.	0	1	2	3	4	5
Brix	21·2	21·6	21·7	21·8	22·3	22·5
Polarization	19·93	20·20	20·25	19·69	19·07	18·45
Quotient of purity ..	94·0	93·5	93·3	90·3	85·5	82·0
Reducing sugar	0·3	0·3	0·4	0·8	1·6	2·1
Available sugar	15·7	15·6	15·3	14·5	13·0	12·0
Loss of weight in per cent.	0	1·1	2·1	3·0	3·9	4·7

The percentage of available sugar was calculated by assuming that 100 parts of cane contain 84 parts of juice of a composition equal to that obtained by the laboratory mill ; and, further, that the loss in weight is only due to evaporation of water, therefore the degrees Brix are the same for all the samples, and these are assumed to contain the same juice content. The available sugar is then calculated as being represented by the product of sucrose and quotient of purity divided by 100.

Another parcel of canes which had lain in the field for 24 hours exposed to sunshine and afterwards kept indoors, gave the following figures :—

* "Archief voor de Java Suikerindustrie," 1900, 610.

Days after removal from field.	0	1	2	3	4	5
Degrees Brix	21·5	22·1	22·4	22·7	23·0	23·3
Polarization	20·35	20·82	19·36	18·17	17·77	17·29
Quotient of purity ..	94·6	94·2	86·4	80·0	77·2	74·2
Reducing sugar	0·2	0·3	1·0	1·9	2·3	—
Available sugar	16·2	16·0	13·5	11·6	10·8	9·9
Loss of weight per cent. ..	0	2·1	3·3	4·3	5·4	6·6

Savornin Lohman* obtained similar results when analysing cane which had been kept indoors for 24 hours, as compared with cane that had been left in the field during that time, and with cane just brought in.

Constituents.	Crushed immediately.	Kept in the field for 24 hours.	Kept indoors for 24 hours.
Brix	17·13	16·48	16·89
Polarization ..	14·63	13·54	14·17
Purity	85·45	82·13	83·90
Available sugar ..	13·64	12·36	13·08
Acidity	0·190	0·202	0·200

On calculating the available sugar to the same figures for degrees Brix, we find 13·64 per cent. for the canes crushed immediately, 12·88 per cent. for those kept out for 24 hours, and 13·27 per cent. for those kept indoors during that time, which means a loss per 24 hours on 100 parts of juice of respectively 0·76 and 0·37, or on 100 parts of original available sugar of 5·57 and 2·71 per cent.

At the meetings of the Association of Advisers to the Java Sugar Industry of 1922 and 1923, the problem was discussed whether the new varieties have a similar degree of deterioration after being cut as the canes which had been the subject of the afore-mentioned investigations.

The very important series of figures and data, brought forward on that occasion, showed that the new varieties planted nowadays in Java behave in pretty much the same way as their predecessors.

The above figures were found in the tropics (Java), but it appears that in cooler climates deterioration is not so rapid, as is shown in H. Pellet's† table of Egyptian cane given below. H. Pellet allowed cane to lie for a long time and analysed samples from the heap at different dates during the month of January, 1896.

* "Archief voor de Java Suikerindustrie," 1900, 1175.
† "Etudes sur la Canne à Sucre," page 40.

Days kept.	Loss of Weight in per cent.	Spec. Gr. of the Juice.	Sucrose in 100 Juice.	Reducing Sugars on 100 Sucrose.	Quotient of Purity.
0	0	1·0705	13·5	3·3	88·5
4	2·5	1·0729	13·4	4·17	89·2
7	4·3	1·0715	13·6	3·37	88·1
11	10·0	1·072	14·2	3·80	89·6
15	8·7	1·074	13·7	4·80	87·0
20	9·0	1·076	14·3	3·10	87·8
23	12·5	1·0765	13·8	3·70	86·5
25	15·0	1·081	14·7	3·75	87·1
27	18·0	1·084	14·9	4·50	85·3

In the comparatively hotter months of December and February the deterioration was much greater, without, however, approaching that shown in the Java instances.

Preservation of Cut Canes.—In conjunction with Went the author made experiments* as to the practicability of preserving cut canes for a long time without their undergoing too great a change. They came to the conclusion that sound canes, kept out of contact with each other and covered with moist sheets, remain unchanged for several days. But it should be stated that these experiments were only made in the laboratory, so that the results are not applicable to ordinary conditions where canes are stacked and stored in a much rougher way.

A stool of Black Java cane, consisting of 10 stalks, was cut, two of the canes were inspected under the microscope and afterwards crushed; half of the remaining eight canes (portion A) were placed in a dry spot in the sunshine, while the other half (portion B) were covered with moist sheets. After 1, 2, 3, and 5 days respectively, from every one of the two portions one stalk was inspected microscopically, then crushed and the juice analysed. The microscopical inspection showed that immediately after the cutting of the cane the parenchyma cells contained much sucrose and little reducing sugar. A day later a small increase in the reducing sugar content was to be observed in cane A but not in B. Even after two or three days the B canes remained unchanged, while in those from A a good deal of reducing sugar could be detected, chiefly in those cells which were nearest to the periphery of the cane. At the end of the fifth day this decomposition of sucrose had also reached the centre of the stalk, so that in the canes from A all the parenchyma cells contained considerable quantities of reducing sugar; in B, on the contrary, only

* "Archief voor de Java Suikerindustrie," 1894, 249.

a very slight difference from the original state could be observed. We could only detect an accumulation of reducing sugar in those cells of the B canes which were situated close to the buds and to the sprouting rootlets.

The analyses of the juice confirmed the microscopical tests.

Constituents.	Immediately after.		After 1 day.		After 2 days.		After 3 days.		After 5 days.	
	1	2	A	B	A	B	A	B	A	B
Sucrose	20·7	21·8	21·4	21·0	21·0	21·0	20·4	20·0	19·8	20·0
Reducing Sugar	0·13	0·12	0·24	0·22	0·69	0·35	1·83	0·38	2·21	0·68
Purity	93·7	92·0	94·6	93·3	92·8	93·1	86·6	94·8	84·8	92·2

Windrowing.—As long as the cells remain alive, the composition of the juice does not appreciably alter, but as soon as they die the reducing sugars increase at the cost of the sucrose. The chief cause of the death of the cells is the drying up of the cane. When we succeed in preventing the cut cane from drying (a very difficult problem in tropical countries), or from fermenting, it may be kept for a long time before the juice deteriorates. In cooler climates the cane may be kept for a long time after cutting by the practice known in Louisiana as "windrowing." When there is cane in the field at the time that frost is expected, the standing canes are cut, thrown between rows, and covered with cane trash to keep off the frost until they can be conveyed to the mill to be crushed. Stubbs* showed that, in windrowing, much sucrose is lost by inversion, so that the practice is only resorted to in order to avoid still larger losses by splitting frost.

The following figures are given by Stubbs: cane cut 7th November, windrowed till 13th December or during 35 days, maximum temperature 25·5° C., minimum 3·3° C., average 15° C., rainfall 2·6 inches.

	Brix.	Sucrose.	Reducing Sugar.	Purity.	Glucose Quotient.	Available Sugar.
Analysis on 7th November	14·09	10·42	1·94	73·95	18·6	7·70
,, ,, 13th December	14·23	9·02	2·99	64·02	33·1	5·77

* "Bulletin 37, Louisiana Exp. Station," page 1294.

Cane cut November 22nd and windrowed till January 2nd, or during 42 days, maximum temperature 25° C., minimum — 6° C., average 11·1° C., rainfall 4 inches.

	Brix.	Sucrose.	Reducing Sugar.	Purity.	Glucose Quotient.	Available Sugar.
Analysis on 22nd November	14·22	11·33	1·60	80·0	14·1	9·06
,, ,, 2nd January	14·22	10·40	2·38	73·1	22·9	7·60

Browne and Blouin* ascribe this gradual falling off in sucrose content of cane which had been windrowed for any length of time to spontaneous inversion by enzymes from the upper end of the cane, for if the green tops are removed at the time of the cutting the loss of sucrose is much less. This can easily be seen in the following series of experiments which were carried out at Audubon Park in 1903. Several lots of canes were windrowed, one half of each lot having the tops removed. In all other respects the conditions of the experiments were alike. At the end of a month all tops were removed, the stalks from the different lots ground and the juices analysed with the following results :—

		Brix.	Sucrose.	Reducing Sugar.
Lot 1.	Windrowed with tops cut ..	16·1	13·3	1·25
	,, ,, ,, on ..	15·9	12·1	1·85
Lot 2.	,, ,, ,, cut ..	15·8	12·8	1·22
	,, ,, ,, on ..	15·4	11·5	1·53
Lot 3.	,, ,, ,, cut ..	16·3	13·5	1·25
	,, ,, ,, on ..	16·1	12·6	1·92
Lot 4.	,, ,, ,, cut ..	16·2	13·7	1·00
	,, ,, ,, on ..	15·8	11·8	1·85
Lot 5.	,, ,, ,, cut ..	15·9	12·8	1·39
	,, ,, ,, on ..	15·0	10·7	2·17

Effect of Frost on Cane.—The injurious effect of frost on sugar cane is illustrated by the following examples reported by Stubbs.† On the 27th December, the canes were exposed to a sharp frost, and on the 28th December

* "Bulletin 91, Louisiana Exp. Station," page 18.
† "Bulletin 37, Louisiana Exp. Station," 1224.

a few canes were cut and analysed, the remainder being partly windrowed, and the balance left standing. The temperature was —7·2° C. on the 27th December, and it kept on freezing till 2nd January. The cane was cut on 15th January. Thus after 20 days (maximum temperature 25° C., average 8·4° C., rainfall 8 inches) the analyses of the juices were as follows :—

	Brix.	Sucrose.	Reducing Sugar.	Purity.	Available Sugar.
Analysis on 27th December	14·91	11·85	1·42	79·5	9·42
15th January (windrowed)	13·60	9·09	1·65	66·9	6·62
15th Jan. (left standing) ..	12·84	8·66	1·29	67·5	5·84

Stubbs expresses the view that the frost killed the cane cells, causing an intermixture of the different kinds of sap, which then decomposed.

Effect of Fire.—The deterioration of cane occasioned by the death of the cells is still more rapid after a fire. When a canefield is burnt, only the dry trash is actually consumed ; but the stalk itself is overheated, with the result that the cells die and the cane perishes. During the first days after a fire only a trifling change in the composition of the juice is noticeable, so that if the burnt canes are milled at once the quality of the juice is not much inferior to that of unburnt canes. The real drawback is not the actual loss of sucrose, but the necessity of crushing the burnt canes at once, which means a large pecuniary loss if the canes are not yet ripe. In the examples given below, the

	Cane variety.	Brix.	Polarization.	Purity.
1st day ..	Black Java	18·3	14·75	80·6
2nd ,, ..		17·8	14·15	79·5
3rd ,, ..		17·3	13·68	79·1
1st ,, ..	Black Java	21·4	19·91	87·7
3rd ,, ..		21·3	18·67	77·3
1st ,, ..	Seedling 247	14·4	10·95	76·0
2nd ,, ..		13·2	9·80	74·2
3rd ,, ..		12·6	9·18	72·8
1st ,, ..	Seedling 71	17·3	14·04	81·1
2nd ,, ..		17·2	13·96	81·2
3rd ,, ..		16·0	12·32	77·0

sucrose and the quotient of purity are very low, but these are only indirect consequences of fire, because the planter was compelled to bring the cane to the mill much earlier, and in a less ripe condition, than would otherwise have been the case.

Usually the cane can be crushed soon enough, and then the damage is not considerable, as is shown in the Table preceding.

But if circumstances prevent the canes being crushed at once, the deterioration goes on rapidly and uninterruptedly. In one case a field was planted with Seedling No. 247 and, on the 30th March, a plot of six acres caught fire and was destroyed. The mill was out of order just then, and the cane could not be harvested, but in order to investigate the rapidity of the deterioration, a few canes were cut from time to time, divided into pieces and analysed. Every one of the stalks was cut into 10 pieces, and the Nos. 1, 2, 3, etc., to 10 of each cane combined, crushed and analysed. The cane was harvested on 10th and 11th April, and crushed on the 12th April, while on that same date a portion of these canes was crushed and analysed in the laboratory.

Date.	Sample taken.	Brix.									
		Top	2	3	4	5	6	7	8	9	10
March 21st	Previous to the fire	11·5	14·47	15·48	15·68	15·78	15·98	16·03	16·18	16·58	17·13
,, 31st	Burnt cane	12·2	15·2	15·9	16·09	15·94	15·84	15·59	15·59	15·75	16·35
,, 31st	Unburnt, from the same field	11·85	14·67	15·57	15·82	16·17	16·17	16·12	16·37	16·72	17·47
April 3rd ..	Burnt cane	13·04	15·10	15·65	15·64	15·39	14·74	13·99	13·34	12·8	14·01
,, 5th	,, ,, ...	12·24	14·64	15·25	15·04	14·54	13·84	13·09	12·53	12·23	13·74
,, 7th ..	,, ,, ...	14·23	15·62	16·20	15·75	15·40	13·69	13·13	11·67	10·77	11·97
,, 9th ..	,, ,, ...	11·86	14·49	14·80	14·39	13·59	12·84	12·09	11·38	11·28	12·04

Date	Sample taken.	Polarization.									
		Top	2	3	4	5	6	7	8	9	10
March 21st	Previous to the fire	6·13	10·49	11·80	12·10	12·26	12·59	12·70	13·0	13·63	14·47
,, 31st	Burnt cane	7·45	11·57	12·53	12·78	12·72	12·67	12·55	12·61	13·96	13·06
,, 31st	Unburnt, from the same field	6·82	10·82	12·28	12·75	13·21	13·21	13·18	13·72	14·5	15·57
April 3rd ..	Burnt cane	8·28	11·50	12·48	12·56	12·42	11·88	11·28	10·82	10·58	12·08
,, 5th ..	,, ,, ...	7·63	10·98	12·15	12·15	11·76	11·15	10·42	10·07	10·01	11·68
,, 7th ..	,, ,, ...	8·34	11·43	12·31	11·91	11·66	10·70	9·68	8·55	7·97	9·42
,, 9th ..	,, ,, ...	7·13	10·44	11·09	10·73	9·91	9·27	8·64	7·99	8·37	9·48

Date.	Sample Taken.	Purity.										Reducing Sugar.				
		Top	2	3	4	5	6	7	8	9	10	Top	2	3	9	10
March 21st	Previous to the fire	53·3	72·5	76·2	77·2	77·7	78·8	79·2	80·3	82·2	81·6	3·21	2·60	2·24	1·98	1·60
,, 31st	Burnt cane	60·8	76·2	78·8	79·4	79·8	80·0	80·5	80·9	82·9	85·4	2·40	1·80	1·54	1·18	1·04
,, 31st	Unburnt, from the same field	52·5	73·7	78·8	80·6	81·7	81·7	81·7	83·8	86·7	89·1	2·64	2·0	1·90	1·29	0·94
April 3rd ..	Burnt cane	63·5	76·16	79·54	80·3	80·7	80·6	80·6	81·6	82·6	86·2	2·38	1·62	1·60	1·19	1·08
,, 5th ..	,, ,, ...	62·3	75·0	79·5	80·78	80·88	80·56	79·6	80·37	81·85	85·01	2·62	2·0	1·72	1·17	0·96
,, 7th ..	,, ,, ...	58·8	73·17	76·0	75·6	75·7	78·16	73·7	73·26	74·0	78·7	2·91	2·44	2·12	1·60	1·27
,, 9th ..	,, ,, ...	60·1	72·05	74·9	74·56	72·9	72·2	71·46	70·2	74·2	78·7	2·79	2·10	2·0	1·56	1·30

The mill juice from those canes cut on two consecutive days had the following composition :—

	Brix.	Pol.	Purity.
Cut 10th April ..	9·86	4·73	47·78
Cut 11th April ..	9·62	4·86	50·62

while the sample crushed in the laboratory mill gave 10·7, 6·79, 63·4.

In another instance where cane was left in the field for a long time after a fire, from April 9th until May 4th, the average weight of the canes diminished from 1·31 kg. to 1·05 ; and the composition of the juice, which was originally Brix 12·75, sucrose 8·60, reducing sugar 2·63, and purity 67·45, became Brix 8·78, sucrose 4·74, reducing sugar 3·29, and purity 53·99.

Influence of Climate on Sucrose Content.—In sub-tropical countries temperature has a very marked influence on the sucrose content of the cane. This has been shown for Louisiana by Browne and Blouin, in the results of years 1903 and 1904, which were very dissimilar as regards weather conditions.

1903.

	August 1.	September 1.	October 1.	November 1.	November 15.
Sucrose ..	2·70	5·97	11·27	13·60	15·86
Reducing Sugar ..	3·80	3·68	2·51	1·02	0·63
Purity ..	36·00	57·02	76·72	87·85	92·10

1904.

Sucrose ..	2·35	5·13	8·04	9·13	12·00
Reducing Sugar ..	4·04	3·75	3·55	2·82	1·66
Purity ..	32·28	52·35	66·61	71·55	80·53

The weather conditions for the two years in question during the growing season were as follows :—

	July.	August.	September	October.	November	December
Av. daily temp., F., 1903	82·80	82·10	77·00	67·90	58·00	49·00
,, ,, F., 1904	80·90	84·10	84·20	71·20	64·30	58·40
Rainfall, inches, 1903..	5·55	5·98	1·27	0·36	0·23	3·89
,, ,, 1904..	6·47	5·75	3·24	0·75	1·50	3·02

The results for the two years show but little variation up to the middle of September. After this date the sucrose content of the 1903 canes increased considerably, and this increase continued until the end of the season. The average daily temperature and rainfall for the two years were also about the same during June, July, and August; for the remaining months of the year, however, the conditions were very unlike September, October, November, and December. 1903 showed far lower daily averages in temperature than the corresponding months of 1904, while it also showed a deficiency of rainfall. These conditions for 1903 were very adverse to the growth of the cane, yet hastened the ripening to an extent rarely attained in Louisiana. On the other hand, the unusually warm weather of the fall of 1904, together with favouring rains, promoted the growth of canes even into December, but retarded the ripening.

In tropical countries where the average temperature does not vary much in different years, the sucrose content of the cane is not so much influenced by the temperature as by the rainfall. Kobus* illustrated this in the table below,

Year.	Sugar extracted from 100 Cane.	Sucrose in 100 Cane.	May—October (Ripening Time).		October, November, December (Soon after Planting).	
			Average Monthly Rainfall.	In per 1000 of the Year's Total.	Rainfall in Three Months	In per 1000 of the Year's Total.
1894	10·36		2·16	127	23·62	368
1895	9·79		4·66	249	18·74	340
1896	10·55		0·74	51	17·60	310
1897	10·06		1·38	87	19·37	339
1898	10·21		3·46	185	27·95	417
1899	10·94	13·99	2·28	160	18·23	326
1900	9·57	12·26	4·56	263	17·72	288
1901	10·16	12·68	3·42	176	19·10	310
1902	10·77	13·43	1·10	67	11·85	96
1903	10·03	12·40	2·48	131	25·78	418
1904	10·74	13·04	3·07	174	20·59	347
1905	10·37	12·66	2·12	129	13·27	233
1906	10·04	12·44	3·11	184	26·61	396

* "Proceedings of the 8th Sugar Congress, Sourabaya," 1907, 40.

in which he put together the inches of rainfall during the early growth of the cane and during the ripening time, and the average sucrose content of the cane in Java, and the yield of sugar on 100 cane. As cane is planted there in June-September and crushed in May-October of the following year, the figures for October-December, 1904, for instance, apply to the cane crushed in May-October, 1905.

The figures for rainfall during the ripening time are highest in 1895 and 1900, and they correspond with the lowest extraction, *i.e.*, low sucrose content and low purity; while, on the other hand, the driest ripening seasons, 1896 and 1902, correspond with high extraction. The rainfall during the ripening period is, however, not the only factor, as the rather rainy years, 1899 and 1904, also showed high extraction. Another factor is the meteorological state during the early period of growth, as cane can be fully ripe in May if an early growth is favoured by a plentiful rainfall in October. Here we find the highest figures for 1898, 1903, and 1906, and, in accordance with these, high extraction in the corresponding years, 1899, 1904, and 1907, notwithstanding excessive rainfalls during the ripening periods in those years.

Geerts has studied* very closely the factors determining the product of a canefield, both as regards the weight and the sugar content of the crop.

The character of the soil, the time of planting, the meteorological circumstances, in short, anything apt to influence the growth and the development of the cane also exerts an influence on its sugar content. Cane ripens slowly in a moist soil, where it may continue its growth during a long period; on soils which are drying up in the dry season it will ripen much more quickly. On plantations situated considerably above sea-level the low night temperatures stop the growth and promote ripening. If the circumstances of growth and development have been very favourable, the storage accommodation for sugars in the canestalk is very spaciously built; a great deal of sugar may therefore be accumulated in the cells, without the liquid in the cell becoming as concentrated a solution as is the case in cane grown under less favourable conditions where less spacious parenchyma cells are present.

Cane having had an abnormal growth owing to drought often ripens very rapidly; the ripening period proves very short, the cane soon becomes overripe and loses in sugar content.

Fertilization with nitrogenous manure influences the growth of the cane, and therefore likewise its sugar content. This influence does not act in a direct manner but rather in an indirect one, since such fertilization may retard or prolong the vegetative period.

* "Archief voor de Java Suikerindustrie," 1923, 1171.

If the fertilizer has been applied late, or if a long-acting manure, such as filter-press mud or dung has been used, the growth does not cease and ripening will be retarded. A very heavy dressing with fertilizers decreases both the weight and the sugar content of the cane.

It has been proved that the sugar content of the cane depends on a great many factors, which are steadily in mutual intercourse. A dry harvesting time is favourable for the ripening on an estate having moist and low-lying land, but unfavourable for estates on a loamy, easily cracking soil, where the rootlets may be torn asunder, if the cane is not cut before the land has dried up.

Increase of Sucrose in Cane during Ripening.—The following summary of hundreds of analyses of cane, which were made with a view to ascertaining the period of maturity, shows how the sucrose content and the purity increase as maturity proceeds :—

I.

Date of Analysis.	Brix.	Sucrose.	Purity.	Available Sugar.	Date of Planting.
27th March ..	19·4	17·81	91·9	13·09	15th May
10th April ..	19·7	18·34	93·1	13·67	*idem*
20th April ..	20·1	18·77	93·3	14·01	*idem*
30th April ..	20·1	18·75	93·2	13·98	*idem*

II.

Date of Analysis.	Brix.	Sucrose.	Purity.	Available Sugar.	Date of Planting.
27th April ..	19·5	17·89	91·8	13·14	28th May
17th May ..	19·9	18·23	91·6	13·36	*idem*
27th May ..	19·9	18·55	93·2	13·83	*idem*
8th June ..	19·9	18·66	93·7	14·00	*idem*

Decrease of Sucrose in Over-ripe Cane.—If cane is allowed to stand too long the sucrose content and purity fall, as the following figures demonstrate :—

III.

Date of Analysis.	Brix.	Sucrose.	Purity.	Available Sugar.	
6th May ..	18·9	17·26	91·4	13·62	
24th May ..	19·3	17·76	92·1	13·09	Black Java
3rd June ..	19·7	18·41	93·4	13·75	12 months
12th June ..	19·9	18·59	93·4	13·88	of age.
2nd July ..	19·3	17·59	91·7	12·96	

It is evident that in the practice of sugar factories the sucrose content and the quotient of purity do not rise and fall regularly, since all kinds of factors such as rain and drought, attacks of cane diseases and pests, intervene and influence the course of ripening. Kuyper* mentions the rain in this respect, and states that the lowest part especially of the cane stalk suffers first and strongest from the action of lightly affective factors, such as relatively light rains.

The quotient of purity shows in general the strongest oscillations and is therefore the best criterion for the detection of irregularities during ripening. A considerable decrease in the quotient of purity in the juice of every part of the cane points to large disturbances in the growth, especially so if this decrease is accompanied by a rise in the figure for the solids.

Period of Ripening of different Varieties.—One variety of cane ripens more quickly than another, which may be due to the fact that in general the increase in available sugar in one cane variety is much more rapid than it is in others. Every factor accelerating or retarding the ripening of the cane exerts its influence on every variety in a similar though not identical manner, so that the differences occasioned by them are more qualitative than quantitative.

For every estate, for every canefield, and for every year the progress of the ripening is a different one, so that no generally standard figures can be given in this respect.

It is therefore only meant as a broad illustration when we give here the increase in the available sugar at intervals of 10 days for different varieties :—

* "Archief voor de Java Suikerindustrie," 1922, II., 320.

Black Java.	Seedlings No.			
	100	247	146	135
11·62	11·02	7·87	10·4	9·77
12·64	11·50	8·15	10·76	10·08
13·20	12·27	9·22	10·97	10·47
13·88	12·57	9·34	11·81	10·52
14·38	12·94	9·35	12·08	
	13·71	9·87	12·35	
	13·72		12·96	
	13·54			

The increase in available sugar was for

Black Java	..	2·76	per cent.	in 50	days or	0·55	per cent.	in 10 days
Seedling 100	..	2·52	,,	,, 60	,,	0·42	,,	,,
Seedling 247	..	2·00	,,	,, 60	,,	0·33	,,	,,
Seedling 146	..	2·55	,,	,, 70	,,	0·36	,,	,,
Seedling 135	..	0·75	,,	,, 40	,,	0·10	,,	,,

Ratoons.—When stools are allowed to ratoon, the sucrose content of the ratoon canes is generally superior to that of the plant canes, although the tonnage is apt to diminish each year, unless the field is " supplied " with new plants which cannot be counted as ratoons.

Chiquelin and Verret* report the following results of their analyses of plants and of first and second ratoons of two varieties of cane,which clearly illustrate the falling off in weight and the improvement in sucrose content.

There can, of course, be no general rule, since the two succeeding crops ripen in two different years under sometimes very different circumstances. In some years a long drought retards the growth of the plant canes and, in such a year, a poor crop will be obtained, while in the following year if there is more rain the ratoons of that cane may yield a heavy return.

It is therefore obvious that the difference in yield per acre between plant canes and the different ratoons cannot be exactly stated, but, generally speaking, the weight of cane per acre diminishes every time the canes are ratooned.

* Bulletin No. 91, Louisiana Exp. Station.

		Plant.	1st Year Ratoons.	2nd Year Ratoons.
Striped cane.	Weight of Stalk	1894 gm.	1262 gm.	1042 gm.
	Fibre	6·56%	7·45%	8·02%
	Sucrose	4·79	6·03	8·45
	Dextrose	2·05	2·27	1·97
	Levulose	1·60	1·73	1·64
	Ash	0·39	0·27	0·27
	Acids	0·21	0·18	0·11
	Albuminoids	0·08	0·06	0·07
	Amides	0·08	0·02	0·02
	Gums	0·06	0·07	0·08
D. 74 cane.	Weight of Stalk	1575 gm.	1497 gm.	1163 gm.
	Fibre	6·28%	7·12%	7·16%
	Sucrose	6·33	7·36	8·24
	Dextrose	1·84	1·65	1·83
	Levulose	1·35	1·20	1·12
	Ash	·40	·41	·39
	Acids	·21	·22	·18
	Albuminoids	·06	·04	·07
	Amides	·10	·03	·02
	Gums	·07	·07	·09

A couple of analyses out of many hundreds made by Francis Watts* in Antigua show the quality of the juice from plant canes and their first ratoons, the figures being obtained by analysing a few canes from every parcel.

I.—Sealy Seedling, Cassada Garden.

	Weight Cane. Tons per acre.	Per cent. Juice.	Sucrose lbs. per gallon.	Glucose lbs. per gallon.	Non-sugar lbs. per gallon.	Total Solids lbs. per gallon.	Purity.
Plant, 1906, *a* ..	14·6	53·2	2·254	0·033	0·136	2·423	93·0
,, ,, *b* ..	12·9	55·5	2·361	0·027	0·186	2·361	90·9
First Ratoons, *a* ..	20·2	53·0	2·145	0·088	0·167	2·400	89·4
,, ,, *b* ..	20·2	56·0	2·228	0·069	0·151	2·448	91·0

* "Sugar Cane Experiments in the Leeward Islands." Report 1905-6, 1906-7.

II.—Queensland Creole, Diamond.

Plant, 1906, *a* ..	32·4	54·5	2·106	0·023	0·132	2·261	93·0
,, ,, *b* ..	37·7	57·0	2·184	0·043	0·107	2·334	93·6
First Ratoons, *a* ..	16·8	52·0	1·685	0·063	0·165	1·913	88·1
,, ,, *b* ..	16 6	52·0	1·937	0·112	0·165	2·214	87·5

Variations in Sucrose Content and Purity.—In order to show the differences in sucrose content of cane and first mill juice during the course of the grinding season, we here give the average sucrose content of cane and the Brix, sucrose, and quotient of the undiluted juice extracted during the various periods of the season, on three different estates.

I.

Period	Sucrose in Cane.	Juice.		
		Brix.	Sucrose	Quotient
April 28—May 10	13·03	17·85	15·00	84·03
May 11—20 ..	13·41	18·26	14·96	81·92
May 21—31 ..	13·72	17·97	15·90	88·48
June 1—10 ..	13·65	18·96	15·87	83·70
June 11—20 ..	13·44	18·93	15·92	84·09
June 21—30 ..	13·22	19·19	16·51	86·03
July 1—10 ..	13·69	19·28	16·69	86·56
July 11—20 ..	14·30	19·32	16·85	87·21
July 21—31 ..	13·38	18·77	15·98	85·13
August 1—10 ..	14·26	18·78	16·30	86·79
August 11—20 ..	13·96	19·04	16·56	86·97
August 21—31 ..	13·74	18·96	16·22	85·54
September 1—10..	14·53	19·54	16·72	85·60
Total	13·73	18·94	16·16	85·35

II.

Period	Sucrose in Cane.	Juice.		
		Brix	Sucrose	Quotient
May 23—31 ..	13·97	19·16	17·09	89·19
June 1—10 ..	14·19	19·29	17·27	89·25
June 11—20 ..	14·44	19·20	17·14	89·27
June 21—30 ..	14·27	18·92	16·91	89·37
July 1—10 ..	15·40	19·53	18·09	92·62
July 11—20 ..	15·07	19·43	18·02	93·16
July 21—31 ..	14·40	19·15	17·43	91·01
August 1—10 ..	14·40	19 11	16·95	88·69
August 11—20 ..	12·81	18·90	16·48	87·19
August 21—31 ..	15·15	19·71	17·67	89·64
September 1—10	14·78	18·46	16·61	89·97
September 11—20	14·12	18·54	—	—
September 21—30	14·28	18·73	16·46	87·88
Total	14·72	19·20	17·26	89·89

III.

April 23—May 15..	14·74	19·40	17·56	90·5
May 16—31 ..	13·65	18·20	16·01	88·0
June 1—15.. ..	14·15	19·1	16·45	86·07
June 16—30 ..	14·72	19·2	17·10	89·1
July 1—15	14·44	18·7	16·46	88·0
July 16—31 ..	14·51	18·8	16·78	89·3
August 1—15 ..	13·91	18·5	15·98	86·4
Aug. 16—Sept. 5..	14·12	19·5	17·28	88·6
Total	14·39	18·9	16·78	88·8

Sucrose Content in various Countries.—It is extremely difficult to obtain figures for the average sucrose content of the canes in certain countries. Figures can be quoted, but they relate chiefly to the results of a single estate in one season, and cannot be considered as representing averages. One must therefore abstain from repeating here the various items of information to be found scattered throughout the literature of the subject, and need only mention that in countries where the period of vegetation is shortened by frost, the sucrose content generally remains low, because the great bulk of the cane is harvested before maturity. In countries having little rainfall, as in some parts of Hawaii and Peru, where the cane sometimes stands 20 months or more and is liable to become partially dry when fully ripe, the sucrose content may rise as high as 16 per cent. and that of the reducing sugar fall very low. In the broad belt of sugar-producing countries with a hot and moist climate, viz., Central America, the Antilles, and Java, the sucrose content is fairly constant and, though occasionally exceptionally high or low, this percentage may be put down as 13 to 15 per cent. in the cane when it reaches the mill.

After the statistics published annually by the Java Sugar Experiment Station, the sucrose content of the cane in that island has been the following in the years since 1899 :—

1899	13·99	..	1907	13·11	..	1915	11·63
1900	12·66	..	1908	12·30	..	1916	12·42
1901	12·68	..	1909	12·16	..	1917	12·82
1902	13·43	..	1910	12·54	..	1918	13·63
1903	12·40	..	1911	12·71	..	1919	12·38
1904	13·04	..	1912	12·35	..	1920	12·94
1905	12·66	..	1913	12·54	..	1921	13·41
1906	12·44	..	1914	11·91	..	1922	12·87
						1923	13·06

J. Kuyper* investigated the distribution of the solids in the juice of the different parts of the cane stalk and published the following conclusions as the outcome of his investigations :—

1. The percentage of solids in the ripe cane increases absolutely regularly from the top to the bottom end ; the only exception to this rule is given by the topmost joints in which sometimes the concentration is higher than in those immediately underneath.

2. In well-growing cane three zones may very clearly be distinguished : No. 1, consisting of joints 1—6 from the top, the really growing part, possesses a low and constant percentage ; No. 2, joints 7—12, the intermediate part, shows a rapidly increasing solids content ; and No. 3, the rest of the stalk (the real sugar store) has a slowly but constantly increasing percentage of solids in the juice.

* "Archief voor de Java Suikerindustrie," 1918, 1665.

3. In every joint a zone of 0·5 to 1 inch in height on both sides of the place of attachment of the leaf-sheath contains a low percentage of solids in the juice.

4. In most cases the juice in the periphery of the stalk has a higher solids content than that of the centre, both in the joint and in the node. The rind to a thickness of 0·05 to 0·1 inch shows the highest percentage of solids in the juice. The juices of the hardest parts of the cane have therefore the highest solids content.

5. The figures vary according to the varieties.

Ratio of the three Sugars in Cane.—The proportion in which the three sugars occur in the cane largely depends on the age of the different parts of the cane.

We gave elsewhere 0·73 per cent. sucrose and 0·60 per cent. reducing sugar, consisting of glucose and fructose, in cane leaves which are actively assimilating.

The following figures are for cane at different periods of development.

	Sucrose.	Glucose.	Fructose.
White tops of cane 6 months old ..	1·02	1·24	1·25
,, ,, 9 ,, ,, ..	1·90	1·30	0·70
Bottom joints ,, 12 ,, ,, ..	16·50	0·60	0·20

The fructose content decreases gradually; but even in the juice of the riper joints its presence may always be detected by means of the reaction with ammonium molybdate and acetic acid, mentioned on page 44.

The table of Browne given below also shows that the amount of reducing sugar and especially of fructose decreases as ripening proceeds.

Decrease in the Reducing Sugar Content during Ripening.

Constituents.	9th September	23rd September	7th October	21st October	4th November	18th November	28th November
Brix	10·47	11·52	14·69	14·70	15·48	17·22	19·45
Sucrose	5·97	7·32	11·27	11·60	13·60	15·86	18·29
Glucose	2·15	2·00	1·58	1·20	0·46	0·36	0·21
Fructose	1·53	1·55	0·93	0·97	0·56	0·27	0·04
Ash	0·36	0·32	0·32	0·35	0·36	0·32	0·32
Free Acids	0·07	0·07	0·09	0·08	0·10	0·07	0·06
Combined Acids ..	0·08	0·08	0·09	0·10	0·10	0·09	0·17
Albuminoids.. ..	0·09	0·08	0·08	0·08	0·08	0·10	0·18
Gum	0·08	0·07	0·07	0·05	0·17	0·14	0·18
Purity	57·02	63·54	76·72	78·91	87·85	92·10	94·04
Glucose Co-efficient	61·64	48·50	22·27	18·70	7·50	3·97	1·37

Rotatory Power of Reducing Sugars.—The following table gives analytical data concerning the juice of ripe and nearly-ripe cane, in which the rotatory power of the reducing sugar is expressed in degrees Ventzke.

Character of the Juice.		Brix.	Polarization, before Inversion.	Polarization after Inversion.	Calculated Rotation due to Sucrose.	Rotation of the Reducing Sugar	Reducing Sugar.	Specific Rotation of the Reducing Sugar.	Sucrose.	Glucose.	Fructose.
Bourbon Cane	Bottom piece ..	18·8	71·9	—21·1	72·6	—0·66	1·27	—13·5	17·57	0·76	0·51
	Middle piece ..	18·5	69·3	—20·9	70·5	—1·17	1·34	—22·5	17·06	0·73	0·61
	Top piece ..	14·3	44·8	—14·7	46·5	—1·70	2·30	—19·4	11·44	1·17	1·03
Black Java	Bottom piece ..	16·3	60·3	—18·0	61·2	—0·90	1·42	—16·0	14·93	0·72	0·60
	Middle piece ..	15·4	54·3	—16·5	55·2	—0·90	1·61	—14·2	13·52	0·97	0·64
	Top piece ..	11·7	30·2	—11·0	32·2	—2·00	2·30	—20·3	8·02	1·27	1·03
" "	Bottom half ..	14·4	48·0	—13·9	48·3	—0·30	2·20	— 3·9	11·88	1·43	0·77
	Top half ..	11·2	29·8	—10·5	31·5	—1·70	3·02	— 1·4	7·85	2·11	0·91
" "	Bottom half ..	19·9	75·3	—21·9	75·9	—0·6	0·67	—21·2	18·25	0·34	0·31
	Top half ..	17·5	65·3	—19·7	66·4	—1·1	1·05	—27·2	16·13	0·57	0·58
" "	Bottom half ..	22·5	86·2	—24·2	86·2	0	0·50	0	20·52	0·35	0·15
	Top half	22·76	93·0	—26·0	92·9	+0·1	0·35	+ 0·7	22·11	0·25	0·10
" "	Bottom half ..	22·07	89·3	—25·0	89·3	0	0·37	0	21·30	0·25	0·12
	Top half ..	22·73	92·4	—25·6	92·2	+0·2	0·37	+14	21·94	0·30	0·07
" "	Bottom piece ..	19·94	78·21	—22·0	78·3	—0·1	0·83	— 3	18·82	0·54	0·29
	Middle piece ..	20·38	80·3	—23·0	80·7	—0·4	0·55	—20	19·41	0·30	0·25
	Top piece ..	21·05	82·7	—23·6	83·0	—0·3	0·64	—12	19·88	0·46	0·18
" "	First Mill Juice..	18·87	69·5	—19·8	69·8	—0·3	1·10	— 7	16·86	0·71	0·39
" "	Mixed Juice ..	18·40	67·2	—18·7	67·1	—0·1	1·46	— 2	16·22	1·01	0·45
" "	" " ..	19·7	70·5	—20·0	70·7	—0·2	1·16	— 1·4	16·92	0·81	0·35
" "	" " ..	19·2	67·5	—19·25	67·7	—0·2	1·61	— 3·6	16·21	1·06	0·56
" "	" " ..	16·0	48·5	—13·9	48·7	—0·2	2·39	— 2	11·88	1·59	0·80
" "	" " ..	18·8	61·0	—17·8	61·6	—0·6	2·00	— 7·1	14·88	1·30	0·70
" "	" " ..	19·1	69·1	—18·9	68·8	—0·3	1·42	— 5·4	16·56	0·92	0·50
" "	" " ..	19·3	68·4	—16·2	69·2	—0·8	2·27	— 9·1	16·66	1·45	0·82
" "	" " ..	19·0	70·0	—20·0	70·3	—0·3	1·22	— 6·4	16·92	0·79	0·43
" "	" " ..	17·9	64·5	—18·5	65·0	—0·5	1·46	— 8·6	15·62	0·92	0·54

From the rotations at a temperature of 28° (glucose = 80 and fructose = — 134), the proportions of the two reducing sugars may be calculated from their total quantity and the rotation of the mixture.

As a rule the ripest cane contains the smallest percentage of fructose, though it must here be explained that these figures possess only relative values, since the slightest deviation in temperature or error in polariscopic reading is sufficient, when so small a quantity is under consideration, to influence the result considerably. A difference of 0·1° in the reading is sufficient, in some cases, to change a positive rotatory power into a negative one, so that the figures given in the preceding table should not be regarded as rigidly accurate.

Fibre Content of Different Parts.—The fibre content in the different parts of the cane varies considerably; not only do the bottom joints contain more fibre than the middle and upper ones, as is clearly shown by the many analyses on page 66 and following, but in the same joint the fibre content varies considerably according to the larger or smaller percentage of fibrovascular bundles and parenchyma cells which are present in the different parts.

Winter* separated, as far as possible, the hard fibrovascular bundles from the soft pith of the cane, which had previously been skinned, and ascertained the weight of both constituents; but he admitted that the separation was by no means complete.

		Sucrose.	Fibre.	Juice.	Sucrose in 100 Juice.
1	Fibrovascular bundles ..	15·63	14·22	85·78	18·22
	Parenchyma cells ..	18·88	5·00	95·00	19·87
2	Fibrovascular bundles ..	15·47	11·75	88·25	17·53
	Parenchyma cells ..	19·29	4·50	95·50	20·20
3	Fibrovascular bundles ..	14·04	12·28	87·72	16·01
	Parenchyma cells ..	17·40	4·41	95·59	18·20
4	Fibrovascular bundles ..	9·83	9·00	91·00	10·80
	Parenchyma cells ..	11·11	4·20	95·80	11·60
5	Fibrovascular bundles ..	14·54	9·17	90·83	16·01
	Parenchyma cells ..	16·15	4·00	96·00	16·82

In a second series with ripe and unripe cane the purity of the juice was also determined.

	Cane.						Juice.			
	Sucrose.	Fibre.	Juice.	Dry Substance.	Non-Sugar	Purity.	Sucrose.	Dry Substance.	Non sugar.	Purity.
Fibrovascular bundles ..	14·54	9·17	90·83	26·50	2·79	54·87	16·01	19·08	3·07	83·91
Parenchyma	16·15	4·00	90·60	21·82	1·67	74·01	16·82	18·56	1·74	90·63
Fibrovascular bundles ..	11·55	9·61	90·39	23·06	1·90	50·09	12·78	14·88	2·10	85·89
Parenchyma	12·24	4·25	95·75	18·04	1·55	67·85	12·78	14·40	1·62	88·75
Fibrovascular bundles ..	10·98	12·57	87·43	25·64	2·09	42·82	12·56	14·95	2·39	84·01
Parenchyma	11·00	5·76	94·24	18·70	1·94	58·82	11·67	13·73	2·06	85·00

* "Mededeelingen van het Proefstation Kagok" I., page 30.

Sugar-free Sap in Fibrovascular Bundles.—Winter remarks that the fibrovascular part of the cane contains so little sugar that he firmly believes the bundles to be totally sugar-free, which is in accord with the fact mentioned by the same investigator that the watery drops which ooze out from the ends of cane stalks while these are being crushed in the factory mills contain no sugar and only a little starch and salts. Went observes that although the fibrovascular bundles are the channels by which the sugar is conducted through the stalk, yet it is not altogether surprising that they contain a sugarless sap, since the sugars are transported in so dilute a solution as not to be detected by the usual analysis.

Difference in Fibre Content of a Single Joint.—Winter found a great difference in composition in various parts of the same joint, according as they belonged to the rind, the periphery, or the centre of the internode. The joints of a cane were cut from the stalk with a saw and the nodes separated, so that only the internodes were examined. A cylindrical piece, 1·5 cm. in diameter, was cut out from the middle of each internode in a longitudinal direction by means of a cork-borer to represent the " centre." The remainder, after being peeled, served to represent the " periphery," whilst the " rind " was cut into pieces not over 1 mm. thick with scissors.

		Sucrose.	Fibre.	Juice.	Sucrose in Juice.
1	Centre	17·6	4·46	95·54	18·42
	Periphery	18·5	6·15	92·85	19·92
	Rind	9·6	25·31	74·69	12·85
2	Centre	15·0	3·57	96·43	15·56
	Periphery	14·6	6·45	93·55	15·61
	Rind	—	29·29	70·71	—
3	Centre	19·2	4·72	95·28	20·15
	Periphery	19·0	9·29	90·71	20·95
	Rind	5·99	41·75	58·25	10·28
4	Centre	17·6	4·68	95·32	18·46
	Periphery	17·1	8·60	91·40	18·71
	Rind	5·21	46·11	53·89	9·67

Winter also ascertained the fibre content of the internodes and the nodes belonging to them, in which case the piece of the stalk 1·5 cm. on either side of the axis of the leaf was considered to be the node.

		Sucrose.	Fibre.	Juice.	Sucrose in Juice.
1	Nodes	15·5	16·46	83·54	18·55
	Internodes	17·5	11·69	88·31	19·82
2	Nodes	15·7	11·62	88·38	17·76
	Internodes	17·5	9·15	90·85	19·26
3	Nodes	13·8	17·77	82·23	16·78
	Internodes	16·9	11·02	88·98	18·99
4	Nodes	17·5	18·31	81·69	21·42
	Internodes	19·1	11·52	88·48	21·59
5	Nodes	13·4	16·51	83·49	16·05
	Internodes	16·4	10·02	89·98	18·23

Beeson* investigated the composition of nodes and joints of the different parts of the stalk, and obtained the following data :—

		Dry Substance.	Sucrose.	Reducing Sugar.	Purity.	Non-sugar.	Glucose ratio.	Fibre in Cane.
Top end ..	Nodes ..	15·3	11·30	0·10	73·9	3·82	1·60	15·86
	Internodes ..	16·9	14·30	1·25	84·6	1·35	8·37	8·60
Middle ..	Nodes ..	16·7	13·7	0·07	82·0	2·90	0·57	15·90
	Internodes ..	17·7	16·0	0·98	90·4	0·72	6·13	8·00
Bottom end	Nodes ..	15·7	12·8	0·15	81·5	2·75	1·17	18·28
	Internodes ..	17·7	16·4	0·61	92·6	0·69	3·78	8·00

The nodes and the rind contain the largest percentage of fibre, the older parts more so than the younger ones. Winter, as well as Beeson, Bonâme,† and Pellet,‡ observe that the hardest parts yield the least pure juice, while the latter two also mention that the hard rind contains the most impure juice, rich in reducing sugars and darkly coloured.

* "Bull. Assoc. Chim. Sucr. et Dist.", 1895–6, 362. † "La Canne à Sucre."
‡ "Etudes sur la Canne à Sucre," 30.

Noël Deerr* examined a number of cane varieties grown in the Hawaiian Islands and went to work as follows :—

He divided the cane into pith and rind and node. Thus the cane under analysis was first separated into node and internode by sawing through the node, the datum mark he adopted being the outer line of the zone of adventitious roots and a corresponding distance on the other side. The portion thus obtained formed the material analysed as the node. The rind was not separated from the node, so that this portion consisted mainly of rind, fibrovascular bundles, and some pith. The other part consisting of the internode was stripped of the rind. On examining a clean-cut cane, a fairly sharp line of demarcation between pith and rind could be found and the rind was stripped off as nearly as possible through this line ; this portion formed the rind, the remainder being analysed as pith. The separate portions were weighed and analysed as regards moisture, fibre, solids and sugar. The moisture was determined by drying in an air-bath at 100° C., the soluble solids and the sugar by extraction in a Soxhlet apparatus and evaporation of the extract, which afterwards was filled up to a given volume, in which solution the dry substance was found by means of the refractometer and the sucrose by polarization ; the fibre was not determined in a direct way but found indirectly. The figures are tabulated on next page

On examining the results it appears that that part of the cane which has been taken as representative of the node is intermediate in composition between the parts representative of the parenchyma and rind tissue ; and in a very rough sense it may be considered as composed of those parts which go to make up the parenchyma and rind tissues. It is then permissible to regard the cane as consisting of a soft interior portion made up of a low proportion of spongy, absorbent fibre, and a juice of a higher sugar content and purity, and of a hard outer portion containing a large proportion of a resistant non-absorbent fibre and a juice of low sugar content and low purity. Dividing the part called " node " equally between the pith and the rind tissue, the average composition of the canes forming the basis of the experiments referred to here will be as given on page 99.

* Bulletin No. 30, Hawaiian Exp. Station, 34. " Int. Sugar Journal," 1910, 395, 453.

		Constituents.	Rose Bamboo.	Yellow Caledonia	Lahaina	Lahaina.	Yellow Caledonia
Juice of	Whole Cane	Weight on 100 cane ..	100·00	100·00	100·00	100·00	100·00
		Juice	87·12	84·91	86·25	88·40	84·45
		Fibre	12·88	15·09	13·75	11·60	15·55
		Solids	14·70	15·83	16·03	20·10	17·92
		Sucrose	13·25	13·04	13·28	18·14	15·36
		Water..	72·12	69·08	70·22	68·30	66·53
	Parenchyma	Weight on 100 cane ..	74·28	66·90	72·45	61·77	67·15
		Juice	91·90	90·43	90·11	94·78	91·20
		Fibre	8·10	9·57	9·89	5·22	8·80
		Solids	15·93	17·34	17·45	22·21	19·62
		Sucrose	14·80	15·06	15·11	21·11	17·52
		Water..	75·97	73·09	72·66	72·57	71·60
Juice of	Rind	Weight on 100 cane ..	9·57	15·27	12·28	14·34	15·80
		Juice	62·11	65·92	72·73	69·98	64·92
		Fibre	37·89	34·71	27·27	30·02	35·08
		Solids	9·38	11·52	11·54	15·40	13·87
		Sucrose	6·46	7·44	7·50	11·10	10·00
		Water..	52·73	53·77	51·19	57·58	51·05
	Node	Weight on 100 cane ..	16·15	17·83	15·27	23·89	17·05
		Juice	79·98	80·40	78·78	82·80	75·97
		Fibre	20·02	19·60	21·22	17·20	24·03
		Solids	12·22	13·86	12·88	17·43	15·07
		Sucrose	10·14	10·27	9·22	14·62	11·83
		Water..	67·76	66·54	65·90	65·37	60·90

The constitution of the juice contained in the different parts of the cane was calculated in the following way:—

		Constituents.	Rose Bamboo.	Yellow Caledonia	Lahaina	Lahaina	Yellow Caledonia
Juice of	Whole Cane	Weight on 100 cane.. ..	87·12	84·91	86·25	88·40	84·45
		Solids	16·87	18·52	18·59	22·72	21·22
		Sucrose	15·22	15·35	15·39	20·52	18·19
		Quotient	90·22	82·94	82·79	90·32	85·71
	Parenchyma	Weight on 100 cane.. ..	68·22	60·49	65·49	58·62	61·24
		Solids	17·33	19·17	19·37	23·43	21·49
		Sucrose	16·10	16·65	16·76	22·27	19·20
		Quotient	92·90	86·85	86·53	95·05	89·29
	Rind	Weight on 100 cane.. ..	6·04	10·06	8·91	10·00	10·26
		Solids	15·08	17·41	15·87	22·00	21·37
		Sucrose	10·40	11·29	10·30	15·86	15·40
		Quotient	69·10	64·85	64·91	72·09	72·10
	Node	Weight on 100 cane.. ..	12·82	14·36	11·85	19·78	12·95
		Solids	15·28	17·24	16·29	21·05	19·83
		Sucrose	12·68	12·77	11·70	17·66	15·57
		Quotient	82·98	74·07	71·81	83·50	78·50

WHOLE CANE.		JUICE.	
Weight per 100 cane ..	100·00	Weight per 100 cane ..	86·3
Fibre per cent. ..	13·7	Solids per cent.	19·8
Solids ,, ...	17·1	Sucrose ,, ...	17·1
Sucrose ,, ...	14·8	Purity ,, ...	86·4
SOFT PART.		JUICE.	
Weight per 100 cane ..	77·0	Weight per 100 cane ..	70·8
Fibre per cent. ..	8·0	Solids per cent.	20·2
Solids ,, ...	18·5	Sucrose ,, ...	18·5
Sucrose ,, ...	16·7	Purity ,, ...	90·3
HARD PART.		JUICE.	
Weight per 100 cane. ..	23·0	Weight per 100 cane ..	15·5
Fibre per cent. ..	33·0	Solids per cent.	18·3
Solids ,, ...	12·3	Sucrose ,, ...	12·7
Sucrose ,, ...	8·5	Purity ,, ...	69·1

Fibre Content of Pith Bundles and Rind.—According to Browne,* the pith occupies 70 per cent of the volume of the cane, but its fibre content is so little that it does not amount to more than 25 per cent. of the whole quantity of dry fibre. A mechanical separation of the tissues of the cane from one another gave the following percentage composition. The analyses were performed upon a mature stalk of the Louisiana Purple cane.

	Pith. Per cent.	Bundles. Per cent.	Rind. Per cent.
Whole cane (three analyses) ..	2·39 ..	1·81 ..	5·51
Dry fibre	24·66 ..	18·60 ..	56·74

The following figures were found for some very common Java varieties :—

	Cheribon.	No. 36.	No. 100.	No. 139.	No. 247.
Pith	25·6	20·0	22·82	20·0	20·0
Bundles	31·5	26·5	25·37	35·97	26·1
Rind	42·9	53·5	51·81	44·03	53·9

From a physical point of view these fibres show a distinctly marked difference. The rind and the bundles are woody, while the pith consists of thin-walled cells. The difference in water-absorbing power is very considerable. The bundles only absorb five times their weight of water, whereas the fibre from the parenchyma absorbs as much as 30 times its own weight.

* Bulletin No. 117, Louisiana Exp. Station.

As the chemical composition of the fibre does not vary very much it is mainly its physical condition which accounts for the observed differences in the water-absorbing power of the bagasse of the different cane varieties.

Constituents.	Fibre from the Pith.	Fibre from the Bundles.	Fibre from the Rind.
Cellulose	49·00	50·00	51·05
Pentosan	32·04	28·67	26·93
Albuminoids	1·94	2·00	2·19
Cane wax and fat	0·41	0·72	0·98
Ash	1·68	3·58	1·64
Lignin (by difference) ..	14·93	15·03	17·17

Pentosan in the Juice.—Hazewinkel and Langguth Steuerwald* found the pentosan content in the juice to increase in proportion to the time elapsing between the cutting and the grinding of the cane.

Variety.	Pentosan on 100 Parts of Solids in Raw Juice on grinding the Cane 1—6 Days after its being cut.			
	1	3	5	6
No. 100 ..	—	0·22	—	0·36
No. 100 ..	0·25	0·30	0·38	—
No. 247 ..	0·23	0·27	0·29	—

Nitrogenous Bodies.—According to van Lookeren Campagne,† cane juice contains an average of 0·07 per cent. of albuminoids, consisting of two kinds, one of which is coagulated by heat and lime and amounts to 80 per cent. of the total quantity, whilst the other remains in solution after liming and boiling. Furthermore, the juice contains the same amount of other nitrogenous bodies, chiefly amino acids. After heavy crushing, the resulting juice contains more albumin than after moderate crushing.

The nitrogenous bodies are not distributed evenly throughout the stalk, the work of Beeson having shown a larger accumulation of albuminoids in the

* "Int. Sugar Journal," 1911, 284.
† "Archief voor de Java Suikerindustrie," 1899, 757.

nodes and of amides in the internodes, which accounts for the higher percentage of albuminoids in juice extracted by heavy mills, since these are able to crush the hard nodes also.

	Albuminoid Nitrogen.	Amide Nitrogen.	Total Nitrogen.
Nodes	0·1778 ..	0·0051 ..	0·1829
Internodes	0·0559 ..	0·0258 ..	0·0817

Mineral Matters (Ash) in Cane.—As already stated the mineral matter occupies a rather subordinate place in the quantitative composition of the cane. We have already given the inorganic constituents of the whole cane and of the fibre, and now include in the table on next page those of the juices of different cane varieties.

Lime. Magnesia. Soda. Potash.—Calcium phosphate is very prominent among the inorganic elements of the cane, but the amount of lime otherwise combined is not considerable. Magnesia is also always present but only in small proportions even when the cane is grown on salt land. If the soil contains sodium sulphate, or sodium, calcium, or magnesium chlorides, these salts act on the potash silicates of the soil in such a manner that potash is liberated and rendered available to the cane. This is the reason why potash is about the only alkali base in the cane, unless the soil is quite destitute of potash and rich in sodium chloride. In such a case the cane is compelled to absorb sodium chloride, but does not thrive and soon dies. The juice of such a cane gave the following figures calculated on the weight of 460 grms. of cane and on 100 parts of the cane.

Soda	1·016 grm.	in per cent.	0·221
Potash	0·199 ,,	,, ,,	0·043
Lime	0·358 ,,	,, ,,	0·078
Magnesia	0·146 ,,	,, ,,	0·032
Sulphuric acid	0·680 ,,	,, ,,	0·147
Phosphoric acid	0·107 ,,	,, ,,	0·023
Carbonic acid	0·295 ,,	,, ,,	0·064
Silica	0·915 ,,	,, ,,	0·199
Chlorine	0·870 ,,	,, ,,	0·190
	4·586		0·997
Subtract oxygen equivalent to chlorine	0·196 ,,	,, ,,	0·043
	4·390		0·954

Estate.	Variety.	On 100 Juice.												
		Brix.	Quotient of Purity.	Ash.	Silica.	Ferric and Calcium Phosphate.	Lime otherwise combined.	Magnesia.	Sulphuric Acid.	Chlorine.	Potash.	Carbonic Acid in the Ash.	Carbonic Acid on 100 Ash.
Wonopringgo ..	Cheribon	21·51	95·12	0·110	0·022	0·034	0·010	—	0·005	0·003	0·016	0·011	10·0
,, ..	No. 247	18·87	85·10	0·131	0·035	0·041	0·013	—	0·009	0·002	0·016	0·011	8.4
Djatibarang .	Cheribon	20·4	88·82	0·189	0·034	0·045	0·004	—	0·038	0·009	0·057	0·011	5·3
Asembagoes ..	No. 105 ..	13·7	74·38	0·767	0·040	0·035	0·012	0·013	0·097	0·172	0·403	0·025	3·2
,, ..	Louziers	19·0	83·10	0·521	0·025	0·059	0·00	0·019	0·052	0·111	0·241	0·016	3.1
,, ..	No. 105 ..	16·6	81·02	0·776	0·027	0·018	0·007	0·009	0·101	0·181	0·428	0·034	4.4
Kentjong	G. Z. A.	15·3	79·02	0·230	0·039	0·052	0·007	0·008	0·039	0·005	0·056	0·001	0·3
Poerwodadi ..	——	16·5	83·6	0·342	0·156	0·081	0·006	0·002	0·038	0·017	0·113	0·015	4·4
,, ..	——	17·7	91·90	0·297	0·022	0·032	0·003	0·005	0·061	0·025	0·140	0·017	5·7
Karangsoewoeng	——	20·53	83·05	1·027	0·042	0·190	0·022	0·018	0·163	0·153	0·474	0·064	6·2
Balapoelang ..	——	19·6	87·96	0·514	0·064	0·025	0·010	0·016	0·025	0 040	0·222	0·080	15·6
,, ..	——	16·5	81·52	0·748	0·080	0·070	0·007	0·008	0·092	0·065	0·342	0·084	11·2
Bantoel	——	—	—	0·454	0·020	0·037	0·051	0·012	0·037	0·036	0·170	0·040	8·9
Waroe	No. 247 ..	15·37	81·86	0·540	0·042	0·084	0·008	0·003	0·064	0·100	0·233	0·017	3·2
.. .. ..	No. 247 ..	18·18	87·36	0·349	0·016	0·062	0 007	0·003	0·024	0·065	0·155	0·024	7·0
Soedhono	——	16·65	89·3	0·517	0·271	0·101	0·016	0·003	0·018	0·008	0·067	0·025	4·8
Kanigoro	No. 139 ..	15·68	82·5	0·252	0·057	0·052	0·002	0·006	0·029	0·011	0·076	0·018	7·2
,, ..	Striped Preanger	16·16	85·5	0·275	0·043	0·044	0·003	0·004	0·043	0·017	0·094	0·016	6·8
.. .. ..	No. 36 ..	14·52	80·7	0·472	0·138	0·088	0·009	0·010	0·061	0·021	0·115	0·018	3·8
,, ...	Manilla ..	15·18	80·8	0·420	0·029	0·100	0·009	0·010	0·076	0·008	0·125	0·043	10·2
,, ...	No 71 ..	12·15	72·3	0·200	0·042	0·057	0·004	0·002	0·040	0·00	0·045	0·009	4·5
,, ...	Cheribon	18·21	89·7	0·164	0·014	0·018	0·005	0·007	0·024	0·003	0·052	0·021	12·8
,, ...	No. 86 ..	16·34	85·1	0·285	0·017	0·036	0·010	0·016	0·057	0·017	0·099	0·034	11·9
,, ...	No. 142 ..	14·11	80·1	0·187	0·016	0·048	0·006	0·015	0·026	0·006	0·054	0·008	4·3
,, ...	No. 33a ..	14·35	79·3	0·224	0·026	0·054	0·010	0·022	0·038	0·006	0·060	0·012	5·0
,, ...	No. 33a ..	14·35	79·7	0·238	0·029	0·058	0·011	0·022	0·039	0·008	0·062	0·013	5·5
,, ...	No. 146 ..	13·10	76·2	0·458	0·017	0·040	0·004	0·005	0·074	0·010	0·243	0·058	12·7
,, ...	No. 135 ..	11·98	69·6	0·222	0·033	0·047	0·005	0·002	0·036	0·013	0·068	0·018	8·5
,, ...	No. 247 ..	14·96	75·2	0·195	0·027	0·054	0·006	0·005	0·030	0·012	0·051	0·014	7·2
,, ...	No. 247 ..	14·27	72·0	0·214	0·032	0·059	0·005	0·005	0·034	0·005	0·057	0·017	7·9

Inorganic Acids.—If, on the contrary, the soil contains sufficient potash, the cane does not absorb soda, but much more potash than if no sodium salts had been present. Lime and magnesia salts also have the property of liberating potash from the silicates and putting it at the disposal of the cane. The cane absorbs those salts also, but more lime than magnesia. Cane first requires potash, and only in a minor degree lime and magnesia, and, lastly, soda in the total absence of other alkali salts; but this latter constituent can by no means take the place of potash in the nutrition of the cane. When much potash is absorbed, the cane ash contains also much chlorine and sulphate, as that base is chiefly combined with inorganic salts in the juice. The figure for organic acids combined with bases, which is indicated by that for carbonic acid in the juice, does not rise accordingly.

Silica.—Ch. Muller* has found certain colloidal organic combinations of silica to occur in juice of cane grown under special conditions. These viscous

* "Int. Sugar Journal," 1921, 579.

bodies which constitute a great impediment in clarification may by heating the juice to a temperature of 116° C. be split up into silica and the organic rest which is coagulated by the action of the high temperature.

Influence of Soil on Inorganic Constituents.—The quantity and quality of the inorganic constituents of the cane depend largely on the character of the soil. A salt soil causes much more inorganic substance to enter into the composition of the cane than a non-saline one, as is clearly shown by the following analyses of cane planted in pots and watered with dilute solutions of various salts. The figures represent milligrams of every constituent on 100 grms. of juice.

	I.	II.	III.	IV.	V.	VI.	VII.	VIII.	IX.	X.	XI.	XII.
NaCl. .. grms.	—	—	—	128·7	128·7	257·4	386·0	257·4	—	—	—	—
$MgCl_2$ .. grms.	120	240	—	—	—	—	—	—	—	—	—	—
$CaCl_2$ .. grms.	—	—	—	—	—	—	—	—	—	211·0	315·0	—
Insoluble Ash ..	147·9	147·0	80·5	75·1	100·7	88·5	148·2	113·0	72·9	106·5	102·0	68·0
Soluble Ash ..	296·5	408·6	178·0	342·5	410·1	500·2	626·6	—	279·5	499·4	633·5	277·7
Total Ash.. ..	444·4	555·6	258·5	417·6	510·8	588·7	774·8	—	352·4	605·9	735·5	345·7
SiO_2	38·0	32·2	27·2	20·2	27·6	22·8	42·8	—	35·0	26·4	28·6	28·4
CO_2	23·2	20·8	32·5	25·1	27·8	11·8	24·6	—	43·4	16·2	16·8	26·8
P_2O_5	59·7	51·3	48·0	47·5	47·4	39·8	52·8	—	28·9	31·9	16·4	59·4
$F_2O_3+Al_2O_3$ ..	15·4	11·5	7·5	2·9	9·8	5·9	13·6	9·5	7·4	5·8	8·6	3·7
CaO	21·9	22·9	14·0	45·6	19·3	18·4	27·0	22·4	14·5	31·1	33·6	12·3
MgO	24·3	31·8	9·6	13·2	14·8	15·8	22·0	16·2	7·0	12·5	13·5	8·8
Cl_2	97·0	161·7	8·4	85·9	114·0	182·1	296·6	*	28·1	198·9	272·1	10·5
SO_3	8·9	8·8	5·6	19·2	23·6	20·9	27·3	—	18·1	21·1	17·0	46·0
K_2O	176·2	251·0	103·2	191·8	243·8	284·2	299·7	—	173·1	305·9	384·3	147·1
Na_2O	1·7	None	4·4	15·6	8·4	28·0	56·1	—	3·2	1·0	5·9	5·1

* The soluble part of the ash of No. VIII. was lost during analysis.

Influence of Age on Ash Content.—Bonâme's investigations† proved that the same cane possesses varying amounts of inorganic substances in the different periods of its growth. The dates of the analyses of whole canes, deprived of the leaves, are recorded below.

It also follows from the analyses of Browne, on page 92, that if the ash content of cane is not high, it does not decrease much during ripening. A certain decrease will always take place, since, during the ripening process, potash, soda, and chlorine are transferred from the cane into the leaves, therefore the green tops contain the largest amount of ash of any part of the cane.

† "Rapport Annuel de la Station Agronomique de l'Ile de Maurice," 1896.

Constituents.	6th May.	6th June.	6th July.	6th August.	6th Sept.	6th October.
Silica	0·805	1·373	0·824	0·913	0·830	1·024
Chlorine	0·074	0·092	0·018	0·010	0·007	0·012
Sulphuric Acid	0·135	0·260	0·121	0·167	0·142	0·173
Phosphoric Acid	0·104	0·155	0·078	0·075	0·070	0·110
Lime	0·115	0·205	0·115	0·136	0·127	0·158
Magnesia	0·154	0·255	0·170	0·158	0·141	0·133
Potash	0·687	0·803	0·250	0·208	0·181	0·387
Soda	0·026	0·017	0·008	0·006	0·008	0·011
Iron Oxide	0·046	0·050	0·020	0·029	0·015	0·024
Total	2·146	3·210	1·604	1·702	1·521	2·032

Browne gives the following results of unripe cane, which he divided into sections, and crushed, and the juice of which he analysed :—

Constituents.	Green Tops cut in the Morning.	Green Tops cut in the Evening.	Green Top Joints.	Middle Joints.	Bottom Joints.
Brix	5·09	5·77	5·06	8·79	12·65
Sucrose	0·54	0·94	0·76	4·12	8·93
Glucose	0·86	0·63	1·81	2·07	1·83
Fructose	0·77	0·69	1·70	1·82	1·23
Ash	1·24	1·42	0·50	0·37	0·33
Free Acids	0·27	0·27	0·18	0·09	0·07
Combined Acids ..	0·54	0·62	0·36	0·18	0·14
Albuminoids	0·18	0·15	0·07	0·08	0·08
Gums	0·17	0·30	0·15	0·11	0·05
Purity	10·60	16·29	15·02	46·87	70·59
Glucose ratio	301·85	137·23	461·84	94·41	34·26

The author has also analysed juice of ripe and unripe canes, which he divided into three parts, crushed, and analysed the juices, as under :—

Constituents.	Unripe Cane from a Field with High Purity			Ripe Cane from a Field with High Purity.			Ripe Cane from a Field with Low Purity.		
	Top.	Middle.	Bottom.	Top.	Middle.	Bottom.	Top.	Middle.	Bottom.
Brix	11·9	13·5	14·2	20·3	20·48	20·37	17·8	19·6	19·3
Sucrose	7·47	10·44	11·57	18·96	19·51	19·30	15·05	17·91	17·52
Reducing sugars	3·40	2·34	2·06	0·50	0·36	0·35	not determined.		
Quotient of purity ..	59·8	74·0	78·1	93·4	95·25	95·20	84·55	91·38	90·78
Ash	not determined			0·208	0·141	0·158	0·300	0·257	0·278
Soluble ash				0·111	0·067	0·081	0·194	0·193	0·206
Potash	0·119	0·073	0·052	0·063	0·038	0·047	0·106	0·104	0·109
Sulphuric acid	not determined			0·027	0·017	0·020	0·078	0·077	0·081
Chlorine				0·020	0·012	0·016	0·008	0·010	0·011
Carbonic acid in the ash..				0·002	0·001	0·001	0·002	0·003	0·003

Changes in Inorganic Matter during Ripening.—The variations in the inorganic constituents during the ripening are shown by the following series of cane juices, from the same fields, which were extracted and analysed at various stages of maturity :—

POERWODADI ESTATE

Cheribon (Black Java) Cane, planted July 1st, 1905.					Seedling No. 247, planted July 15th, 1905.				
Constituents.	Date of the Analyses.								
	9th Feb., 1906.	9th March, 1906.	11th April, 1906.	20th June, 1906.	9th Feb., 1906.	9th March, 1906.	11th April, 1906.	20th June, 1906.	11th July, 1906.
Brix	13·2	15·0	16·3	19·50	12·0	13·0	13·7	16·50	16·90
Polarization	8·75	11·80	13·74	18·32	7·13	9·04	10·46	14·04	14·90
Purity	66·3	78·70	84·30	93·95	59·4	69·5	76·25	85·61	88·20
Reducing sugars	3·8	2·32	1·18	0·49	3·30	2·92	1·51	1·52	1·17
Ash	0·207	0·205	0·224	0·262	0·299	0·304	0·207	0·216	0·273
Insoluble ash	0·068	0·099	0·092	0·063	0·089	0·133	0·075	0·081	0·075
Soluble ash	0·139	0·106	0·152	0·199	0·210	0·171	0·132	0·135	0·198
Potash	0·079	0·052	0·072	0·115	0·120	0·091	0·077	0·072	0·114
Carbonic acid in the ash..	0·009	—	—	0·016	0·017	0·008	—	—	0·012
Sulphuric acid	0·040	0·036	0·056	0·062	0·052	0·048	0·059	0·060	0·063
Chlorine	0·009	0·010	0·004	0·008	0·022	0·016	0·007	0·003	0·012

ASEMBAGOES ESTATE

Constituents.	Cheribon (Black Java) Cane, planted June 25th, 1905					Seedling No. 247 planted September 20th, 1905.			
	Date of the Analyses								
	14th Feb., 1906.	14th March, 1906.	23rd April, 1906.	14th May, 1906.	13th June, 1906.	14th Feb., 1906.	24th April, 1906.	14th May, 1906.	15th June, 1906.
Brix	14·1	16·30	20·0	20·50	21·20	8·90	12·30	11·90	14·40
Polarization	10·46	13·40	18·17	18·44	17·68	3·60	8·14	7·87	10·20
Purity	74·18	82·14	90·85	89·95	83·39	40·45	66·18	66·13	70·83
Reducing sugars	0·81	1·14	0·62	0·82	—	3·10	3·09	1·64	3·07
Ash	0·825	0·519	0·475	0·426	0·585	0·648	0·522	0·530	0·641
Insoluble ash	0·202	0·079	0·073	0·072	0·083	0·086	0·153	0·070	0·096
Soluble ash	0·623	0·440	0·402	0·354	0·502	0·526	0·369	0·460	0·545
Potash	0·362	0·255	0·219	0·186	0·285	0·341	0·230	0·270	0·326
Carbonic acid in the ash..	0·030	0·028	—	—	0·015	0·022	0·016	0·002	0·017
Sulphuric acid	0·101	0·070	0·181	0·144	0·181	0·101	0·054	0·126	0·125
Chlorine	0·136	0·088	0·003	0·021	0·030	0·126	0·103	0·095	0·103

KENTJONG ESTATE

Constituents.	Cheribon (Black Java) Cane.						Seedling No. 100.					
	Date of the Analyses.											
	15th Feb., 1906.	14th March, 1906.	16th April, 1906.	17th °May, 1906.	15th June, 1906.	14th July, 1906.	14th Feb., 1906.	14th March, 1906.	16th April, 1906.	17th May, 1906.	15th June, 1906.	14th July, 1906.
Brix	11·8	14·20	17·10	17·80	19·40	19·70	12·4	13·1	14·60	19·30	16·90	19·50
Polarization ..	7·44	10·66	15·19	16·18	17·97	18·70	7·75	8·90	11·72	17·29	15·35	17·86
Purity	63·05	75·0	88·83	90·90	92·63	94·92	62·5	67·94	80·72	89·59	90·83	91·59
Reducing sugars	3·58	2·88	1·64	1·28	0·79	0·62	3·84	3·84	2·16	1·22	2·39	0·89
Ash	0·239	0·274	0·266	0·204	0·201	0·189	0·343	0·255	0·204	0·328	0·263	0·311
Insoluble ash ..	0·088	0·072	0·070	0·075	0·089	0·067	0·154	0·056	0·067	0·086	0·076	0·070
Soluble ash ..	0·151	0·202	0·190	0·129	0·112	0·122	0·189	0·199	0·137	0·230	0·187	0·241
Potash	0·082	0·110	0·103	0·062	0·064	0·070	0·092	0·099	0·074	0·128	0·109	0·143
Carbonic acid in the ash ..	0·005	0·010	—	—	0·006	0·011	0·016	0·012	—	—	0·017	0·025
Sulphuric acid ..	0·050	0·056	0·088	0·053	0·042	0·041	0·048	0·048	0·055	0·095	0·053	0·067
Chlorine ..	0·012	0·014	0·002	0·003	—	—	0·015	0·020	0·008	0·010	0·011	0·008

Influence of Milling Power on Inorganic Substances.—The power of the mills in which the cane is crushed has a very marked influence on the ash content of the juice, inasmuch as the juice extracted by the heaviest crushing contains more, both soluble (potash ash) and insoluble (silicate ash) than juice which is extracted by light crushing. The following analyses of juice extracted in a triple crushing mill distinctly show the difference. The juice was obtained by triple crushing without maceration, and, in order to have comparable figures, the percentage of the different constituents is calculated to 100 parts of juice of the same density as the first-mill juice.

Constituents.	I.	II.	III.	I.	II.	III.	I.	II.	III.
Ash (total)	0·247	0·360	0·376	0·202	0·251	0·418	0·602	0·567	0·661
Soluble ash	0·136	0·171	2·233	0·101	0·120	0·131	0·466	0·445	0·493
Insoluble ash ..	0·111	0·189	0·143	0·101	0·131	0·247	0·136	0·122	0·168
Potash	0·080	0·097	0·133	0·057	0·069	0·098	0·265	0·282	0·280
Sulphuric acid ..	0·029	0·041	0·049	0·025	0·030	0·042	0·128	0·099	0·147
Chlorine	0·027	0·034	0·045	0·018	0·024	0·033	0·054	0·025	0·056
Carbonic acid in soluble ash ..	0·004	0·002	0·007	0·001	0·001	0·002	0·022	0·028	0·015

Varying Mineral Constituents of Juice.—Different cane varieties can absorb varying amounts of inorganic matter from the soil of the same field. The author analysed the juice of Muntok and Canne Morte canes which were planted in the same field as Cheribon cane, and in almost every case found them to contain more ash than the latter, although grown in the same field and under the same conditions.

Constituents.	Kemanglen Estate.		Klampok Estate.		Kalimati Estate.		Wono-pringgo Estate.		Modjopangoeng Estate.				Remboen Estate.	
									Kentos Field.		Pakoentjen Field.			
	Muntok.	Cheribon.	Muntok.	Cheribon.	Muntok.	Cheribon.	Muntok.	Cheribon.	Fiji.	Cheri-bon.	Fiji.	Cheri-bon.	Fiji.	Cheribon.
Ash	0·262	0·240	0·360	0·252	0·180	0·084	0·174	0·151	0·311	0·165	0·260	0·167	0·120	0·130
Soluble ash	0·192	0·200	0·311	0·194	0·090	0·042	0·128	0·051	0·224	0·136	0·128	0·084	0·045	0·053
Insoluble ash	0·070	0·040	0·049	0·058	0·090	0·042	0·046	0·100	0·087	0·029	0·132	0·080	0·075	0·077
Potash	0·106	0·110	0·210	0·119	0·051	0·024	0·081	0·034	0·151	0·078	0·088	0·047	0·045	0·044
Sulphuric acid	0·077	0·081	0·090	0·054	0·036	0·018	0·022	0·010	0·001	—	0·017	0·012	0·025	0·034
Chlorine	0·004	0·004	0·005	0·001	0·006	0·00	0·002	0·001	0·068	0·034	0·011	0·017	—	—
Carbonic acid in soluble ash	0·039	0·037	0·003	0·003	—	0·00	0·022	0·009	0·020	0·010	0·012	0·018	0·008	—

So far as we can judge from the limited data at our disposal, it is very probable that in ripe cane a high potash content accompanies a low quotient of purity in the juice and a high percentage of reducing sugar.

As regards the distribution of the mineral substances in a horizontal direction, we may quote an analysis of Bonâme, who found the rind of the cane to contain 0·61 per cent. of ash, against 0·25 per cent. in the pith.

It will be seen that the proportion in which the different constituents of our raw material occur varies within rather wide limits. For ripe, sound, and normal cane, we may state the following limits, which will only be exceeded in certain rare cases :—

Sucrose	11—16
Reducing Sugar	0·4—1·5
Fibre	10—15
Ash	0·5—1·0

SECOND PART

SUGAR MANUFACTURE

CHAPTER I

EXTRACTION OF THE JUICE

I.--General observations

Introduction.—From the most remote times in which the sugar industry has been heard of, crushing has been the mode generally practised for the extraction of the juice.

The simplest operation to which the cane was submitted to this end consisted in squeezing the cane stalks by means of a movable beam. Later the pieces of cane were ground in a mortar by means of a wooden pestle moved by oxen or mules. Very rapidly, however, the industry made great strides, and in every cane-growing country were seen mills first with vertical and later with horizontal rollers of wood or stone. Initially, these were moved by cattle or by water power, but ultimately came the iron mills driven by steam or by explosion motors or by electricity. Although cane mills in later years were constructed of greater power, yet the single pressing which was the universal practice during a very long period allowed too much juice (which means sugar) to remain in the bagasse, and owing to this fact milling proved a very wasteful process of extraction.

In the general condition of the cane sugar industry at that earlier period it could possibly afford these losses, but there came a time when so considerable a waste of sugar could no longer be allowed. The European beet sugar industry dumped ever-increasing quantities of sugar on the markets and thereby was the cause of the great financial sugar crisis of 1883, which endangered the very existence of the cane sugar industry.

The sugar manufacturers in the colonies realized that something would have to be done to improve both cultural and technical methods if they could hope for a survival of their estates in this relentless competition.

It was evident that among the methods coming into consideration for improvement, the obviously wasteful milling process must take prior place. It was inevitable that the example of the beet sugar industry, in which the diffusion process had triumphed so rapidly over the previously used pressing process

and had so greatly reduced the loss of sugar in extraction, should attract the attention of the cane sugar manufacturers. In 1885 experiments were made with the diffusion of cane both in Spain and in Java, and resulted in a much greater extraction being obtained than by the milling plants then in use. Although these experiments showed that the calorific value of the diffusion bagasse was far below that of the bagasse from the mills, the advantage of the much higher extraction induced manufacturers in Java, Cuba, Egypt, Louisiana and elsewhere to erect diffusion plants, which all gave very satisfactory results as regards the extraction of the juice.

The fuel question, however, remained the great problem in this process, since the very fine bagasse resulting from the crushing of the cane chips offered a great resistance to its complete combustion and necessitated far more extra fuel than with the ordinary milling bagasse. Moreover, the constructors of mills did not remain idle but steadily improved the efficiency of the milling plants. The application of multiple crushing, of maceration with water and with last-mill juice, of grooved rollers, of preparatory devices, etc., so improved the extraction of juice by mills, that it not only equalled the achievements of diffusion but even surpassed them, while leaving the calorific value of the bagasse undiminished. The consequences of this brilliant work have been that, save in a few places, where very exceptional circumstances as to fuel supply exist, diffusion in all its different forms, including the lixiviation of the bagasse, has been abandoned everywhere and the milling process is now the only one universally employed by the cane sugar industry.

II.—Milling Plants

Three-Roller Mills.—The most common form of the mill is the three-roller type or else combinations of the three-roller mill. Two of the rollers on the same level turn in the same direction and bear the names of " feed roller " and " bagasse roller " respectively, while the third, the " top roller," is placed above the other two and turns in the opposite direction.

Distance apart of Rollers.—As a rule the rollers of the first mill are set in such a way that the cane, either treated in preparatory machines or not, enters easily and is but slightly squeezed ; but the space between the top and the bagasse rollers is so small that only the well-crushed fibre can pass and it is here that the real crushing takes place. Care must however be taken that the forward motion of the crushed bagasse is not hampered by too small an outlet between the top and bagasse rollers. The distance between the top roller and the feed roller and between the top roller and the bagasse roller of the second mill in a train is smaller than with the rolls of the first one, and is still

smaller in the third and further mills of the train in order to obtain the maximum extraction.

The Bedplate.—The extracted juice streams from the rollers on to the bedplate of the mills, which takes the form of a collecting trough with a perforated cover to keep back the coarser particles of cane and allow the juice to pass; thence it is conveyed through open gutters to the measuring or weighing tanks.

The Bagasse Turner.—In order to carry the cane or the bagasse from the feed roller to the bagasse roller, a curved metal plate, called the trash or bagasse turner, is fixed under the top roller, but is capable of adjustment. The accurate adjustment of this plate is one of the principal points in mill-work and like the adjustment of the rollers requires constant attention on the part of the engineer. It would be out of place to enter into details of the subject* in the present work which is mainly addressed to chemists, but the following remarks may be offered.

Adjustment of the Rollers.—The proper adjustment of the rollers, i.e., the space between the feed roller and the bagasse roller depends entirely on the amount of cane that has to be worked up in twenty-four hours with a given velocity of the mill, and on the quantity of juice that is allowed to remain in the bagasse according to the capacity of the later units in the train. The relation between those two spaces is not constant for different cane varieties but depends on the toughness of the fibre. The position and the curve of the trash-turner exert a great influence on the efficiency of the mill-work; but at present we do not know with sufficient accuracy how these should be modified to suit different circumstances.

In practice the tendency is to reduce the space between the bagasse turner and the top roller as much as possible, without the risk of jamming the cane fibre between them. The space between the bagasse turner and the bagasse roller depends on the position of the mill in a combination of similar mills. It is larger in a first mill where only cane is crushed than in the second, third or fourth where the already finely divided bagasse has to be more heavily pressed.

Speed of Rollers.—No definite figures can be given as to the surface-speed of the rollers, but this should be as low as the required output will permit. The surface of the roller, whether rough or smooth, has also a great influence in this connection.

* This subject is admirably treated by Noël Deerr on page 182 of his work, "Cane Sugar."

Grooved Rollers.—Formerly mill rollers were smooth or only superficially grooved or indented, but about the year 1906 several types of top rollers came into use, in which very deep patterns were made to project.

In some cases the patterns were diamond-shaped or took a herring-bone or cup-like form ; certainly a great variety of more or less deep carvings were made in mill rollers by different constructors, to be used not only in the top roller of the first mill, but also in that of the other units of the same tandem. The aim was not only to have a better grip on the cane or on the bagasse, but also to increase the capacity. It is clear that the juice extraction was not increased by the deep serrating of the top roller; but, owing to the larger surface of the roller caused by the grooves and projections, the mill capacity became greater than with smooth or superficially-grooved rollers of a similar size.

Messchaert Grooves.—Another device of grooved rollers, invented by Messchaert* and used for the first time in 1914 in the Oahu Sugar Co.'s mill at Waipahu, Hawaii, claims to increase the extraction considerably. It is said to render possible a 2 per cent. higher extraction ; a 10-15 per cent. greater mill capacity ; an improved feeding of the mills ; less wear on the rollers ; a closer setting ; less power consumption ; and a drier bagasse than in the case of the ordinary ungrooved roller. Messchaert gives the following account of the discovery of his invention : In mills working badly, it is noticeable that the bagasse shows wet in spots, owing to an accumulation of juice in a break of the blanket as it passes between the top and feed rollers, which break is not filled up on the returner bar. Consequently the blanket is thinner and breaks at that place when it passes the bagasse roller. If the top roller is very smooth in places, the break of the blanket will be large on account of the greater slip. The quantity of juice accumulated at that spot will be shot out at the first relief of pressure. From this it was deduced that the fault does not rest with the trash-plate, as was commonly supposed. It seemed that the remedy would have to be applied at the point of the greatest pressure, viz., at the point of contact between the top and feed rollers. By putting in 15 circumferential grooves, $1\frac{3}{4}$ in. deep and $\frac{3}{8}$ in. wide, on the feed roller of the 4th mill, the success of the idea was fully demonstrated.

The explanation of the working of the grooves is that when these are absent, the juice is impelled upwards, forming thus a little pool on the top of the feed roller. The bagasse feeding into the mill has to force its way down through this pool against the considerable pressure of the stream operating against it. But with the grooved feed roller no pool forms and the juice escapes where the pressure is greatest, thus clearing the way for the bagasse blanket to enter

* "Int. Sugar Journal," 1914, 483.

between the rollers. With grooved rollers all the juice flows down the front and back of the roller and practically none of the bagasse enters the grooves because of its natural resiliency. Scrapers may, however, be used to prevent the grooves from clogging.

With the adoption of the grooved rollers, some trouble has been experienced with the increased amount of trash dropping into the juice pan through the grooves. James Ogg has eliminated this difficulty by means of a special scraper, through which the trash is carried across the returner bar with the bagasse. By the use of this apparatus it is claimed that the ordinary scrapers may be dispensed with, so that the juice pan is kept practically free from trash.

Subsequently many factories put in heavy and large grooved rollers in the first mill, and likewise juice grooves in the discharge rollers, and claimed to increase considerably the sugar extraction by those devices.

The exhausted bagasse passes out of the mill on the top of the bagasse roller and gravitates down an inclined plate which is adjusted as near to the bagasse roller as possible. It is here received upon an intermediate carrier which elevates and discharges it upon a second inclined plate, whence it enters the second or third mill. The bagasse from the last mill falls on to a bagasse-carrier, or into waggons or baskets by means of which it is transported to the furnaces, where it may be used as fuel without drying it beforehand.

Mills with Multiple Rollers.—Apart from three-roller mills, mills with two rollers are also used, the rolls being placed one above the other and the cane introduced between them by means of a carrier. Moreover, we find mills having 4, 5 or 8 rollers, but, as already stated, the three-roller mill is the most common type. Mills with 9, 12 or 14 rollers are merely combinations of three-roller mills placed in tandem or combined with a preliminary cane crusher, having only two rollers. These compound mills are placed very close together and connected by means of intermediate carriers, the whole plant being driven by one or two engines through multiple gearing.

Shredders and Crushers.—Special appliances have been adopted in order to relieve the mills and enable them to grind more cane in a given time with a given power, also for the purpose of disintegrating the whole cane, so that the bagasse passing out of the first mill may be sufficiently loose and finely divided to permit of its being thoroughly moistened by the maceration water which is applied at this stage.

Revolving Knives.—These appliances range from very simple types to rather intricate apparatus. In order to feed the mills regularly the canes are thrown on to long carriers, upon which they are spread out by hand before they reach the mill. In case of fallen canes, which are usually crooked and therefore

difficult to spread out, a spindle provided with revolving knives is sometimes adjusted across the carrier, so that the canes passing under them are cut into short lengths and more evenly distributed.

In the Hawaiian Islands much more powerful slicing machines have been constructed which cut the cane on the carrier into very fine particles. These knives have also the advantage of detecting pieces of metal which might be present among the canes and would damage the rollers if they passed into the mill. Provided that the knives are kept sharp, the power used by this contrivance is not great.

Cane Cutters and Shredders.—Other appliances placed between the carrier and the first mill include the Ross Cane Cutter and the Cane Shredder. The former cuts the cane in pieces about four inches long and afterwards divides it into thin slices, while the latter reduces the cane to fibre between two rows of teeth before it enters the mill. If the knives of the Cutter and the teeth of the Shredder are sufficiently sharp, this subdivision can be effected without any loss of juice; this cane is afterwards crushed in the same way as the canes themselves, by mills, the rollers of which must be further apart than in the case of whole canes, because they receive the feed in a more voluminous form.

The Searby Shredder.—In the Searby Shredder* which is extensively used in the Hawaiian Islands, the cane, having passed through a crusher where it has been crushed and flattened, drops on a table along which it is carried to a thrashing or fibre-forming device. In this element of the system, angle anvil bars, spaced somewhat apart, form a sort of open cage or grating. A rotating member carrying pivoted beater arms co-operates with these angle bars to disintegrate the cane. The fibrous material formed by this action from the cane drops through the spaces between the bars and is delivered by a conveyor to an ordinary type of three-roller mill or series of such mills.

This instrument disintegrates the cane very powerfully and also renders the hard parts of the cane open to the action of maceration water, so that a full extraction is possible with a comparatively small amount of maceration water.

It appears, however, that the bagasse obtained by the crushing of the so finely divided fibre has the same drawback as that resulting from the diffusion process, viz., that it is not easy to get it completely burned with the highest possible calorific effect.

Crushers.—A third type, the Crusher, consists of a set of rollers provided with prominent teeth, which are placed in such wise that the canes passing through them are slightly squeezed and therefore the first mill can crush them

* "Int. Sugar Journal," 1915, 578.

more easily than it can whole cane. The Crusher extracts a part of the juice and in that respect differs from the other types referred to above. The first crushers were of the Krajewski type with V-shaped teeth, while, since then, the Fulton type with a much more intricate pattern has found much application.

Summary.—Nowadays all modern cane sugar factories are equipped with extraction plants, consisting of one or more preparatory apparatus followed by a number of three-roller mills with smooth or grooved cylinders, which are so adjusted that the uninterrupted mat of cane passing through them is expressed with progressive force and finally leaves the train in the shape of practically exhausted bagasse.

On its way from one unit of the train to the other, the partly expressed bagasse is macerated with water or last-mill juice, or both, in order to reduce the sugar content of the juice ultimately remaining in the final bagasse.

III.—Regulation of the Mill Work

Power of the Different Units.—It is very useful, for a good extraction, to have a plant in which the work done by the different units stands in a good relative proportion. The new plants designed and built by competent constructors fulfil this requirement; but in many cases the milling train consists of units of different types and powers put together haphazardly. In such cases it is advisable not only to examine the work done by the whole tandem, but also that performed by the different units individually. It may be that defective work of one of the units spoils the good effects of the others, and thereby renders the total extraction less thorough than it might otherwise be. It is advisable to use the most powerful mill as first mill, so that it may express 65 per cent. of the weight of the cane, leaving only 25 per cent. of juice in the bagasse, when the fibre content is about 10 per cent. If higher the first mill will not express so much. The following mills express a little more juice from the bagasse, but it is evident that even the heaviest pressure will fail to express all the juice. Owing to the colloidal water present in the fibre, the juice remaining in the bagasse will not have the same concentration as that which is expressed, but as it contains sugar some loss is inevitable.

Maceration.—When water is poured on the bagasse, this residual juice is diluted, and after re-crushing the bagasse to its former content of juice it will then contain the same amount of diluted, and therefore less saccharine juice, causing less loss of sugar, so that maceration considerably improves the juice extraction. Cane that has been crushed once, or in some factories even twice, and thus has lost a great deal of its juice, is macerated at the moment when it emerges from between the roller and (thus relieved from the heavy

pressure) expands by its own elasticity eagerly absorbing water. As the result of the crushing in the mill, the cell walls of the parenchyma have already been torn, allowing the maceration water at once to dilute the juice still remaining. Additional water is poured on the partially exhausted bagasse on its way from one mill to another, and it may be moreover turned over in some factories, the result being that the juice which is expressed by the next mill becomes highly diluted.

The simplest method of applying the maceration water is to spray it on the bagasse by means of a perforated pipe or by Körtings injectors, at the moment when it emerges from between the cylinders, and freed from the pressure is most eager to absorb liquid. Of course in the short time that the bagasse requires to pass from one mill to another, the maceration water cannot mix completely with the juice still remaining in the bagasse, and a mixture of 80 per cent. is considered very satisfactory.

The heavy pressure of the first mill tears and opens the parenchyma cells but is unable to break all those of the rind and the nodes, which become crushed in the later units where the distance between the rollers is so much smaller. By the use of such appliances as cutters, shredders and crushers, it is however possible to obtain the first mill bagasse in such a spongy condition that it readily absorbs the maceration fluids, but, nevertheless, it is quite possible that even the best prepared bagasse may not be completely moistened by the maceration water, thereby unduly decreasing its effect.

In order to remedy this defect, many contrivances have been devised to turn over the bagasse on its way from one mill to the other, either by revolving parts on the carrier or by an inclined shoot, so that it may be moistened on the top side first and then on the bottom side.

Immersion Methods.—As, however, all these methods have only a local effect, it has often been tried to immerse the bagasse totally in water. Years ago a trough full of water was frequently placed between the mills, through which the carrier with the partly expressed bagasse was conveyed, but one never heard of the complete success of this device, the rapid souring of the juice in the trough accounting for its unfavourable operation.

All these systems have the disadvantage that the bagasse coming out of the mill has already had an opportunity of expanding before coming into contact with the water.

This drawback is prevented in Ramsay's Scraper, in which device two heavy metal scrapers bear tangentially on the top and bagasse rollers, and force the bagasse between two plates. Attached to the perforated scrapers are hollow boxes into which water is pumped under a heavy pressure. As soon as the bagasse comes out of the mill it meets the water and expands without contact with the air. The moist bagasse is carried by a rake over a metal plate to the next mill and there expressed.

Hot or Cold Maceration.—There has been much difference of opinion as to the advantages of hot or cold maceration. Hot water has the disadvantage of dissolving more gummy matter from the bagasse and introducing it into the juice, but all the same it is to be preferred from a practical point of view. The cold water used for the purpose in sugar mills is usually dirty and needs filtration; therefore with a view likewise of preventing the choking up of the apertures in the supply pipes or in the sprayers, it is better to use the condensed water from the hot well. As it is an advantage to force the water against the bagasse, the maceration supply is generally connected with the feed pump of the boilers. In this simple way one obtains a steady supply of hot water under considerable pressure.

Some believe that hot water mixes more readily with the juice in the bagasse than does cold, owing to a kind of diffusion which takes place between the juice in the surviving cells of the bagasse and the surrounding hot water, but which is impossible in the case of cold water. But by microscopical inspection Went* showed that all the cells are already broken by the heavy pressure of the first mill, so that, even if we admit a certain amount of diffusion through those ruptured cells, this takes place as easily with cold water as with hot.

Müller von Czernicky† carried out comparative experiments with hot and cold maceration and found no difference, as is seen in the following table:—

	Cold Maceration.	Hot Maceration.
Brix of first-mill juice	17·9	17·7
Brix of mixed juice	15·9	15·9
Brix of last-mill juice	7·2	7·4
Maceration on 100 parts of first-mill juice	11·3	11·0
Sucrose in bagasse	4·33	4·40
Quotient of sucrose in bagasse and Brix last-mill juice	0·60	0·60
Q'tient of sucrose in bagasse and sucrose last-mill juice	0·78	0·78
Quotient of purity first-mill juice	83·1	82·9
Quotient of purity last-mill juice	76·7	76·5

Two further experiments showed that even after clarification no difference in the quotient of purity could be detected.

	Cold Maceration.	Hot Maceration.
Increase in purity of the last-mill juice by clarification	3·7	3·8
	3·9	3·6
Original purity	74·8	74·4
	73·0	72·5

As the risk of dissolving gummy matter in hot water is not very great and as, on the other hand, no better extraction can be expected, it is immaterial

* "Archief voor de Java Suikerindustrie," 1895, 673. † *Ibid.*, 1899, 174.

whether hot or cold maceration be adopted, but from a practical standpoint the author would advise boiler feed-water for this purpose.

Hazewinkel and Langguth Steuerwald* showed that the pentosan content of mill juice obtained by maceration with hot water is not higher than if cold water had been used, proving that hot water does not dissolve more gums from the bagasse than cold.

Maceration with Last-Mill Juice.—In order to make the maceration water serve several times, it has become the custom to use the juice from the third mill for macerating the bagasse between the first and the second mills, and the juice from the fourth for the bagasse of the third mill, while pure water is applied behind the third. These rules are not fixed ones and differ according to the composition of the mill-train, but the general rule is that the heaviest juice serves for the first maceration, the lighter juices for subsequent ones, and water for the last maceration.

This maceration with the last-mill juice can only be applied if the cane is crushed so heavily in the first mill that the first bagasse can easily absorb liquid. A less powerful mill only squeezes the canes, and they emerge with a smooth surface and are unable to absorb the last-mill juice. In this case this added saccharine liquid is again extracted by the second mill without having diluted the juice in the bagasse, or it soaks through the planks of the carrier on to the floor and is consequently lost.

Condition of Bagasse for Maceration.—By the use of such appliances as cutters, shredders, or crushers, it is, however, possible to obtain the first-mill bagasse in such a spongy condition that it eagerly absorbs the last-mill juice and, therefore, after leaving the second mill, contains a more dilute juice than when the maceration with last-mill juice has been omitted, or when that juice has only moistened its surface and has not penetrated into the interior of the layer of bagasse on the carrier. It is evident that these cane-preparing machines not only allow quicker working and decrease the risk of breakdowns, but, for the above reason, also bring about a better juice extraction while maintaining the same dilution.

Advantages of Maceration with Last Juice.—Müller von Czernicky† published the following data on the advantages of maceration with last-mill juice:—

During 14 consecutive days cane was crushed by a set of three three-roller mills, and macerated alternately one day with water and the next day with last-mill juice. Great care was taken to grind about the same quantity of canes every day, and to ensure that the canes were as similar as possible. The fact that he succeeded in both conditions is proved by the uniform composition of the first-mill juice, and the constant fibre content of cane and bagasse.

* "Archief voor de Java Suikerindustrie," 1911, 383
† *Ibid.*, 1899, 170.

Maceration with Water									Maceration with Last-Mill Juice.								
Brix Normal Juice.	Brix. Mixed Juice.	Maceration.	Brix. Last Mill Juice.	Sucrose in Bagasse.	Dry Substance in Bagasse.	Fibre in Bagasse.	Fibre in Cane.	Sucrose lost on 100 cane.	Brix. Normal Juice.	Brix. Mixed Juice.	Maceration.	Brix. Last-Mill Juice.	Sucrose in Bagasse.	Dry Substance in Bagasse.	Fibre in Bagasse.	Fibre in Cane.	Sucrose lost on 100 Cane.
17·7	15·7	12·5	7·16	4·07	55·0	48·0	11·05	0·97	—	—	—	—	—	—	—	—	—
17·3	15·2	14·0	6·54	4·03	54·7	47·5	10·9	0·92	—	—	—	—	—	—	—	—	—
17·5	15·2	15·5	6·35	3·90	55·8	47·3	11·2	0·92	17·4	15·3	13·5	5·69	3·77	55·9	47·4	11·2	0·89
17·3	15·3	12·7	6·50	3·97	56·4	46·6	10·7	0·91	17·5	15·7	11·0	6·38	3·88	56·1	48·3	11·1	0·89
17·7	15·5	13·9	6·51	4·09	55·8	47·5	11·1	0·95	17·6	15·8	11·3	6·40	4·00	55·8	48·4	11·2	0·92
17·6	15·6	12·7	6·49	4·02	56·9	47·8	10·8	0·91	17·6	15·9	10·7	6·52	4·03	56·1	47·6	11·0	0·93
17·5	15·2	15·2	6·37	4·03	56·1	47·6	10·7	0·91	17·3	15·4	12·4	6·37	4·10	56·4	47·8	11·0	0·94
17·6	15·3	15·2	6·40	4·08	54·5	47·2	11·5	0·99	17·4	15·4	13·1	6·27	3·95	55·5	48·1	11·1	0·91
17·7	14·9	18·7	6·32	4·05	55·0	47·4	11·0	0·94	—	—	—	—	—	—	—	—	—
17·5	15·3	14·5	6·51	4·02	55·6	47·4	11·0	0·94	17·5	15·6	12·0	6·27	3·76	55·7	47·6	11·0	0·91

We see that when macerating with last-mill juice, 2·5 per cent. less water causes a smaller loss of sugar in bagasse than with water alone. The water so added is therefore used twice over, viz., once in the form of last-mill juice of about 6·5 per cent. dry substance (in order to dilute the juice of 17·5 per cent.), and the second time in the form of water between the second and third mills to reduce the juice left in the second bagasse to the low density of the last-mill juice.

With the object of obtaining some information in regard to the economics of the extraction of juice by milling, Noël Deerr* made some trials with maceration with cold and warm water and last-mill juice, and different quantities of water alone, of which we give the gist here. The results, which are given below, were obtained by a nine-roller mill and crusher of recent construction, the rollers being of size 34in. × 78in. During the test it was grinding at the rate of 35 to 37 tons of cane per hour. The cane milled was Lahaina, and judging from the uniformity of the samples obtained from the crusher rolls, cane of even quality was milled during the whole series of experiments. The following four methods of applying maceration water were carried out :—

(*a*) Hot water before last mill with return of third-mill juice before second mill.

(*b*) Hot water before last mill with no return of third-mill juice.

(*c*) Cold water before last mill with return of third-mill juice before second mill.

(*d*) Hot water before both second and third mills and no return of diluted juice.

* Bulletin No. 30, Hawaiian Experiment Station.

Two trials were made of the two first-mentioned methods and one of the last two.

As the results of these experiments, it was found that the highest extraction is obtained when a system of compound saturation is followed. The lowest result is obtained when all the water is added before the last mill; and an intermediate result, when the water is divided before the second and the third mills. Between the use of hot water and cold water little difference, if any, is to be found. The very small advantage in favour of hot water is perhaps accounted for by the slightly higher dilution.

		Maceration with Hot Water and Last-Mill Juice. A	Hot Water before the 3rd Mill. B	Maceration with Cold Water and Last-Mill Juice. C	Hot Water before the 2nd and 3rd Mills. D
Analysis of the fibre obtained from the	Cane. Fibre	11·40	11·40	11·40	11·40
	Cane. Sucrose	16·40	16·47	16·31	16·51
	1st Mill. Sucrose	10·09	9·90	9·98	10·20
	1st Mill. Moisture	52·20	53·20	53·11	53·07
	1st Mill. Fibre	36·00	36·00	36·00	36·00
	2nd Mill. Sucrose	6·90	7·94	7·09	7·40
	2nd Mill. Moisture	48·90	47·40	49·45	48·15
	2nd Mill. Fibre	41·00	43·00	40·00	43·00
	3rd Mill. Sucrose	4·37	4·75	4·48	4·60
	3rd Mill. Moisture	49·23	47·96	48·63	48·87
	3rd Mill. Fibre	45·00	45·00	45·00	45·00

	A		B		C		D	
	Brix.	Sucrose.	Brix.	Sucrose.	Brix.	Sucrose.	Brix.	Sucrose.
Juice. Crusher	20·9	19·52	21·1	19·61	20·8	19·42	21·1	19·64
Juice. 1st Mill	20·7	19·12	20·7	19·20	20·9	19·40	21·0	19·24
Juice. 2nd Mill	10·6	8·78	18·1	15·66	8·4	6·90	8·95	7·83
Juice. 3rd Mill	3·9	3·15	3·9	3·16	3·6	2·89	3·7	2·93
Juice. Mixed	15·6	14·30	14·9	13·69	15·9	14·34	15·0	13·78

Dilution	33·9	41·62	30·8	40·6
Crusher and 1st Mill	80·50	81·02	80·63	79·95
2nd Mill	7·83	6·81	5·42	6·97
3rd Mill	4·92	4·89	7·08	6·19
TOTAL	93·25	92·72	93·13	92·94

In a twelve-roller mill, of similar size and construction to the nine-roller mill, a series of trials was made with hot water and cold water, the system of saturation being as follows: Water applied before the fourth mill; the fourth

mill juice was returned in front of the third mill; the third in front of the second; and the second in front of the first on to the canes coming from the crusher, the juice going to the boiling-house being taken from the crusher and from the first mill.

Analysis of the fibre obtained from the		Maceration with Hot Water and return of the different Juices.	Maceration with Cold Water and return of the different Juices.	Maceration with Hot Water before the 3rd and 4th Mills.
Cane.	Fibre	10·40	10·40	10·40
	Sucrose	17·35	17·55	17·72
1st Mill.	Sucrose	11·89	11·87	11·14
	Moisture	56·10	56·07	54·75
	Fibre	30·00	30·00	31·00
2nd Mill.	Sucrose	9·40	9·03	8·23
	Moisture	49·66	50·71	46·75
	Fibre	38·00	38·00	43·00
3rd Mill.	Sucrose	7·14	7·38	5·30
	Moisture	47·18	48·13	49·65
	Fibre	43·00	43·00	44·00
4th Mill.	Sucrose	4·90	4·82	4·40
	Moisture	47·27	47·63	44·38
	Fibre	45·00	45·00	50·00

Juices.	Brix.	Sucrose.	Brix.	Sucrose.	Brix.	Sucrose.
Crusher	22·35	20·77	22·66	20·90	23·20	21·12
1st Mill	19·56	16·44	19·83	16·87	17·80	15·58
2nd Mill	18·30	15·08	18·94	15·66	13·20	11·22
3rd Mill	14·70	11·75	14·35	11·38	10·45	8·40
4th Mill	9·55	7·47	9·73	7·62	7·70	5·57
Mixed	20·00	17·47	20·32	17·88	18·35	15·95

Dilution	11·7	11·5	26·4
Crusher and 1st Mill	76·2	76·6	78·9
2nd Mill	9·0	8·9	9·8
3rd Mill	4·9	4·3	4·1
4th Mill	3·4	3·8	2·0
TOTAL	93·5	93·6	94·8

As with the nine-roller mill, the sugar in the canes was determined by use of a ratio connecting sucrose in cane and sucrose in expressed juice. In this case one of 0·835 was experimentally found. The comparison of the effect of hot water and of cold water leads to the same result as was obtained with the nine-roller mill; namely, that either is equally efficient.

Souring of Last-Mill Juice.—It has been observed many a time that in practical working the quotient of purity of the last-mill juice is found to be much lower than corresponds with the natural decrease mentioned on pages 107 and 129, which fact is ascribed to souring during the course of the juice used for maceration. If this is really the case, it might be that the loss of sucrose by inversion is heavier than the financial advantage arising from the saving of fuel by the maceration with mill juice instead of water.

Van der Linden* admits that some inversion cannot be denied, but believes the great fall in quotient of purity to be more apparent than real and blames the sampling for the phenomenon observed. He quotes an instance where a very large drop in purity had been found in a factory where, owing to circumstances, the mills had not been washed for a whole week.

Date.	Quotient of purity of First-Mill juice.	Quotient of purity of Last-Mill juice.	Difference.
22nd June ..	Mills	washed.	
23rd June ..	84·8	76·3	8·5
24th June ..	85·5	76·1	9·4
25th June ..	85·1	76·5	8·6
26th June ..	85·7	74·9	10·8
27th June ..	87·1	71·7	15·4
28th June ..	86·7	69·4	17·3
29th June ..	86·7	63·7	23·0
30th June ..	85·7	59·7	26·0
1st July ..	Mills	washed.	
2nd July ..	86·2	77·4	8·8
3rd July ..	86·5	76·2	10·3

It was, however, soon found that the suction tank of the maceration juice, from which the samples had been taken, was half full of *Leuconostoc*, which had only locally caused souring and inversion. The bulk of the juice had passed uninjured and without any perceptible inversion.

If, therefore, the necessary cleanliness is observed in the mill work, no souring, inversion, or deterioration of the macerating juices is to be feared and the expenses for fuel must be very low indeed if maceration with last-mill juice is not a financial gain over maceration with pure water only.

Method of Macerating with Last-Mill Juice.—Most factories pass the last-mill juice through a strainer and apply it to the bagasse coming from the first-mill without further treatment. In some cases, viz., with the Thomas and

* "Archief voor de Java Suikerindustrie," 1923, 103.

Petree process, the juice is clarified before being used for maceration. As the unclarified juice always contains cane fibre, the pipes through which it is poured on the bagasse have to possess a large diameter and be provided with wide apertures in order to prevent choking. The juice may be applied by means of an open inclined gutter placed across the bagasse carrier.

Maceration with Sweet-Water.—In factories which employ the carbonatation process the sweet-waters from the presses (containing 1 to 2 per cent. of sucrose) are also employed for maceration, and since they are free from fibre may also be supplied through pipes having fine apertures, or by injectors.

IV.—Results of the Mill Work.

Influence of Cane Fibre on the Extraction.—If it were possible to crush the bagasse of the different cane varieties always to the same juice content, it would be feasible to obtain a constant sucrose content in the bagasse by keeping the density of the last-mill juice constant. The quantity of juice extracted from 100 parts of cane would then only depend on the amount of fibre, and this quantity diminishes for two reasons when the amount of fibre rises. Firstly, cane with much fibre contains a proportionately smaller amount of juice, and, secondly, the quantity of bagasse is increased, and hence also the quantity of juice which it contains and that of the sucrose in it which is to be eliminated.

It is not, however, quite so simple, because the fibre of every variety of cane has its own power to resist pressure. Thus the Java Seedling No. 247 yields a dry bagasse much more readily than No. 100 or the Black Java cane, and in most cases, canes having a high fibre content will yield bagasse, the fibre of which offers but little resistance to pressure. This to some extent compensates for the increased loss of sugar occasioned by the large amount of bagasse obtained from canes of high fibre content.

From the average of a great number of determinations it is seen that a high fibre content of the cane corresponds with a high fibre content of the bagasse, so that a hard cane yields a drier and more exhausted bagasse than a soft one.

Very probably the considerable difference in juice content of bagasse of soft and hard canes, when crushed in the same mill, in a layer of equal thickness, is occasioned by the difference in the absorbing power of the fibre. The fibre of soft cane is more spongy than that of hard cane, and therefore absorbs more juice than the latter. At the moment when the bagasse is relieved from the heaviest pressure, all of the expressed juice has not escaped out of contact

with the bagasse and a film of juice still remains on the rollers, consequently the expanding fibre readily absorbs whatever juice remains in contact with it, although such juice has already been expressed from it. The more spongy the fibre the more it will absorb, and though this is still an unproved hypothesis, it may explain why soft cane yields bagasse containing so much more juice than does hard cane.

Improvements in Mill Work.—Since the mill work has been carefully studied and, in particular, the work to be done by every one of the units of the train has been made the subject of careful investigations, it has become possible to regulate the work in such wise that every cane variety can be crushed to the same exhausted bagasse, so that nowadays the influence of the hardness or softness of the fibre has become immaterial.

Results in Practical Working.—From data supplied by the Mutual Control of the Java Sugar Factories, the figures for the extraction throughout Java during the last 25 years have been as follows :—

Year.	Fibre in cane.	Sugar extraction.	Loss of Sucrose in Bagasse on 100 cane.	Year.	Fibre in cane.	Sugar extraction	Loss of Sucrose in Bagasse on 100 cane.
1899	—	90·3	1·36	1912	12·17	90·8	1·14
1900	10·65	90·1	1·22	1913	12·40	90·7	1·16
1901	10·95	90·2	1·24	1914	12·61	90·4	1·14
1902	10·91	91·0	1·21	1915	13·26	91·9	0·94
1903	11·21	90·6	1·17	1916	13·14	91·1	1·10
1904	11·29	91·4	1·12	1917	13·02	92·3	0·99
1905	11·61	90·4	1·12	1918	12·99	92·1	1·08
1906	11·78	90·9	1·13	1919	13·01	92·0	0·99
1907	12·00	91·2	1·15	1920	12·98	92·5	0·97
1908	12·01	91·0	1·11	1921	12·98	92·5	1·00
1909	12·15	91·0	1·09	1922	13·19	92·9	0·91
1910	12·27	91·2	1·11	1923	13·14	92·2	1·02
1911	12·32	90·6	1·19				

These are averages in which poor and excellent results are combined. We quote here the best among the data for 1923, to give an idea of what may be attained.

Description of the Train.	On 100 Cane:		Maceration on 100 Fibre.	On 100 Bagasse.		Extraction.
	Sucrose.	Fibre.		Sucrose.	Moisture	
Crusher 4 mills	13·4	11·7	150	2·4	44·7	96·0
,, 4 ,,	14·4	11·8	154	2·7	45·9	95·8
,, 4 ,,	14·0	10·1	157	3·1	50·2	95·2
,, 4 ,,	13·6	13·6	139	2·5	46·2	95·1
4 Mills	13·1	12·2	122	2·7	45·7	95·1
Crusher 4 mills	13·2	13·3	143	2·7	45·6	94·7
,, 4 ,,	12·6	13·4	127	2·7	43·4	94·6
,, 4 ,,	12·1	11·9	156	2·7	48·4	94·4
,, 4 ,,	13·1	14·1	125	2·6	47·4	94·4
,, 3 ,,	13·3	12·4	146	3·2	45·2	94·2

Van Heemskerk Duker* mentions the results obtained on a factory in the Hawaiian Islands during a series of years with mill-trains of the following composition :—

Crop.	Equipment.	Per hour, Tons of Cane.	Dilution.	Extraction.
1914	Knives ; Crusher, 72 in ; 12-roller mill, 78 in.	52·94	33·66	95·57
1915	Knives ; Crusher, 72 in. ; Shredder†, 54 in.; 12-roller mill, 78 in.	53·76	35·98	97·32
1916	Knives ; Crusher, 72 in. ; Shredder, 72 in. ; 12-roller mill, 78 in.	55·6	40·27	97·73
1917	Knives ; Crusher, 72 in. ; Shredder, 72 in. ; 12-roller mill, 78 in.	59·6	35·18	98·56
1918	Knives ; Crusher, 78 in. ; Shredder, 72 in. ; 12-roller mill, 78 in.	55·37	39·18	98·47
1919	Knives ; Crusher, 78 in. ; Shredder, 72 in. ; 12-roller mill ..	48·25	50·64	98·99
1920	Knives ; Crusher, 78 in. ; Shredder, 72 in. ; 12-roller mill, 78 in.	58·08	43·93	98·92
1921	Knives ; Crusher, 78 in. ; Shredder, 72 in. ; 19-roller mill, 78 in.	67·27	36·6	99·07

* Report of Assoc. of Hawaiian Sugar Technologists, 1922.
† Searby type.

Westley* stated with reference to the mills in the Hawaiian Islands, that the improvement was, to a great extent, due to additions made to the milling equipment of several of the mills, but many mills with old equipment showed improvement in extraction. Better milling results than for the previous crop were reported by 64 per cent. of the factories. One gave an average extraction of over 99 per cent. The average for all was 97·45 per cent. as against 97·30 per cent. in 1919. The milling loss, that is the sucrose lost per 100 of fibre in the bagasse, was 2·75 per cent., and the water content was 41·05 per cent.

With a 15-roller mill and crusher equipped with Searby Shredder, an extraction was obtained of 98·98 per cent., while with a nine-roller mill and crusher only 98·13 per cent. could be obtained.

We give here maximum figures for extraction of a number of Cuban mills for 1923.

Sucrose in Cane.	Extraction.	Loss of Sucrose in Bagasse on 100 Cane.
11·98 ..	96·78 ..	0·38
13·74 ..	94·91 ..	—
14·03 ..	94·68 ..	—
14·25 ..	94·18 ..	0·83
13·93 ..	94·14 ..	0·83
13·81 ..	94·08 ..	0·81
14·33 ..	93·92 ..	—
13·79 ..	93·89 ..	0·84
13·51 ..	93·87 ..	0·83
14·55 ..	93·69 ..	0·92

Conclusions.—These figures show that in every country every kind of cane may be practically completely exhausted by means of a powerful mill-train, equipped with preparatory machinery and a rational maceration. The results obtainable do not only attain to those witnessed with the diffusion of beetroot, but in many instances even exceed them.

Since the bagasse obtained by the milling process is immediately fit for fuel and does not need to be expressed first, as is the case in the diffusion and lixiviation processes; since mill extraction only takes a few minutes, and does not allow time or opportunity either for visible or invisible, for mechanical or for fermentative losses; since mill work is very elastic and may at any moment start and end with its full capacity and without delay—the milling process has proved its superiority over that of the diffusion of cane and is now the universal one in practice.

* "Int. Sugar Journal," 1921, 574.

V.—Composition of the Juice

Character of the Juice.—Cane juice, as it comes from the mills, is an opaque liquid, covered with froth, owing to the imprisoned air bubbles it contains, and having a coloration varying from grey to dark green according to the colour of the cane from which it has been expressed. It contains in solution all the soluble constituents of the cane—sucrose, reducing sugar, salts, organic acids, etc., and besides the air already mentioned it carries in suspension gums, fine bagasse, fibre, wax, sand and clay (adhering to the cane), colouring matter, and albumin, which latter body occurs in considerable quantity whenever unripe canes or green cane tops have been crushed with the ripe canes.

Fresh mill juice has an acid reaction, and, owing to the presence of albumin and especially gummy matter, is a viscous liquid that cannot be filtered cold, hence the suspended constituents cannot be removed without steaming, and we have therefore to be content with keeping back the larger particles of bagasse by means of copper strainers.

Proportion in which Constituents occur in Juice.—We have already seen from the data given in Chapter II, Part I, that the proportion in which the chief constituents occur in the juice varies greatly, and depends, among other things, on the variety and age of the cane, the nature of the soil, the manures applied, the climate, weather, etc. In the main, the figures for sucrose and reducing sugar show the greatest differences, while the other elements do not vary much, at least in sound and ripe canes. In unripe cane the juice sometimes contains much dissolved albumin, which forms a thick layer of froth in the measuring tanks, and as this froth remains on the top of the liquid for a long time it hinders the measuring of the juice. Later on, at the clarification stage, this albumin coagulates and is easily removed in the scums. A quantity of this froth was removed from the measuring vessels and gutters, washed, dried, and analysed, with the following results :—

Cane Fibre	62·50
Wax	4·12
Albumin	26·25
Moisture	7·13
	100·00

In dead or burnt cane, the amount of free acid is larger than ordinarily occurs, but on the whole the other constituents do not show any considerable deviations.

Influence of Mill Power on the Proportion.—Apart from the difference between the juice of different kinds of cane, the same cane may yield juice of a varying composition, according to the pressure it has undergone in the mill.

This phenomenon is easily explained when one remembers how many tissues having different degrees of hardness and containing different kinds of sap are present in the cane.

Different Kinds of Sap.—The sap in the vacuoles contains nothing but sucrose, reducing sugar, a little salt, and organic acids. The protoplasm consists for the most part of water, which contains albumin and salts; and finally the sap in the fibrovascular bundles also holds salts in solution but no sugar. We find therefore not less than three different kinds of sap in the cane, viz., of the vacuoles, of the protoplasm, and of the fibrovascular bundles. In the first and the last the constituents occur only in solution, whereas that of the protoplasm contains suspended matter also. The softest tissues contain the purest and richest sap, so that this is expressed by the gentlest pressure. With a heavier pressure this juice is mixed with that of the protoplasm, whilst the fibrovascular bundles part with their watery juice only under the heaviest pressure. Hot maceration causes the soft parenchyma cells to swell, and during recrushing a quantity of finely divided cellular matter passes into the juice.

Moreover the hard, woody rind is only broken by the heaviest pressure, hence the second- and third-mill juices are much darker in colour than the first. The only thing that seems improbable is that the first-mill juice is richer in glucose than the later ones, but this is ascribed to the top of the cane being the softest part and also containing the most glucose. Gentle pressure therefore expresses this easily and imparts to the first juice proportionally more glucose than to the other juices, which are mainly derived from the older tissues of the cane.

Composition of Mill Juices.—The following analyses illustrate the differences in the juices obtained by three successive crushings of the same canes with and without maceration.

I.—TRIPLE CRUSHING, WITHOUT MACERATION.

Black Java Cane.

Constituents.	First Mill.	Second Mill.	Third Mill.
Brix	19·2	19·3	19·0
Sucrose	16·49	16·33	15·95
Reducing sugar	1·98	1·57	1·52
Ash	0·28	0·41	0·42
Gum and pectin	0·125	0·376	1·250
Albumin	0·025	0·092	0·054
Free acids	0·048	0·072	0·096
Quotient of purity	85·9	84·4	84·0
Glucose ratio	12·1	9·6	9·5
Colour	light.	dark	very dark

II.—TRIPLE CRUSHING, WITH MACERATION WATER ADDED AFTER THE SECOND, AND LAST-MILL JUICE ADDED AFTER THE FIRST MILL.

Borneo Cane.

Constituents.	First Mill.	Second Mill.		Third Mill.	
		Diluted Juice.	Calculated on Brix = 18·5	Diluted Juice.	Calculated on Brix = 18·5
Brix	18·5	14·8	18·5	6·7	18·5
Polarization	16·76	12·65	15·81	5·50	15·18
Purity	90·59	85·47	85·47	82·10	82·10
Reducing sugar	0·89	0·59	0·74	0·33	0·90
Glucose ratio	5·3	4·7	4·7	6·0	6·0
Ash	0·244	0·425	0·531	0·199	0·550
Free acids as SO_3	0·042	0·039	0·048	0·016	0·044
Combined organic acids as SO_3	0·077	0·080	0·100	0·027	0·073
Albuminoids	0·024	0·068	0·085	0·047	0·138
Gums	0·063	0·150	0·185	0·099	0·273

III.—TRIPLE CRUSHING, WITHOUT MACERATION.*

Purple Cane. Second Year Ratoons.

	Per cent.
Extraction, First Mill	64·50
,, Second Mill	5·50
,, Third Mill	2·13
Total Extraction	72·13

	First Mill.	Second Mill.	Third Mill.
Brix	15·36	14·60	14·60
Sucrose	12·93	11·41	11·30
Reducing sugar	1·54	1·29	1·23
Ash	0·37	0·58	0·77
Albuminoids	0·18	0·50	0·58
Free acids	0·10	0·11	0·14
Combined acids	0·14	0·15	0·12
Gums	0·10	0·56	0·51
Coefficient of purity	84·07	78·15	77·39
Glucose ratio	11·91	11·30	10·88

* Bulletin No. 91, Louisiana Exp. Station, 31.

IV.—TRIPLE CRUSHING, WITH STEAM MACERATION.*

Purple Cane.—First Year's Ratoon.

	Per cent.
Extraction, First Mill	64·31
,, Second Mill	8·00
,, Third Mill	4·52
Total Extraction ..	76·83

Constituents.	First Mill.	Second Mill.		Third Mill.	
		Diluted Juice.	Calculated to Brix = 15·84	Diluted Juice.	Calculated to Brix = 15·84.
Brix	15·84	12·05	15·84	11·53	15·84
Sucrose	13·50	9·40	12·31	8·70	12·01
Reducing sugar ..	1·37	0·97	1·27	0·88	1·21
Ash	0·37	0·57	0·75	0·65	0·90
Albumin ..	0·14	0·28	0·37	0·20	0·28
Free acids ..	0·09	0·13	0·17	0·14	0·19
Combined acids ..	0·13	0·16	0·21	0·19	0·27
Gums	0·24	0·54	0·70	0·83	1·14
Purity	85·23	78·00	78·00	75·45	75·45
Glucose ratio ..	10·01	10·32	10·32	9·42	9·42

V.—TRIPLE CRUSHING, WITH COLD MACERATION 20 PER CENT.†

Purple Cane.

	Per cent.
Extraction, First Mill	66·50
,, Second Mill	12·15
,, Third Mill	4·51
Total extraction ..	83·16

*Bulletin No. 91, Louisiana Exp. Station, 31.
† *Ibid.*, 32.

Constituents.	First Mill.	Second Mill.		Third Mill.	
		Diluted Juice.	Calculated to Brix = 15·73.	Diluted Juice.	Calculated to Brix = 15·73.
Brix	15·73	10·48	15·73	6·93	15·73
Sucrose	14·01	8·65	12·97	5·64	12·80
Reducing sugar ..	0·83	0·46	0·69	0·34	0·78
Ash	0·35	0·37	0·55	0·31	0·71
Albumin ..	0·12	0·22	0·33	0·21	0·48
Free acids ..	0·08	0·07	0·10	0·06	0·13
Combined acids ..	0·10	0·09	0·13	0·08	0·18
Gums	0·24	0·62	0·93	0·29	0·66
Purity	89·07	82·53	82·53	81·38	81·38
Glucose ratio ..	5·92	5·32	5·32	6·03	6·03

Noël Deerr* made a series of experiments at crushing cane in a two-manual hand mill. The mill was a three-roller one with rollers 5 in. × 4 in., and had the rollers adjusted by set screws passing through the headstocks. The mill was then essentially a rigid mill; and, as the experiment showed, a "crushing" was obtained, which, judged by the fibre content of the bagasse, was comparable in every way with that obtained by large mills on the factory scale. Indeed, the bagasse finally obtained was more disintegrated and pulverized than that obtained in most mills. In conducting the experiments detailed below, one kilogram of cane split into strips was passed three times through the mill, increasing the thickness of the feed at each operation. After the third crushing, when about 65 per cent. of the juice had been obtained, about 100 grams of water was distributed over the bagasse which was then again crushed. This operation was done in all five times, the expressed diluted juice being collected separately at each crushing. Finally, the bagasse was weighed and analysed. In this way eight fractions of juice were collected, the analysis of each fraction being as below:—

* Bulletin No. 30, Hawaiian Exp. Station, page 25.

Fraction.	Weight per cent. Cane.	Sucrose obtained per 100 Sucrose in Cane.	Total Solids per cent.	Sucrose per cent.	Purity.	Value of $\frac{s\,(j-m)}{j\,(s-m)} \times 100$	Product of Cols. 2 and 4 ÷ 100
1	34·7	43·2	19·10	17·71	92·7	95·5	41·3
2	16·0	19·1	19·20	17·23	89·7	92·6	17·8
3	13·0	15·1	19·04	16·70	87·7	90·4	13·8
4	9·8	8·0	13·22	11·64	88·0	90·7	7·3
5	11·3	4 7	7·22	6·01	83·2	85·3	4·0
6	10·0	3·0	5·50	4·44	80·8	82·3	2·5
7	12·0	2·6	4·00	3·16	79·0	79·9	2·2
8	11·6	1·7	2·86	2·17	75·9	75·7	1·3
Bagasse	28·0	2·6	2·05	1·33	65·0	57·3	1·5

Referring to the above table, which represents the mean result of a series of experiments, the decreasing purity of each successive fraction of juice is well shown, except at the fourth fraction where an increase over the third is noticed. This increase, obtained in all the experiments, is not due to accident or error. The third fraction was obtained under very heavy pressure and probably contained much rind tissue juice. The fourth fraction was the first obtained by adding water to the bagasse, and probably consisted largely of pith tissue juice (which had remained unexpressed), to the exclusion of rind tissue juice, as the rind had not yet been sufficiently broken up to take up its proportion of water.

As the purity decreases, so also does the amount of available sugar contained in a quantity of juice. The percentage of available sugar in each fraction has been calculated from the formula :—

$$\text{Available Sugar} = \frac{s\,(j-m)}{j\,(s-m)}$$

where s is the purity of the sugar, j that of the juice, and m that of the molasses, giving to s the value 0·975, m the value 0·45, and taking for j the observed purities ; hence the product of the extraction into the corresponding value of the expression $\frac{j\,(s-m)}{s\,(j-m)}$ gives the amount of available sugar obtained at that operation. In the table reproduced above is given the extraction at each operation, the value of $\frac{s\,(j-m)}{j\,(s-m)}$ and the product of these two quantities, or the amount of sugar available at each operation per 100 sugar in cane. Referring to that table, it appears that of the 43·2 parts of sugar

per 100 sugar in cane obtained at the first operation, 41·3 are available; of the 4·7 parts obtained at the fifth operation, 4·0 are available; and of the 2·6 parts left in the bagasse, only 1·5 is available.

Since the purity of each successive fraction of juice obtained decreases, the earlier expressed portions have a higher available sugar value than the later ones and 43·2 parts of sugar per 100 sugar in cane correspond to more than 43·2 parts of available sugar per 100 of available sugar in cane. Accordingly, Noël Deerr has calculated the combined purities of the first and second, of the first, second and third fractions, and so on, the corresponding values of $\frac{s\,(j-m)}{j\,(s-m)}$, the available sugar obtained at each extraction, and its percentage on the whole available sugar of the cane. For this last quantity he proposed the term "available extraction," i.e., 93·1 parts of sugar per 100 sugar in cane have been extracted, 94·4 parts of the available sugar in the cane have been obtained, and this figure he thought more nearly represented the factor controlling the economic work of the mill. These values are given in the next table:—

Extraction.	Purity.	Value of $\frac{s\,(j-m)}{j\,(s-m)} \times 100$	Available extraction.
43·2	92·7	95·5	45·0
62·3	91·9	94·7	64·4
77·4	91·1	94·0	79·5
85·4	90·8	93·7	87·4
90·1	90·4	93·2	91·8
93·1	90·1	92·9	94·4
95·7	89·8	92·6	96·9
97·4	89·5	92·3	98·4
100·0	88·9	91·7	100·0

The results already given afford a means of answering a question often put, namely, if the extraction is increased from, say, 93·1, which represents average work with a nine-roller mill, what gain can be expected from an extraction of 95·7, which might be obtained with a twelve-roller mill? According to results tabulated above, the economic extraction at these points is 94·4 and 96·9, so that the percentage increase in sugar in bags will be 2·64 per cent. and not 2·79 per cent as calculated from the extraction alone.

The decrease in purity is caused by an increase of gums, albuminoids, acids, and ash in the second and third juices. In the experiment with steam macera-

tion, a jet of steam was introduced through the trash plates between the rollers of the second and third mills. The steam coagulated the albuminoids, so that less of these was removed than in the process of dry extraction ; on the other hand, the percentage of gums in the juice was very much increased, so that the resulting juice was very difficult to clarify.

Hazewinkel and Langguth Steuerwald* showed that first-mill juice does not contain pentosans, but that these bodies pass over into the juice after a more powerful crushing. In an experiment in which the first-mill juice was quite free from pentosans, they found in the mixed juice from the second and third mill, 0·15 per cent. of this constituent, and in the total mixed juice 0·07 per cent.

It has been proved that a very powerful crushing and ample maceration may result in an extraction of 99 per cent. of the total sucrose content of the cane ; and that, notwithstanding, in such a case the purity of the last-mill juice remains far above that of the final molasses. Although the last mill causes many impurities to pass over in the juice, the advantage of the further extraction is maintained, and the opinion often pronounced that the final juice only serves to increase the output of molasses and not of sugar is an erroneous one. We are fully convinced that the mill work may even lead to a complete extraction of the sucrose without fear of a too great increase of objectionable non-sugar expressed from the cane.

VI.—Composition and Value of Bagasse as Fuel

Composition of Bagasse.—The bagasse (or megass as it is also called) coming from the mills contains an average of 46·5 per cent. fibre, 4·5 per cent. sucrose and 47·5 per cent. moisture, though, as observed in a previous chapter, higher or lower values may occur, depending on the quality of the crushing and the hardness of the cane fibre.

Use of Bagasse for Fuel.—Bagasse is generally used as fuel and in most cases it is sufficient to furnish all the steam required for driving the machinery and evaporating the juice in a modern factory turning out refining crystals. In the case of defective furnaces, or when making white sugar which requires more manipulation and consequently more steam, the bagasse is not sufficient and has to be supplemented by additional fuel, *e.g.*, cane trash, firewood, liquid fuel or coal.

* "Archief voor de Java Sulkerindustrie," 1911, 320.

It is evident that the fibre content of the cane and the density of the juice also come into account, as a high fibre content of the cane furnishes more bagasse than a low one, while juice having a high density will, as a rule, get more diluted by maceration than a poor one, which does not justify the use of much maceration water.

Besides the quantity of bagasse yielded by 100 parts of cane, its physical character also influences its calorific value.

Calculation of Calorific Value.—This latter can be calculated from the composition of any sample of bagasse and the calorific values of its constituents mentioned in Part I after the following formula :—

$$C\ V = 8550\ F + 7119\ S + 6750\ G - 972\ W : 100$$

in which C V = calorific value in British Thermal Units for 1 lb. of bagasse, containing F per cent. fibre, S per cent. sucrose, G per cent. reducing sugar, and W per cent. water.

The calorific values of three different samples of bagasse were as follows (ash is put down as 3 per cent.) :—

$F = 50 \quad S + G = 4{\cdot}5 \quad W = 42{\cdot}5 \quad C\ V = 4275 + 320{\cdot}3 - 412{\cdot}7 = 4182{\cdot}6$
$F = 40 \quad S + G = 6{\cdot}0 \quad W = 51{\cdot}0 \quad C\ V = 3420 + 427{\cdot}1 - 495{\cdot}7 = 3351{\cdot}4$
$F = 33{\cdot}3 \quad S + G = 7{\cdot}0 \quad W = 56{\cdot}7 \quad C\ V = 2850 + 498{\cdot}3 - 551{\cdot}0 = 2797{\cdot}3$

An average Java bagasse, having 46·5 per cent. fibre, 4·50 per cent. sucrose, 0·5 per cent. glucose, and 47·5 per cent. moisture, possesses this calorific value :—

$$\frac{46{\cdot}5 \times 8550}{100} + \frac{4{\cdot}5 \times 7119}{100} + \frac{0{\cdot}5 \times 6750}{100} - \frac{47{\cdot}5 \times 972}{100} =$$

$$3975{\cdot}7 + 320{\cdot}3 + 33{\cdot}7 - 461{\cdot}7 = 3868 \text{ B.T.U.}$$

Bolk* determined the chemical constitution and the calorific value of a few samples of bagasse, and found the concordance between the values calculated and determined to be entirely satisfactory.

I.	Dry substance	57·25
	Purity of residual juice ..	60·40
	Sucrose in the bagasse ..	7·175

Therefore crude fibre in bagasse: $57{\cdot}25 - \frac{7{\cdot}175}{60{\cdot}40} \times 100 = 45{\cdot}37$

Ash in bagasse		1·146
Ash-free fibre ..	45·37 − 1·15 =	44·27

* Annual Report of the Technical Section of the Java Sugar Exp. Station, 1914, 69.

Calculated calorific value :—

$44{\cdot}27 \times 85{\cdot}50 + 7{\cdot}175 \times 71{\cdot}19 - 42{\cdot}75 \times 9{\cdot}72 = 3868$ B.T.U.

Determined in the bomb calorimeter 3879 ,,

II. Dry substance .. .. 40·875

Purity residual juice .. 69·51

Sucrose in the bagasse .. .. 6·00

Therefore crude fibre in the bagasse : $40{\cdot}875 - \frac{6}{69{\cdot}51} \times 100 = 32{\cdot}24$

Ash in the bagasse .. .. 1·76

Ash-free fibre .. $32{\cdot}24 - 1{\cdot}76 = 30{\cdot}48$

Calculated calorific value :—

$30{\cdot}48 \times 85{\cdot}50 + 6 \times 71{\cdot}19 - 59{\cdot}125 \times 9{\cdot}72 = 2453$ B.T.U.

Determined in the bomb calorimeter .. 2430 B.T.U.

Kerr and Percy* give data for the calorific value of bagasse of two cane varieties crushed at increasing extraction. The case (*a*) represents a Louisiana cane having a fibre content of 10 per cent. and a juice of 15° Brix, while (*b*) represents a Cuba cane with 12 per cent. fibre and 18° Brix in the juice. The authors state the calorific value both total and nett, without excess of air, and with an excess of 50 per cent. The temperature of the atmosphere was 27° C., that of the flue gases 260° C, while loss of heat in the ash was neglected. The next amount is found by subtracting from the total heat production the calories carried away in the flue-gases, that which is used in evaporation of the water present and formed, and 5 per cent. for radiation.

Extraction.	Bagasse.		Solids in residual juice.	Calorific effect.		
	Moisture.	Fibre.		Total.	Net value.	
					Without excess of air.	Air excess 50 per cent.
70	56·7	33·3	10	3627	2209	2090
75	51·0	40	9	4100	2666	2527
80	42·8	50	7·2	4817	3348	3184
70	49·2	40	10·8	4253	2792	2662
75	42·6	48	9·4	4801	3335	3173
80	32·8	60	7·2	5629	4091	3906

In these calculations it is, of course, assumed that during combustion of the organic matter in the bagasse only the highest products of oxidation are

* Bulletin No. 117, Louisiana Exp. Station.

formed, but according to Bolk's experiments,* this requirement is not always fulfilled, so that the full calorific value is not really utilized. This is especially the case with bagasse, having a water content of over 50 per cent., because this does not burn properly but merely smoulders and produces a large quantity of products of destructive distillation, chiefly heavy hydrocarbons, which escape unburnt and are thus lost.

Causes of Incomplete Combustion.—Some canes with very soft fibre, and consequently yielding a very moist bagasse, were sent to the author for examination, because they would not burn, but smouldered and eventually extinguished the fire. The engineers ascribed this to a high percentage of silica or of salt in the bagasse, but it soon became evident that the excessive amount of water accounted for the phenomenon. Sometimes the silica content was slightly higher than usual, but the divergence never exceeded 0·5 per cent. and one cannot suppose that so trifling a rise in an infusible constituent like silica would render bagasse incombustible. The presence of soluble salts in the bagasse to an appreciable extent must also be rejected, for had they been present in the original canes in excessive quantities, they would have been removed with the sugar and the other soluble juice constituents by the different operations of mill extraction and maceration, so that none would be found in the bagasse. In all those cases the author found a high water content ; the bagasse did not actually burn but smouldered slowly and left a black carbonaceous residue. The calorific value was greatly reduced and the engineers could not maintain the desired steam pressure in the boilers. At first sight one could only ascribe the deficiency in calorific value to the amount of unburnt carbon in the ash ; but a simple calculation showed that this was immaterial and that the diminished heating power was due, partly to the large amount of heat required to evaporate the surplus water, and partly to the formation of products of destructive distillation which escaped unutilized through the flues.

Unburnt Organic Matter left in the Ash.—The blackest ash, containing the largest amount of carbon, was obtained from a bagasse with 52·50 per cent. of moisture ; it contained 74 per cent. of inorganic matter, 21 per cent. of combustible matter, and 5 per cent. of moisture. The original bagasse, in its moist condition, had contained 46·38 per cent. of combustible matter, and 1·12 per cent. of ash. The original fuel therefore contained 1·12 parts of ash for every 46·38 of combustible matter, or 3091 of the latter for every 74 parts of ash. After combustion, we found only 21 parts of combustible matter to 74 parts of ash, which proves that this small amount of unburnt organic matter is of no real significance, when compared with the large amount which was burnt and disappeared in a gaseous form, and with the amount required to evaporate the water from the moist bagasse.

On some estates it was observed that the bagasse of some special cane

* "Archief voor de Java Suikerindustrie," 1906, 319.

variety yielded a more fusible ash than that of other varieties. Whereas the bagasse from certain varieties of cane gave no trouble in the furnaces and no clogged grate-bars, when other varieties were being crushed great care had to be taken to keep the grates free from clinkers, which hampered the draught and hindered the combustion. This, also, was often ascribed to the composition of the ash as containing more fusible constituents at one time than at another; but it soon appeared that the difference in the composition of the ash was imperceptible and that once again the physical condition of the fibre of the bagasse accounted for it. One kind of fibre can burn under conditions of draught and grate area so different from those required by any other kind that in one case the ash fuses and in the other cannot reach such a high temperature. By adjusting the conditions of the furnace any bagasse can be burnt so as to yield a fused or an unfused ash. A good example of this is furnished by generator furnaces where such a high local temperature prevails that the bagasse ash fuses into a glassy mass and clings to the walls of the furnaces in the form of glassy lumps. The special construction of the furnaces keeps the grates free, so that no obstruction in the air supply is experienced. Now this same bagasse can be completely burnt in other furnaces without yielding a fused ash, which proves that it is not so much the character of the ash as that of the furnace which gives rise to the molten state. Indeed, in the same furnace a flaming bagasse may yield a more fusible ash than a smouldering bagasse, and there is much more need to guard against extremes in either direction, by changing the slope of the grate or the space between its bars, or similar expedients. If a bagasse leaves a fused ash, it is much better to see if some change cannot be made in the position of the grate than to continue scraping the grate bars at intervals.

Conditions governing Fuel Value.—Besides the chemical composition, the physical condition of the bagasse influences its fuel value. It often happens that, for a given grate area, sufficient steam is produced when hard canes are being crushed, but that this falls short as soon as soft canes pass through the mill. It soon appears that this difference is due to variations in the specific gravity of bagasse from different cane varieties as mentioned on page 55. Thus, in the case of hard canes, a greater weight of bagasse lies on the grate than in case of soft canes, and this alone is sufficient to affect the draught, the admission of air, and perhaps other circumstances also. Economic stoking may also be impossible in the case of some kinds of bagasse without making alterations in the furnace or in the grate area.

"Molascuit."—The use of bagasse for the manufacture of "Molascuit" will be discussed at the end of this Part (page 328).

Bagasse for Paper Making.—Many attempts have been made to use bagasse as raw material for paper, and though the result has been satisfactory in many cases, paper has not yet been produced from this material in large quantities.

Owing to the comparatively small quantity of cellulose in cane fibre, the large percentage of lignin and the quantity of soda required to remove the latter, the cost price of such paper must be rather high. In countries such as Louisiana, where fuel and chemicals are cheap, and where there is an abundant supply of pure water for washing the pulp, the manufacture of paper from bagasse may have good prospects before it, but for countries where chemicals and fuel are very expensive, or where good water is scarce, the future of this industry is doubtful. The manufacture of paper from bagasse was started some years ago on a sugar estate in Texas but had to be abandoned; but more recently a successful enterprise has been reported from the Tacarigua Estate, Trinidad,* where very fine wrapping paper, that can also be printed on, has been manufactured from bagasse. This paper is, however, not made from bagasse alone, but from Para grass (15 per cent.), bamboo (20 per cent.), and bagasse (65 per cent.).

Dr. Viggo Drewsen succeeded at the Cumberland Mills, Maine, in making an excellent printing and letter paper solely from bagasse from Louisiana and Cuba. The average yield was 50 per cent., and the paper was prepared by heating the bagasse under pressure with a large amount of caustic soda.

But although the prospects appear very promising, high-class paper has not yet been manufactured from bagasse on a commercial scale.

An exception has, however, to be made for a coarse kind of paper made at the Olaa factory in Hawaii, which is extensively used for mulching in order to keep down weeds. When the cane tops have been planted the rows are covered with long strips of the paper in which here and there longitudinal cuts have been made. The paper covering prevents weeds from growing, but the young cane stems pierce through the cuts. This paper, which of course need not be of superior quality, is also used in the pineapple plantations in the Hawaiian Islands, while it is said that in Australia it will also be used in cane cultivation for the same purpose.

At Marrero, in Louisiana, the Celotex Company has built a factory, which converts bagasse into boarding material. The bagasse bales, entering the mill, are opened and fed by conveyor through a shredder to the cooker, in which the resins, waxes and similar constituents are removed, leaving the fibres tough and flexible. As in the case of ordinary paper, fibre separation is obtained by treatment in beaters and refiners. Waterproofing material is added at this stage and the pulp pumped to storage tanks, which feed a machine felting the fibres into a continuous board 12 ft. wide, which board is formed in one operation, and is not built up of separate layers. Passage through an 800 ft. continuous hot air dryer removes absorbed water and all that remains to be done is to cut the board by means of cross-cut and rip saws into standard 4 ft. widths, the length varying from 8 to 12 ft. as desired. It finds use for roofing, the exterior finish for plantation offices and the like, for the construction of refrigerator cars, cold storage warehouses, etc.

* "Int. Sugar Journal," 1908, 264.

CHAPTER II

CLARIFICATION

I.—Introduction

Principle.—The turbid, viscous juice from the mills is not fit to be worked up into sugar without clarification, and the first step is to submit the juice to such treatment as will remove as complete as possible the viscous and gummy constituents. As sucrose is very liable to decomposition by micro-organisms, which results in sourness, inversion and fermentation, and as cane juice is a very suitable medium for their development, it is necessary to boil the juice immediately, whereby the albuminoids are coagulated and the further development of the micro-organisms checked. It is also necessary to remove all impurities, both dissolved and suspended, so far as is practicable, without spending too much on clarifying agents, which may cost more than the results are worth.

The purpose of clarification is, therefore, to free the juice as quickly as possible from all constituents except sucrose, without alteration of the sucrose itself, and to perform the necessary alterations without delay, since it is only in concentrated solution that sugar is unaffected by ferments and micro-organisms.

Influence of Micro-Organisms on the Juice.—The cane hauled into the sugar mill carries an extensive flora of micro-organisms on its rind, while the earth adhering to the rootlets also harbours an innumerable growth of bacteria, so it is not surprising that every now and then some species or other of micro-organism causes trouble, and that in general good care has to be taken to restrict the nefarious consequences as closely as possible.

Bolk* reports the results of mill control in which the amount of sucrose and the quantity of the juices from the individual units of the train were accurately recorded, and compared with the corresponding data of the total mixed juice. The investigations were performed as accurately as possible ; the milling plant had no subterranean conduits, so that no juice could be spilled by leaks or overflowing ; and yet out of the 60 factories under consideration the number

* "Archief voor de Java Suikerindustrie," 1915, 1476.

of those, in which the total quantity of the sucrose in mixed juice was equal to the sum of the sucrose stated in the individual juices, amounted to 4; the balance of 56 reported a shortage.

These deficits were divided as follows, expressed in percentages of the sucrose in mixed juice :—

8	under	0·2
8	were	0·2–0·4
5	,,	0·4–0·6
5	,,	0·6–0·8
10	,,	0·8–1·0
16	,,	1·0–1·5
3	,,	1·5–2·0
1	was	3·1
56		

It may be that analytical errors are included in the figures for the loss, but they cannot be considerable, for in this case where all the weighings and determinations of the intermediate juices have been carried out in exactly the same manner as those of the mixed juice, faulty analytical methods would conduce to identical errors, the influence of which would check the rest. It is, therefore, a fact that between the mill and the measuring and weighing tanks relatively much sugar is lost through inversion, and that therefore a thorough and constant cleaning combined with an occasional disinfection of juice-gutters, sand-boxes, measuring tanks, pumping conduits, etc., is strongly to be recommended, while the route which the juice has to travel from the mill to the defecators needs to be as short as possible.

In case it is thought necessary to include sand-boxes or stone-catchers in the path of the juice, it is advisable to have them shallow, and to empty and clean them repeatedly in order to prevent micro-organisms from accumulating at the bottom and spoiling the juice. If possible, the gutters and tanks should be in duplicate in order to allow one set to be disinfected while the other one is in use, so as to have always apparatus exempt from bacteria so far as this is feasible in practical working. It is also necessary to wash the mills daily, while in case of a serious infection, washing with a one per cent. solution of ammonium fluoride will be found a useful disinfectant.

Among the micro-organisms which do most harm at this stage of manufacture, *Leuconostoc mesenterioides* should receive first mention. *Leuconostoc* grows very rapidly in alkaline sugar solutions and only very slowly in neutral or acid ones. When, therefore, the cold juice is limed in the measuring tanks and flows through pipes, cocks, or valves, even a slight infection with *Leuconostoc* may cause these to become choked. A small deposit of dextran in such places is apt to grow very rapidly in the current of alkaline juice. On the other hand, the growth of *Leuconostoc* is soon checked in a small tank,

because this organism produces acid substances which are detrimental to itself. But, when growing in a pipe or other locality through which alkaline juice is constantly passing, the liquid never becomes acid, or even neutral, and the development of the organism is incredibly rapid. Instances where large pipes have been speedily choked would surprise those who witness the occurrence for the first time. It is evident that in unlimed (acid) juice the formation of dextran cannot proceed, and there is consequently no fear of clogging the bores of cocks or valves. In cases of infection with *Leuconostoc*, it is therefore advisable to pump the unlimed juice through juice heaters and thus kill the germs of the bacteria, and to lime the juice afterwards. Experiments on this point have proved that cane juice may safely be heated for a few minutes to over 100° C. without inversion.

De Haan* found a second organism in the juice, *Oidium terricula*, an inmate of the earth, adhering to the bottom ends of the cane. This organism does not attack sucrose and may in this respect be considered as relatively innocent, but it attacks reducing sugars with the formation of acids having soluble lime-salts, which cause the juice to acquire a large amount of those salts after tempering and to yield a larger amount of molasses than in cases where the *Oidium* is absent. Finally, de Haan detected the presence of lactic acid bacteria which thrive in the juices and also form an acid having a soluble lime-salt. These bacteria, contrary to those referred to above, do attack sucrose and therefore cause likewise a direct loss by inversion.

Since all those organisms are killed by heat, rapid work is the best way to get the juice to the stage where their obnoxious influence is checked, while, as has been said above, cleanliness and disinfection are the best aids to success in this respect.

Principles of Clarification.—The aim of clarification is in the first place to prevent non-sugar being formed afresh in the juice and next to remove as far as possible the objectionable matter which may be present. A further requirement is rapid work, while the high temperature which is required to kill ferments and to promote clarification has, on the other side, the drawback of affecting sucrose by charring orby overheating.

Before starting to discuss the methods of clarification with their advantages and defects, we have first to point out clearly what is to be understood by clarification and which troublesome or obnoxious substances we have to get rid of.

On considering the clarification of cane juice from a general viewpoint, we perceive that its final object is twofold, viz., to make the juice clear, as well as to make it light in colour. Further, we see that of these two requirements the former is the principal one, the latter being only of minor significance, and in most cases being already included in the first mentioned.

* "Archief voor de Java Suikerindustrie," 1914, 1352.

Physical Nature of Clarification.—Up to a short time ago, the chemical side of the question had claimed most attention in the study of clarification. Starting from the fact that lime is the universal clarifying reagent, applied either in small or in large proportions, and is capable of transforming the viscous, slimy and turbid raw juice into a limpid and clear liquid, the chemical reactions of lime on the various constituents of cane juice have been the object of careful investigation and consideration. Under the spell of the idea that clarification by lime throws down not inconsiderable amounts of impurities, great value has been attached to the figures showing the increase in the quotient of purity as the outcome of clarification. Whenever a new clarifying agent has been introduced (and everybody knows that this has occurred quite frequently), an enormous increase in that quotient has always been said to have been one of the salient advantages of the discovery.

It is, however, not clear which of the non-sugars of the juice may be removed by the application of the great variety of chemicals proposed ; nor is it easy to see how a relatively small amount of chemical could possibly precipitate great quantities of objectionable non-sugar on being added to the juice. Finally the analysis of the filter-press mud, in which the harmful or undesirable impurities ought to be present in a concentrated form, has failed to throw any light on the subject.

In consequence, we are forced to the consideration that the clarification of cane juice does not aim so much at the removal of a large amount of impurities as at effecting a radical change in the physical properties of some of the constituents of the juice.

Flocculation of the Colloids.—First of all, we are inclined to think that there occurs a change in the degree of dispersion of bodies present in the juice as colloids, which not only do not subside, but, owing to their protective action, also prevent certain kinds of insoluble matter, such as earth, clay, *bagacillo*, wax, air, etc., from settling or escaping. The properties of the mixture thus resulting account for the sticky and slimy character of the juice.

If these considerations are correct, the chief object of clarification will have to be directed towards changing the degree of dispersion of the colloidal constituents of the juice in such a way that they become insoluble and subside. As soon as the colloids are thrown down in this way, the insoluble matter held in suspension by them is allowed to subside, or to rise to the surface, according to its specific gravity, leaving the juice in any case clear and limpid.

Before making a choice between the different agents which promote the flocculation of the colloids, we have to ascertain the real character of the latter. In the literature of sugar cane are found many statements that gums, pectins, albuminoids, etc., account for the stickiness and resistance against filtration of cane juice, but there is no certain proof that the bodies mentioned are to be considered as the real cause. Neither albuminoids nor gums, nor pectins, occur in appreciable proportions in the juice ; and we ourselves feel inclined to

adhere to the hypothesis of Müller,* who believes a colloidal silicious compound to be one of the principal causes of the viscosity of cane juice.

Among the means generally in use to promote the flocculation of colloids the following may be mentioned here :—(1) Addition of an electrolyte ; (2) Action of the electric current ; (3) Change in the reaction of the solution ; (4) Elevation of the temperature ; (5) Adsorption by surface-attraction using porous or flocculent materials ; and (6) Separation by centrifuging or ultra-filtration.

In this discussion it will be seen that the various methods of clarification in practical use may be classified under one or more of these headings.

Addition of Electrolytes.—Addition of an electrolyte to the juice will be recognized at once as not a very promising possibility, for if the quantity required is large, the expense will be prohibitive ; while, moreover, the great amount of salts introduced will immobilize too much sugar in the final molasses and thus cause too great a loss. Small amounts of any electrolyte are surely useless, for if there existed a material which in small doses could cause the colloids of cane juice to flocculate, it must have been among the many hundreds of chemicals which have been recommended for use in clarification during the course of time. As it is known that none of these has been successful, we do not hold out any hope for the application of electrolytes for the purpose in view.

Action of the Electric Current.—Better assistance may be expected from the electric current, although the results of manifold experiments in treating juices with electric currents of various degrees of intensity, using all kinds of electrodes, combined or not with dialysis, have not been able satisfactorily to solve the problem.

Change in the Reaction of the Solution.—In our opinion the best results may be expected from the proper tempering of the juice, so that the exact point at which the colloids coagulate is reached. This tempering, combined with high temperatures, constitutes the well-known method of Defecation which has been in use for centuries all over the world.

On adding continuously small amounts of milk-of-lime to boiling cane juice, and observing the appearance of the mixture after each addition, a precipitate is seen to arise already after the first portion, this increasing after further additions till it reaches its maximum. At the same time a gradual change in the character of the precipitate is observed. At first it is suspended in the juice, but gradually it becomes less bulky, subsides easily, and leaves the supernatant liquid clear. It may happen that with further additions of lime the precipitate swells more and subsides less rapidly ; but, at any rate, in every cane juice a point may be observed where the proper amount of lime brings along rapid and complete subsidation.

* "Int. Sugar Journal," 1921, 579.

The determination of this point has offered great difficulties up to now because of the lack of a reliable and rapid method which enabled one to find, at once, the real point up to which the juice had to be tempered in order to get the best results. In many cases the reaction to litmus or turmeric paper was considered to be the criterion, or the filtered juice was titrated with N /28 acid, and a certain alkalinity was adopted for general use, but in most cases the degree of tempering was ascertained in an empirical way for the juice under treatment. Whenever canes having a different constitution to those previously treated were being crushed the point in question had to be ascertained anew, which rendered proper tempering a rather uncertain operation.

It has, however, been proved by Gebelin* and others that the proper point of flocculation of the colloids in cane juice stands in direct connexion with the hydrogen ion concentration of the juice. After having determined once and for all empirically, either in the factory or in the laboratory, at which hydrogen ion concentration the subsiding of the hot-tempered juice is at its best and completest, the only thing to be done in future is to temper the juice till it has attained the required potential.

This point may very easily be determined by means of slips of paper soaked in solutions of one or other of the many colour reagents which have come in use since the publication of the experiments of Lubs and Clark.†

Gebelin showed that the optimum tempering point lies at a pH of 7·2 at ordinary temperature ; and, as in that case the juice is almost neutral, there is the great advantage that no action of alkaline bodies on the reducing sugars is to be feared, coloration due to that occurrence being thus excluded.

Adsorption by Surface Attraction.—This simple defecation process permits a good subsiding of the juice, so that the supernatant clear liquid may be syphoned off from a muddy layer ; but this clarification is not thorough enough to yield a juice fit for the manufacture of white sugar. In order to clarify a juice for that purpose, we avail ourselves of the property of colloids to adhere to porous or flocculent materials introduced in the juice to be clarified.

One instance of this property, extensively applied nowadays, is the use of infusorial earth (as "Filtercel") and of various kinds of vegetable carbons, which absorb the colloids and carry them to the bottom of the settling tanks.

The sulphitation and the carbonatation methods are based on the same principle of removing colloids by surface attraction. More lime is added to the cane juice than is necessary for the optimum pH, after which the excess of lime is removed by either sulphurous or carbonic acid till the proper range of hydrogen ion concentration is obtained.

These acids form insoluble lime-salts in the liquid, which, while forming and

* "Louisiana Planter," 1923, 71, No. 9, 172.

† W. Mansfield Clark, "The Determination of Hydrogen Ions." See "Int. Sugar Journal," 1923, 262.

subsiding, allow the colloids to become incorporated in their interstices, thereby leaving the juice clear and limpid. Alternatively, one can sulphite the juice first to a distinct acid reaction, and lime back to the exact pH, in which case the precipitate of calcium sulphite effects the same purpose of attracting the colloids.

Effect of Defecation on the Quotient.—It is seen from these instances that every method in practical use corresponds to one or to a combination of some of the suggested means of causing a change in the degree of dispersion of the colloids. Notwithstanding the seemingly chemical character of many methods, their action is only a physical one, so that in reality no great quantitative changes take place during clarification.

This explains why generally the quotient of purity of a clarified juice does not differ greatly from that of the original raw juice. Very little non-sugar has been eliminated from the juice, and therefore the quotient of purity does not undergo a great increase.

We quote from the statistics issued by the Java Sugar Experiment Station the following data on the increase in quotient of purity from raw to clarified juice during the many years of its existence :—

1905 ..	1·7	1912 ..	1·7	1919 ..	1·6
1906 ..	1·6	1913 ..	1·8	1920 ..	1·5
1907 ..	1·7	1914 ..	0·7	1921 ..	1·6
1908 ..	2·0	1915 ..	1·5	1922 ..	1·6
1909 ..	2·0	1916 ..	1·3	1923 ..	1·7
1910 ..	1·9	1917 ..	1·5		
1911 ..	1·7	1918 ..	1·4		

Nor do the separate individual figures generally show any great divergencies.

It is true that sometimes instances are mentioned of a very great increase or even a decrease. In such cases it has been found that either very heavy substances (as sand or clay) or very light ones (as air) were held in suspension by the colloids of the juice, thus influencing the readings of the Brix hydrometer with the aid of which the quotient of purity is determined. As soon as the colloids flocculated and became insoluble, they released the suspended bodies, which then either settled or escaped, their delusive action on the Brix hydrometer thus being eliminated.

As the colouring matter of cane juice is one of the colloids, any method for removing these bodies serves at the same time as a decolorizing device, which is quite sufficient for the manufacture of raw sugar.

Strainers. Sandboxes. Centrifugals.—Juice from the mills passes through strainers having a gradually decreasing width of mesh, which keep back the coarse pieces of bagasse. Further, it is conducted through a sandbox, where heavy particles, as sand, clay, etc., are separated, but, although these devices keep back not unimportant amounts of impurities, yet they are unable to remove colloids. We know of centrifugals by Hignette, Kopke, Sandford and others, in which the limed juice, either cold or warm, is separated into a hard crust of filter mud and a clear juice, but that is not what we mean here.

The centrifugal invented by Mauss* for use with raw juice claims to remove 96 per cent. of the floating and suspended material from the juice. It comprises a drum composed of a number of separable sections of conical form, which are pressed apart axially by springs, the raw juice being introduced through a pipe, and the solids deposited in the spaces, the clear juice escaping by a channel at the upper end of the drum. After running the machine until a certain amount of solids is deposited in the chambers, the supply is stopped, and the drum sections separated. The fully separated solids escape through the outlet, while the partially separated solids and liquid are carried back to the supply vessel. In the case of a machine in which the diameter of the drum is 2 ft., the speed is 1000-2000 revolutions per minute.

A second apparatus for the removal of colloids is the ultra-filter constructed by Plauson, which may remove particles having a diameter of one millionth part of a millimetre. It consists of a perforated metal cylinder, covered at the outside by perforated rings or plates which may be tightly pressed together by screw bolts. The perforations consist of elongated slots, adjacent plates being arranged so that the slots of one are at right angles to those of the other. This arrangement forms a filter element in which openings of the pores may be varied at will only by changing the position of the screw bolts. A slimy layer is deposited on the interior side of the cylinder, and is kept at a constant thickness by a worm with decreasing pitch towards the discharge, which constantly scrapes off the dirt, only maintaining a thickness of 1-2 millimetres, this in fact constituting the proper filtering surface. The cylinder is therefore internally coated with a felt blanket. The juice is forced into the drum under heavy pressure; passes the filtering layer; and leaves the cylinder through the pores; while the dirt scraped off by the worm is pushed outside through a trap.

In this book we shall only treat of the manufacture of raw sugar. Processes for the manufacture of white sugar in cane sugar factories have been described and discussed in a separate work by the author: "Practical White Sugar Manufacture,"† to which we take the liberty to refer the reader.

* "Int. Sugar Journal," 1921, 592.
† Norman Rodger, London.

II.—Methods of Clarification

A.—TREATMENT OF THE RAW JUICE.

Principle.—As we have shown before, the aim of clarification is to temper the juice with lime up to that concentration of hydrogen ions at which, in connection with the high temperature, the best flocculation and subsidation of the colloidal constituents of the cane juice is attained.

It is therefore evident that, in the first place, the determination of the amount of lime proper for the attainment of the optimum range of hydrogen ions is of the greatest importance, and next comes the raising of the temperature of the juice to the highest point which is still compatible with the risks of charring and overheating,

Since the optimum range for the hydrogen ions in the clarification of cane juice is about pH 7 or slightly under, the breaking up of reducing sugar by alkalis or by lime, which in former days was apt to cause a dark coloration of our juices, is no longer to be feared and may indeed be left undiscussed here.

Tempering.—When determining the amount of lime to be used for a proper tempering, we add as much lime to the juice as seems appropriate, say two gallons of milk-of-lime of 15° Beaumé to 500 gallons of juice, and effect clarification as usual. A sample of that clarified juice is poured into a glass cylinder for subsidation. In the next clarifier a somewhat larger quantity of milk-of-lime is used in tempering, and a sample of the defecated juice is poured into another cylinder glass. Further, a smaller amount of milk-of-lime is used, so that we get a scale of samples representing the different amounts of milk-of-lime added to the juice. That sample in which the sediment settles best and quickest with the least amount of lime is selected as the standard one, and is used for the determination of the pH range to be adopted in future.

Determination of the Value for pH.—In the chemical laboratory the best and most accurate method for the determination of the concentration of hydrogen ions in solutions of pure bodies in water is by the potentiometer. But in the viscous and slimy cane juice the electrodes would very soon become coated with a deposit which would hamper the conductivity, while in addition the rather subtle measurements of a sensitive voltmeter are out of place in the noisy and vibratory atmosphere of a sugar-house laboratory in the turmoil of the grinding season.

Hence, we avail ourselves rather of the many colour indicators that have come into universal use since the brilliant investigations of Clark and Lubs.

Since we have only to deal with a range between 6 and 7·5 for the pH, we may confine ourselves to the following indicators:—

Name of Reagent	Range of pH, from—to	Colour Range from — to
Bromcresol purple	5·2 6·8	yellow purple.
Bromthymol blue	6·0 7·6	colourless blue
Corallin	6·9 8·0	yellow red
Phenol red	6·9 8·4	yellow red

The following methods may be used: A drop of the juice under examination is mixed with a drop of the proper indicator solution* upon a piece of opal glass. About this are placed drops of the standard juice and each is mixed with a drop of the indicator solution. Direct comparison is then made. The porcelain indented plate used in the titrimetric determination of reducing sugar with Fehling's test solution may also be advantageously used.

It is, however, much better and simpler to use slips of paper soaked in the solutions of the colour reagents and afterwards dried. Once and for all, a suitable reagent paper is selected by testing various papers with a juice giving the best practical results in clarifying and subsiding. In the factory every juice is tempered till it produces, at the temperature adopted as the standard one, the proper coloration on the selected paper dipped into it.

Proceeding thus, every juice may very easily and accurately be tempered to the very best degree and all difficulties encountered up to now in tempering the juice extracted from varying lots of cane may be avoided in a very simple manner.

The question of the temperature to be applied will be discussed during the description of the various methods of clarification.

Defecation Methods.—Defecation may be conducted in either of two ways, depending on the manner in which the precipitated impurities separate from the juice. In the first method the juice is allowed to subside in the same vessel in which it is heated, while in the other the limed and heated juice is run into settling tanks.

When adopting the first method the juice is strained through copper gauze screens, measured and limed in the measuring tanks, passed through juice heaters, which raise its temperature to about 90° C., and finally enters the defecators, where it is further heated to the boiling point.

The defecators are hemi-spherical copper pans, with steam jackets into which steam is admitted during the time that the juice fills the pans. The precipitated impurities coagulate, envelop the suspended particles and (air bubbles escaping

* The solutions are prepared in the following manner: 400 mgrms. of the indicators are dissolved in 100 grms. of hot alcohol and the 0·4 per cent. solutions thus obtained are kept as standards. In use these standard solutions are diluted with water; those of bromthymol blue, bromcresol purple and corallin in the ratio of 1 : 10 and that of phenol red of 1 : 20. For the preparation of reagent papers, sheets of filtering paper are impregnated with these diluted solutions, dried on a line in an acid-free atmosphere, and cut into small strips.

from the hot juice), form a frothy, dark-coloured layer of scums, underneath which is the clear juice. Only the heavy impurities, such as sand, clay, &c., sink to the bottom and form another layer, so that the juice is separated into three layers, of which the middle one, consisting of clarified juice, is the greatest.

Settling and Decanting.—The juice is heated until the layer of scum begins to burst (cracking point); the supply of steam is then shut off, for, if not, the juice would boil over, and be partly lost. The juice is now allowed to settle, and afterwards let off through a drain at the bottom of the pan, which is fitted with a pipe closed by a three-way cock. By turning the plug one is able to draw off successively the heavy impurities, the clean juice, and finally the scum, each of these being conducted into its own gutter. The first and third portions, *i.e.*, the heavy impurities and the scums, are run into the scum tank, while the clean juice is conducted to the evaporators or to the eliminating pans. The three-way cock and the gutters should be placed in an easily accessible and well-lighted position, so as to admit of complete control. In order to facilitate the decanting and the separation of the clear liquid from the mud and scums, some defecators have two outlets at the bottom, one for the sediment and the other for the clear juice: the latter carries a pipe which extends above the surface level of the sediment within the tank. When the defecation and subsidation are completed, the latter cock is opened and the clarified juice discharged, without risk of contaminating it with the subsided mud, which remains below the outlet level of this pipe. The clarified juice is allowed to flow out until the scums approach the open end of the pipe, whereupon the cock is closed and the other discharge pipe opened, when the subsided mud, scum, and thin layer of clarified juice between these two, pass into the scum tank for further treatment.

In the other method, the limed juice, after having passed through a juice-heater, is boiled in iron pans, through which steam is led by means of coils. As soon as the juice boils, a cock is opened at the bottom of the pan, and the hot juice flows into the settling tanks, where the separation of the clear juice from the scum takes place. As in this case the air has been completely driven out by the ebullition, the scum does not remain as a froth on the surface, but settles gradually, leaving the juice quite clear.

Pans.—Some factories are provided with special cylindrical iron pans having broad gutters. In these pans a steam calandria is suspended, reaching nearly to the bottom. This is perforated by a number of tubes through which the juice circulates, and is thus caused to boil rapidly.

As the frothy scum rises to the surface, it is driven into the gutters by long skimmers, whence it flows into the scum tank. When no dark-coloured particles are visible at the surface, but only a white mass of steam bubbles, the steam supply is stopped and the juice with the heavy impurities is let off into the settling tanks.

Continuous Defecation.—In many places the juice is heated to the boiling point in the juice-heater, and then allowed to flow through one or more pans, in which it boils rapidly, and thence passes into the settling tanks in a continuous stream. In this case no skimming can take place, but as the boiling breaks up the layer of scums, this mixes with the heavier deposits and does not interfere with the subsiding in the settling tanks.

It is not advisable to pump the juice through the juice heaters too quickly, as it frequently happens that the time during which the juice is heated is not sufficient to allow of complete defecation. Even when using very powerful juice-heaters, one should not omit to boil the juice in a defecator before it is discharged into the settling tanks.

Deming's System of Clarification.—A third method of defecation, devised by Deming, has come into use, in which the limed juice is superheated and subsequently passed through continuous subsiders. The limed juice is pumped through three vessels, in which it is heated under pressure to over 110° C., and afterwards cooled to about 100° C., so that the juice entering the apparatus at the ordinary temperature is heated for about 45 seconds at a temperature of 110-115° C., and leaves it at a temperature of 95-100° C. The three vessels are horizontal cylinders, in which tubes of 2 in. diameter are fixed and connected with each other by chambers at either end of the cylinders in such a way that the juice flows backwards and forwards through the tubes with great velocity. Two of the vessels are called "absorbers"; the third (having the largest dimensions) bears the name of "digester." In the first absorber the cold juice is introduced outside the tubes, but passes through the tubes of the second absorber, around which exhaust steam is circulated. Afterwards the juice flows through the tubes of the digester, is heated by live steam to the required temperature of 110-115° C., and finally passes through the tubes of the first absorber, where it imparts its superheat to the fresh juice surrounding the tubes, heating it to 40-50° C.

Scale on Deming Tubes.—Owing to the high velocity with which the juice is forced through the tubes there is no fear of scaling, in fact they remain quite clean after some months' use. The apparatus only now and then requires a thorough cleaning with hot dilute hydrochloric acid, more especially the outside of the tubes in the first absorber, which get coated with mud from the cane juice.

An analysis of the air-dried scale from the outside of the tubes gave the figures tabulated on page 154. The scale, we see, consisted of calcium phosphate, other lime compounds with organic matter (acids, albumin, pectin), which changed into calcium carbonate during combustion, sand, clay, and partially caramelized constituents of the cane juice, which had become overheated by contact with the tubes.

	Moisture	4·66
	Organic matter	52·56
In the Ash.	Insoluble in hydrochloric acid	9·12
	Lime	14·22
	Magnesia	4·95
	Iron oxide	2·18
	Alumina	0·65
	Phosphoric acid	1·92
	Sulphuric „	0·27
	Carbonic „	8·20
	Undetermined	1·27
		100·00

Advantages and Disadvantages.—Each method of defecation has its advantages and its defects. The advantage of the first method is that the juice is not heated to its boiling point, hence steam is saved, and, further, no danger of inversion or decomposition is to be feared. A drawback, however, is the great number of expensive defecators required if a properly clarified juice is to be obtained.

The common defecation pans with coils are easy to clean, which is not the case with the pans in which the juice is boiled in tubes of small diameter, unless a sufficient number of these are available, enabling one pan to be spared for cleaning while working with the others. The skimming off of the light floating impurities has the disadvantage of keeping the juice for a long time at a high temperature, involving extra expenditure of steam, and possible decomposition of a portion of the sugar; though, as will be shown later, this danger is not a serious one.

Against these disadvantages must be set the prompt removal of the lighter portions of the scums in the skimmings, leaving only the heavier and more quickly subsiding portion to settle in the tanks; this saves much time and increases the working capacity at this stage.

The advantages of superheat clarification are economy of steam, labour, space, and the fact that superheated juice subsides better than boiled juice. Yet no difference in the chemical analysis is to be detected in the juices clarified in either of the methods discussed here. The only real difference lies in the economy of heat and labour.

Accessories to Proper Defecation.—In many cases the volume of the precipitate brought down in the juice by the proper amount of lime necessary for obtaining the optimum pH fails to remove sufficiently the colloids from the juice. In such a case a good clarification may be obtained by adding to the juice bodies which are apt to form a porous or a flocculent deposit, which drags down with the mud the colloidal and floating particles.

Clay.—Years ago an emulsion of clay and water was recommended to promote subsidation, but this rather crude material has not found general employment.

Infusorial Earth.—More recently we have heard a great deal of infusorial earth (kieselguhr) for the same end, and an American preparation especially, called " Filtercel," is claimed to yield good results in this respect. A quantity of 1 to 4 pounds of " Filtercel " to one ton of the limed juice is required if raw sugar is to be made, while a much larger amount up to 25 pounds per ton of juice is thought necessary in the manufacture of plantation whites.

Sulphitation.—Instead of mixing the juice with materials poured into it, we may make use of the capacity of precipitates arising in the liquid of accumulating colloids, and in this respect mention must be made of sulphitation, by which process an abundant deposit of calcium sulphite is formed which admirably serves our purpose. The oldest method of sulphiting cane juices dates from the beginning of the last century, and is still used in a great many sugar-houses.

The Sulphur Box.—It is carried out in the so-called " sulphur box," a wooden upright vertical chamber about 12 ft. high. At about each foot of height are fastened horizontal perforated plates or some other device intended to divide the flow of the falling juice into a shower. The sulphur fumes enter the box at the bottom through a pipe and are drawn upwards through the box by a jet of steam at the top, which acts as an aspirator. The cold mill juice flows into the box at the top, falls on the topmost plate, where it is divided into a shower, trickles down on to the next plate, and so on till it reaches the bottom charged with the sulphurous acid, from the upward current of sulphur fumes which it has encountered.

Sulphuring Tanks.—A more modern and more compact apparatus consists of a tank, some 4 to 5 ft. high, in which the sulphur fumes are conducted through a wide pipe ending a few inches from the bottom. The fumes are forced into the juice by means of an injector, while the juice enters through a pipe at the bottom and overflows through a second outlet placed three feet above the level of the first one. During its passage through the tank it encounters the current of sulphur fumes, absorbs the sulphurous acid, and flows out charged with the proper amount of gas, which may be regulated by the velocity of the flow of the juice or by the cock of the injector.

In another device the juice is sulphited in a tank, in which two baffle plates are fitted, which force the juice, pumped into the tank, to flow first down and afterwards upwards. These plates divide the tank into three compartments: the juice entering the first, passes through the opening between the bottom and the first baffle plate into the second compartment, where the current of

sulphur fumes is forced through it. Combined with the bubbles of gas, the juice passes through the opening between the top and the second baffle plate into the last compartment, whence it is discharged, saturated with sulphurous acid, by a pipe at the bottom.

Quarez Apparatus.—The Quarez system of continuous sulphitation makes the juice itself regulate the rate of flow of the sulphur fumes which it needs. Here the juice from the mills runs through a pipe into a tank, which is divided into two compartments by a plate almost reaching to the bottom; from here it is forced by means of a Worthington pump through an injector, which is in direct communication with the supply pipe from the sulphur furnace. By this arrangement the amount of sulphurous acid absorbed by the cane juice during its course through the sulphitation column may be regulated by the flow of the juice itself, while no draught takes place and no sulphur is uselessly burnt if the current of juice is interrupted for some reason or other. The juice itself is pumped back into the second compartment of the tank and leaves it by the overflow. Although the juice which is injected through the Quarez apparatus has previously passed through a series of copper strainers which deprived it of the greater part of the " cush-cush," or bagacillo, yet this does not by any means preclude some fine particles of cane fibre passing along with the juice and at the end choking the apertures of the injector. It is therefore advisable that two injectors be fitted to every apparatus, so that the operations may be continued with the spare one, in case the other has become choked and needs dismantling and cleaning. When working with the Quarez sulphitation apparatus, the flow of the juice and of the gas is, as nearly as possible, regulated in such a manner that the juice coming out of it has a sulphurous acid (SO_2) content of about 0·06 per cent., equivalent to 300 grms. of sulphur per ton of cane. In the older apparatus just mentioned, the absorption of sulphurous acid cannot be pushed so far; the average amount of sulphur, which after being burnt can be incorporated in the juice, does not exceed 100 to 150 grms. per ton of cane. The large amount of sulphurous acid absorbed in the juice in the Quarez apparatus is certainly sufficient for the proper decolorization of the juice and for the production of an abundant precipitate with the lime added afterwards, while at the low temperature of the raw juice the degree of acidity mentioned above, if caused by the sulphurous acid, the hydrolysing power of which is relatively weak, is not high enough to be dangerous.

Liming back.—The sulphited juice is run or pumped into liming tanks, where as much milk-of-lime is added to it during its ingress as will make its reaction neutral or very slightly acid.

The liming should be conducted so far that the juice does not impart a pink coloration to phenolphthalein paper, nor a red one to blue litmus paper, in which case there is neither an excess of lime nor an excess of free sulphurous acid sufficient to cause inversion during the subsequent heating on defecation.

Brewster and Raines* recommend the use of the Clark and Lubs' reagents and suggest sulphiting the juice to a pH of 3·8, to be controlled by methyl orange or bromphenol blue. Later on, the concentration of hydrogen ions is limed back to a pH of about 7, which may be indicated by bromcresol purple or phenol red.

These colorations are much more distinct than those obtained with litmus paper and give a much more accurate end-point. The liming should be performed or at least completed in large tanks, provided with a stirring apparatus which keeps the contents in slow movement. These tanks should have the capacity of half-an-hour's juice supply so as to facilitate the proper liming, which in this case has only to be finished once every thirty minutes.

This continuous sulphitation should be adopted in every case where it is thought advisable to sulphite first and lime afterwards. It is evident that, notwithstanding the greatest care, it is not always possible to regulate the flow of juice and gas and the sulphurous acid content of the latter so strictly that the juice always comes out with the same acid content ; and therefore it is not advisable to make use of this method of sulphitation if it is necessary just to neutralize a limed juice, as in such a case it might become either too acid or be left still alkaline. Where the juice is sulphited first and neutralized afterwards with lime, there exists no necessity for a constant acidity and it is of no consequence whatever if the acid content varies in the sulphited juice to some extent, since later it is entirely neutralized by the lime in the liming tank.

If, however, the juice is to be limed first and sulphited afterwards, it is better to use intermittent sulphitation, as in that case the limed juice must be neutralized exactly by the acid.

Intermittent Sulphitation.—In the intermittent sulphitation process the limed juice is pumped into iron tanks, in which a current of sulphur fumes from a sulphur furnace is introduced, by which operation the excess of lime is neutralized, and can then be tested with reagent paper. As soon as the point of neutrality is reached, the juice is discharged from the tank, which is filled again with a fresh portion of limed juice, so that every tankful is treated parately.

Usually the sulphitation plant consists of four or five iron cylindrical or rectangular tanks, together having a capacity equivalent to the amount of juice extracted per hour. A factory crushing 1,000 tons of cane per 24 hours therefore requires a total sulphiting capacity of 9,000 gallons. The tanks are each covered with a lid fitted with a flue to allow the escape of the surplus gas. Further, they are provided with valves for the ingress and egress of the juice, a special valve for the sulphur fumes with lead and rubber fittings, and a perforated iron distributing pipe reaching to the bottom of the tank. A small

* Paper read before the Section for Sugar Chemistry at the 63rd Meeting of the American Chemical Society, Birmingham, Ala., 3rd April, 1922.

cock fitted a few inches below the level of the juice, when the tank is full, permits of occasionally taking a sample of the contents in order to check the course of the neutralization.

The Java Sugar Experiment Station gives the following rules to be observed when applying the sulphitation process: The strained juice passes through juice-heaters in which it is warmed to a temperature of 80°–85° C, taking care to keep it as constant as possible within this range. On no account should it fall below 80° C. in the sulphitation tanks. The lime-cream is carefully sifted in order to eliminate all grit or coarse particles.

As soon as the tank has been filled, the sulphurous acid valve is opened and immediately afterwards the milk-of-lime is poured in in a thin constant jet, taking care to keep the reaction of the mixture neutral or faintly alkaline to phenolphthalein. The last quantity of lime-milk is added at once in order to have one short moment of a strongly alkaline reaction. Finally, the sulphitation is completed till a neutral reaction to phenolphthalein results.

In the case of small tanks in which sulphitation only requires a few minutes' operation, the milk-of-lime may be added at once since its excess is neutralized in a short time.

Depending on the quality of the juice, an amount of 3·5 to 5 gallons of lime cream of 15° Bé. per 500 gallons of juice is the proper amount.

A short period of strong alkaline reaction is indispensable for a good clarification; its duration should, however, be restricted to one minute or one minute and a half, in order to prevent the breaking up of reducing sugar and coloration. For this last reason the addition of lime-cream should be temporarily suspended at any moment when owing to untoward circumstances, the sulphur fumes supply is defective.

The proper subsidation is constantly controlled by inspecting samples from every one of the tanks placed in a test-tube in a rack; while, further, the subsided juice is titrated: 10 c.c. of the juice should require not more than 3 to 4 c.c. N/100 soda against phenolphthalein.

The limed and sulphited juice is heated to boiling point in juice-heaters, which is in most cases quite sufficient, while occasionally a passage through a defecation pan is also necessary. It is to be recommended to exchange from time to time, say every 12 hours, the heaters for the raw juice with those of the sulphited juice. This is a great help against incrustations, as those from raw juice dissolve in the sulphitated ones and *vice versa*.

Incrustations.—Incrustation of calcium sulphite in the tubes of the evaporator is not only prevented by a well-conducted sulphitation to the proper degree of acidity, but also by a high temperature in the juice-heaters and in the settling tanks. Calcium sulphite dissolves less in hot juice than in cold, and heating to 100°C. is sure to induce the salt to crystallize out. If, however,

the temperature in the settling tanks should sink, part of the sulphite may redissolve and crystallize out afterwards in the first cells of the evaporator, where the temperature is highest. This same fact teaches us that the temperature at which sulphitation takes place has great importance not so much in regard to clarification as in regard to the incrustation of the juice-heaters.

Formerly when the reaction during sulphitation was started strongly alkaline, the temperature was kept low and did not exceed 50° C., and in those days the tubes of the juice-heaters were badly incrusted. The analysis of such a scale gave the following figures :—

Silica	21·12
Sulphuric acid	7·66
Sulphurous acid	5·05
Phosphoric acid	3·78
Iron oxide	1·33
Lime	16·33
Organic matter	34·75
Water	8·25
Undetermined	1·73
	100·00

A second one showed a much larger amount of sulphite and sulphate :—

Silica	0·28
Sulphuric acid	9·14
Sulphurous acid	27·66
Lime	30·78
Alkalis	0·55
Iron oxide	0·40
Loss on ignition	9·22
Moisture	21·78
Undetermined	0·19
	100·00

Sulphitation applied at a high temperature may be considered a marked improvement, as thereby the troublesome incrustation of juice-heaters is greatly reduced. Yet the proper range of temperature is still an object of discussion. In the Assembly of Advisers to the Java Sugar Industry of 1922,* lower temperatures were advocated, ranging from 60° to 85° C. ; but in no case should temperatures of over 85° C. be allowed. Further, there is some difference of opinion as to the end-reaction on sulphiting.

* "Archief voor de Java Suikerindustrie," 1922, 123.

The Java Sugar Experiment Station* advises a maximum acidity against phenolphthalein of 120 mgrms. SO_2 per litre, with a minimum of 30 mgrms. Other workers keep the acidity between 60 and 80 mgrms.

It is quite likely that the colour reagents of Clark and Lubs may prove useful in the determination of the proper degree of sulphitation. In the event of a suitable colour reagent being established, the procedure would be to continue the gas supply till the reagent paper dipped into the juice shows the desired tinge.

Phosphoric Acid.—Besides sulphurous acid, phosphoric acid and its salts find application in the sugar industry. The acid combines with lime to form a flocculent precipitate which attracts colloids from the juice, thereby cleaning it. The different preparations used for this purpose are discussed on page 171.

B.—SUBSIDATION

Intermittent Subsidation.—The hot juice from the juice-heaters and defecation pans is carried to settling tanks, in which it is allowed to subside and to split into a supernatant layer of clear juice and a submerged one of mud. The intermittent settling tanks are square iron boxes, covered externally with wood to retain the heat, and provided with cocks at varying distances from the bottom. The juice settles, the uppermost cock is opened and afterwards the others, so that only the clear juice flows off and goes to the clarified juice tank. Finally a cock at the bottom is opened, and the scum flows out and runs into the scum tank. Instead of a row of cocks, we often find a single cock connected with a hinged copper pipe, the free end of which is fixed to a hollow copper ball, which floats on the juice, so that only the uppermost and clearest layer of the juice passes through the pipe.

All those tanks for intermittent subsidation have the drawback of leaving far too thick a layer of mud on the bottom, so that too little of the subsided juice is syphoned off, while the length of the subsidation operation causes the juice to cool down excessively, both of which inconveniences are avoided in the many systems of continuous subsidation, of which a few are described here.

Continuous Subsidation.—The hot juice coming from the defecator in the Deming system of clarification is conveyed into settling tanks, whence it is continuously discharged. These tanks are cylindrical with conical bottoms, carrying wide discharge tubes provided with a cock. A large cone of sheet iron (open at the top and bottom) is suspended in the cylinder, the height of the cone being equal to that of the cylinder, so that their upper edges are at the same level and the bottom of the cone level with the conical part of the settling tank; the distance between the cone and the tank at that place being slightly over one inch.

The heated juice enters the tank outside the cone, fills it and overflows

* *Ibid.*, 1922, III, 125.

through a wide tube leading out of the upper part of the cone. This arrangement causes a downward current of juice outside, and an upward current inside the cone. The scum has therefore a tendency to sink to the bottom of the tank, and if it is induced to rise by the upward current inside the cone, it is caught by the inclined side of the cone and again sinks. Care should be taken frequently to discharge subsided scum, so that no layer of dirt forms between the edge of the cone and the cylinder, as in that case lumps of scum may be carried to the surface and spoil the clarified juice.

In the case of slowly settling juice, the same expedient may be used as in the case of the ordinary clarification, i.e., rendering the precipitate heavier by adding more lime and a corresponding amount of phosphoric acid to the mill juice.

Ordinarily, two settling tanks are required; the juice flows first through the smaller, afterwards through the larger one.

The Dorr " thickener " or clarifier, mentioned nowadays in connection with the Thomas and Petree process of mud lixiviation (see page 162), corresponds to the following description: the apparatus consists of a circular tank, 20 feet diameter by 14 feet high, having a conical bottom firmly embedded in concrete, and is divided by plates into four compartments. The slope of the cone towards the centre is the same as that of the plates or bottoms of the compartments. The hot defecated juice flows into the subsider by a central tube and fills the four shallow compartments in which it subsides. In the centre of the tank is placed a vertical shaft, which revolves very slowly, and on which are fastened arms carrying scrapers, which force the subsided mud into a central tube, while the clear juice flows off from every compartment by an outlet tube. In those and similar systems the defecated juice is continuously divided into a large volume, even as much as 95 per cent. of clarified juice ready for evaporation, and a small one, which may be as low as 5 per cent. of thick mud, this being conveyed to the scum tank.

C.—TREATMENT OF THE CLARIFIED JUICE

In many factories the juice, after settling, is pumped without any further treatment into the suction tank of the evaporators; in others it passes through one or other form of filter; in others, again, it is once more boiled in eliminating pans of the shape previously described. This treatment is especially necessary for juice clarified by the first-named method, as a great part of the scums often accompanies the clarified juice owing to want of attention. The juice, after settling, is boiled, skimmed, rendered neutral by phosphoric or sulphurous acid if the juice is too alkaline, or with soda or lime if it is too acid, because, as already mentioned, a neutral or very slightly acid or alkaline juice is the most advantageous.

Juices which have been allowed to settle in special clarifiers do not need elimination, for in this case the juice can be neutralized directly in the defecators.

D.—TREATMENT OF THE SCUMS

The scums from the clarifiers, or from the settling tanks, are run into scum tanks as already mentioned. Sometimes the scums are allowed to settle once more, and the clear juice is again drawn off, but generally the scums are mixed with lime and steam to promote filtration. Many factories only possess a single scum tank, into which the scum continually flows from the clarifiers; it is heated and limed from time to time, and pumped off to the filters, but in this way it is impossible to know whether the scum is sufficiently limed or not. It is much better to have two tanks, and use them alternately, so that the first can be properly limed, steamed and emptied, while the second is being filled. Or instead of this, a mixer can be put in front of the steaming tank in which the scums are limed.

If the juice has been properly tempered and at a proper temperature, the scums can be filtered without further addition of lime; but such an addition helps if clarification has been defective.

The application of continuous settling tanks has greatly contributed to the obtaining of a thick and compact mud, as we have already mentioned, when figures of only 5 per cent. of scums were referred to.

Thomas and Petree have utilized this opportunity to introduce a method by which the thickened mud is diluted with juice of ever decreasing saccharine content, until it is finally poured out along with the fibrous part of the cane.

The application of this system in conjunction with a crusher and four mills may be described as follows: the juices from the crusher, first and second mills are limed and sent through a heater, followed by a passage through a defecation pan. From this pan the juice goes directly to a Dorr clarifier. From this primary clarifier the juice goes directly to the evaporator supply tank. The mud from the primary clarifier is pumped back to the mills, where it is mixed with the juice of the third mill and limed automatically. The mixture is then sent through a heater and then to the secondary clarifier. The clear juice which has a low quotient of purity can be sent either back through the primary clarifier or through the maceration line, back to the first mill.

The mud from the secondary clarifier is mixed with the juice from the last mill and limed, this mixture being then pumped back on to the bagasse behind the second mill.

All the maceration water is used behind the third mill. About 16 per cent. of maceration is amply sufficient to give an extraction of about 93 per cent.

E.—INFLUENCE OF DEFECATION ON THE CONSTITUENTS OF THE JUICE

The quantity of impurities separated from the juice by defecation is not considerable, since the weight of wet mud (chiefly consisting of juice) does not amount to more than 1 per cent. of the weight of the cane.

Effects of Defecation.—The effect of the defecation on the constituents of each of the three mill juices is represented in the following table :—

Constituents.	First Mill.		Second Mill.		Third Mill.		
						After.	
	Before.	After.	Before.	After.	Before.	Little Lime.	Much Lime.
Brix	19·2	19·2	19·3	19·7	19·0	19·4	19·2
Sucrose	16·49	17·06	16·33	17·06	15·95	16·40	16·50
Reducing Sugar ..	1·98	2	1·57	1·58	1·52	1·52	1·41
Gums and Pectin	0·125	0·071	0·376	0·120	1·250	0·840	0·38
Albumin	0·025	—	0·092	—	0·054	—	—
Quotient of Purity	85·9	87·5	84·4	86·6	84	84·5	86·0
Glucose Ratio ..	12·1	11·7	9·6	9·2	9·5	9·3	8·6

Except for the flocculation of colloids and the precipitation of suspended matter, nothing special has occurred, so that the dissolved constituents are not affected either by the common defecation process or by the application of sulphurous and phosphoric acids.

Gummy Matter and Pectin.—Gum is not precipitated by lime in slightly alkaline liquids, for a solution containing 15 per cent. of sucrose and 0·121 per cent. of cane gum, after ordinary defecation, still contained 0·120 per cent. of gummy matter, i.e., the original amount. This percentage did not fall after liming, heating, and treating with sulphurous or phosphoric acids, etc., showing that defecation does not remove the cane gum which is extracted from the cane fibre under heavy pressure.

The dissolved pectin is to some extent precipitated during defecation. A juice which contained 0·634 per cent. of total gums and pectin in the raw state, contained only 0·342 per cent. of these after defecation. Maxwell* found that a large quantity of gum was separated by liming cold, while Winter† mentions a slimy precipitate formed in limed juice, which proved to consist of a compound of lime and a carbohydrate. It contained galactose compounds, but as this substance differs much from galactan, it was more probably a lime-salt of pectin. Xylan was not present, otherwise it would have been recognized among the products of hydrolysis by the melting point of the osazones, but this was not the case.

* Report of the Hawaiian Exp. Station, 1896, 54.
† "Uit het chemische Laboratorium," 49.

Nitrogenous Bodies.—Maxwell* ascertained the percentage of nitrogenous matter (amide and albuminoid nitrogen) in juice during the different stages of manufacture. Clarified juice contained 2·1 parts of nitrogenous matter and syrup 2 parts on 100 of dry substance, so that a clarified juice of 16° Brix contained 0·32 per cent. of nitrogenous bodies or about 0·05 per cent. of nitrogen.

Van Lookeren Campagne† found much lower percentages of amides and other nitrogenous substances, varying between 0·044 and 0·082 per cent. in undiluted juice, and in diluted mixed juice between 0·070 and 0·016 per cent.; so that the nitrogen calculated from these data does not exceed 0·02 per cent.

According to Maxwell, in 100 parts of total nitrogen there are :—

	Albuminoid Nitrogen.	Amide Nitrogen.
Mill juice	35	65
Diffusion juice (cold)	24·5	75·5
Diffusion juice (hot)	10·9	89·1
Clarified juice	3·3	96·7
Syrup	3·2	96·7

And according to Van Lookeren Campagne, the following were the figures for the nitrogenous constituents :—

Description of the Juice.	Total Nitrogen.	Nitrogen × 6·25.	Albuminoids.	Amides and similar bodies.	Albuminoids on 100 nitrogenous matter.
Diluted mixed juice (factory mill) ..	0·024	0·150	0·057	0·093	38
,, ,, ,, ..	0·024	0·150	0·063	0·087	42
,, ,, ,, ..	0·028	0·175	0·059	0·116	34
Undiluted cane juice (test mill) ..	0·021	0·131	0·061	0·070	47
,, ,, ,, ..	0·026	0·162	0·080	0·082	49
,, ,, ,, ..	0·015	0·097	0·053	0·044	55
,, ,, ,, ..	0·020	0·125	0·058	0·068	46

* "Bull. Assoc. Chim. Sucr. et Dist.," 1895, 371.
† "Archief voor de Java Suikerindustrie," 1899, 755.

The diluted juices were not extracted from the same canes as the undiluted juices; the latter being obtained by a laboratory mill and therefore contained less albuminoids (see page 130).

A juice containing 0·057 per cent. of albuminoids lost 0·047 per cent. or 82 per cent. of the total on defecation, and another juice lost 0·067 out of 0·080 or 84 per cent. of the total, so that 17 per cent. passed over into the clarified juice.

Influence of Sulphitation on the Juice Constituents.—The influence exerted by sulphitation does not differ greatly from that obtained by the common defecation process. The heavier precipitate, however, removes more colloidal matter from the juice, and among this all of the colouring matter from the rind, causing the clarified sulphitated juice to be of a lighter colour than when obtained by simple defecation only.

III.—Clarifying Agents

A.—LIME AND LIME-CREAM

Source of the Lime.—Most factories which clarify their juice by the defecation process require so little lime that it is not worth while burning it themselves. As the quantity is so small and lime is the only clarifying agent in cane sugar manufacture, it is evident that great care should be taken to use the purest and best available, and it is better to spend a little more money in order to obtain a pure brand than to use a cheap but impure lime, as the difference in expense is so trifling that it cannot enter into comparison with the heavy osses occasioned by the use of bad lime.

As lime rapidly absorbs moisture and carbonic acid from the atmosphere, it is advisable to keep it in closed packages, by preference in soldered tins, but not in baskets, much less in open heaps, as this absorption is very detrimental.

Test of a good Lime for Clarification.—Lime used for clarification should answer the following tests. When mixed with half its own weight of water it should become very hot within a few minutes; the slaked lime after the addition of ten times its weight of water should form a soft cream, which on being passed through a fine sieve should not leave behind more unslaked particles than one-tenth of the original weight, and most of these particles should become soft after an hour's standing in a moist condition. The lime, after

being slaked, should dissolve in hydrochloric acid without appreciable effervescence, and not leave more than 2 per cent. insoluble matter.

The maximum percentages of impurities should be :—

	Per cent.
Moisture	2
Silica	2
Iron oxide and alumina	2
Magnesia	2
Carbonic acid	2
Sulphuric acid	0·50

Impurities of the Lime.—If burnt at too high a temperature it sinters somewhat and slakes with difficulty or not at all, thus losing its efficiency. Pure lime, however, requires such a high temperature before it is over-burned as can hardly be obtained in an ordinary lime-kiln, and lime becoming over-burnt at a relatively low temperature is commonly impure. For if the limestone (from which the lime is prepared) contains silica together with alumina or iron oxide, fusible silicates are formed and the lime is very liable to become over-burnt. Usually, the appearance of the pieces of lime indicates whether it has been over-burnt, and those pieces are generally picked out and removed at the lime-kiln, so that the buyer very seldom finds them in the lime supplied him. Water and carbonic acid, though not harmful, decrease the effect of a given quantity of lime, and the author has, therefore, fixed limits for these and also for the insoluble impurities, which consist of sand or clay. Iron oxide and alumina give rise to the formation of scale in the evaporators, those caused by alumina being remarkable because formed from soluble aluminates during evaporation.

Magnesia is a very troublesome impurity in lime, when present in appreciable quantity. It discolours the juice and retards subsidation, so that the decanted juice always contains much suspended matter, which forms scale in the evaporating vessels. For this reason the maximum allowed should be fixed at 2 per cent. Sulphates also scale the evaporators and should not occur in any appreciable quantities in the lime used for clarification. Alkalis, on the other hand, are harmless and usually only occur in small proportions in limestone, or may be totally absent. A few analyses of lime follow on page 167.

Nos. 3, 4, 13 and 15 are unsuitable for clarification because of their high magnesia content : Nos. 8, 10, 11 and 12, although they do not contain injurious constituents, are yet of inferior quality owing to their low percentage of calcium oxide. All the other ones may be used without objection.

Methods of adding the Lime.—The lime is added to the juice in different ways, generally as lime-cream, or milk-of-lime, but sometimes as slaked lime in the form of a paste.

ANALYSES OF LIME.

No.	Moisture.	Calcium Oxide.	Magnesia.	Sand and Silica.	Iron Oxide and Alumina	Carbonic Acid.	Sulphuric Acid.	Undetermined.
1	--	—	0·50	0·08	trace	—	—	--
2	0·75	98·08	0·63	0·10	0·44	--	—	—
3	4·33	82·07	13·29	—	—	—	—	0·31
4	—	80·19	16·38	—	—	—	—	—
5	4·29	95·12	0·15	0·18	0·22	—	—	—
6	1·97	96·67	0·51	0·21	0·35	—	0·14	—
7	—	97·76	0·35	—	0·10	—	—	—
8	—	87·20	0·20	1·80	0·84	3·40	—	—
9	0·21	98·56	0·15	0·56	0·52	—	—	—
10	3·04	89·70	0·52	0·98	2·74	3·02	—	—
11	—	87·10	trace	0·20	0·54	4·10	—	—
12	7·59	89·93	0·98	0·24	1·26	—	—	—
13	0·90	91·98	5·84	0·83	trace	—	—	0·35
14	0·25	98·65	0·69	0·12	trace	—	—	0·29
15	0·71	88·44	5·15	4·86	0·23	0·20	—	0·41
16	0·34	98·48	0·72	0·14	trace	—	—	0·32
17	0·90	96·17	1·50	0·52	0·22	0·45	—	0·24
18	0·68	95·68	2·08	0·12	0·20	0·52	—	0·72
19	3·12	91·31	1·40	0·96	1·30	1·40	—	0·51
20	5·44	86·90	1·56	0·51	1·25	3·78	—	0·46

Lime-cream.—The pieces of lime are placed in a shallow iron tank and treated with water. It is not advisable to throw them in a tank full of water as they are thereby cooled too much and do not get properly slaked. After the action is over, the still hot pasty mass is diluted with water, and the milk-of-lime now obtained is deprived of the stones or unburnt particles by passing it through several sieves with decreasing meshes. Then the lime-cream is allowed to settle, the supernatant water (which possibly may contain some dissolved matter) is run off, and the remaining lime is diluted to the desired density.

Slaking the Lime.—It is necessary to mix the lime with water some time previous to use, in order that the hard particles of lime may soften and become slaked. This is especially necessary in factories using the sulphitation process, wherein large quantities of lime are added to the juice which, although neutralized by the sulphurous acid, yet if it contains hard particles are

not attacked by the acid, but remain as quicklime in the juice. Afterwards, they gradually dissolve and impart an alkaline reaction to the juice.

Mixing and Straining the Lime.—The slaked lime, mixed with water to the consistency of a thick cream, is poured through coarse strainers into two tanks, which are filled and emptied alternately. When one of these tanks is filled and diluted to the desired density of 15° or 20° Baumé, this tank is drawn from until empty. A fresh supply is meanwhile prepared in the second tank so as to be in readiness when the first is empty; this ensures only fresh and well-slaked lime being added to the clarifiers.

The contents of the tanks must be kept in steady motion to prevent subsiding of the lime. In factories where only little lime is used, this may be done by stirring the lime-cream with a paddle, but when using much of that clarifying agent the lime-cream is constantly pumped through a pipe which passes near the mixing tanks and returns to the supply tank. The lime-cream is thus forced from this tank, through the pipes and back again. When lime is to be drawn off and added to the juice, a cock in this pipe is opened, otherwise it continues on its way back to the supply tank and is thereby kept in constant motion and no opportunity given for any lime to deposit.

The lime contents of lime-cream corresponding to the readings of the Baumé hydrometer are as follows:—

DENSITY OF LIME-CREAM.

Degrees Baumé.	Weight of 1 litre Lime-cream.	Grms. CaO per litre.	Per cent. CaO.	Degrees Baumé.	Weight of 1 litre Lime-cream.	Grms. CaO per litre.	Per cent. CaO.
	Grms.				Grms.		
1	1007	7·5	0·75	16	1125	159	14·13
2	1014	16·5	1·64	17	1134	170	15·00
3	1022	26	2·54	18	1142	181	15·85
4	1029	36	3·50	19	1152	193	16·75
5	1037	46	4·43	20	1162	206	17·72
6	1045	56	5·36	21	1171	218	18·61
7	1052	65	6·18	22	1180	229	19·40
8	1060	75	7·08	23	1190	242	20·34
9	1067	84	7·87	24	1200	255	21·25
10	1075	94	8·74	25	1210	268	22·15
11	1083	104	9·60	26	1220	281	23·03
12	1091	115	10·54	27	1231	295	23·96
13	1100	126	11·45	28	1241	309	24·90
14	1108	137	12·35	29	1252	324	25·87
15	1116	148	13·26	30	1263	339	26·84

The temperature should not fluctuate much from 30° C. The lime-cream generally used is so thick that an accurate estimation of the density is somewhat difficult, so some prefer to dilute a sample with its own volume of water and then determine the density with the Brix hydrometer.

The composition of the undiluted lime-cream corresponding to the degrees Brix in the diluted state is given here.

Degrees Brix of the DilutedLime-cream	Grms. per litre CaO of the Undiluted Lime-cream.	Degrees Brix of the Diluted Lime-cream.	Grms. per litre CaO of the Undiluted Lime-cream.
9·0	93·20	14·5	150·9
9·5	98·60	15·0	156·0
10·0	104·00	15·5	161·2
10·5	109·00	16·0	166·2
11·0	114·00	16·5	171·3
11·5	119·1	17·0	176·3
12·0	124·3	17·5	181·0
12·5	129·4	18·0	186·9
13·0	134·5	18·5	192·8
13·5	140·1	19·0	198·5
14·0	145·7	19·5	203·7
		20·0	209·0

Slaked Lime in Paste Form.—Lime has sometimes been added to the juice in the form of a paste, portions of which are weighed and placed in the gutter through which the cane juice flows to the measuring tanks. The advantages of this system are not great, and manufacturers are advised to adhere to the old practice of using lime-cream.

B.—SULPHUROUS ACID

Preparation of Sulphurous Acid.—The sulphurous acid, which is applied during the clarification of cane juice, is obtained by burning sulphur, the use of the compressed fluid sulphurous acid being too troublesome and expensive in tropical countries owing to the transport of the heavy cylinders in which it is contained. The raw material for sulphurous acid consists of refined sulphur, which is practically pure, and as it never contains more than 0·2 per cent. of foreign matter, chiefly earth, it burns without leaving any appreciable residue.

In some cases, however, sulphur has been supplied containing a very small amount of bituminous substances which have collected as a film on the surface

of the molten sulphur, and notwithstanding its small bulk prevented combustion. As there is no remedy for this fault, the sulphur has had to be replaced by a purer material.

In sugar factories the sulphur is burnt in iron ovens, which may be cooled by means of a water-jacket on the top, the pipes through which the fumes escape being also provided with a water-jacket in order to condense the volatilized sulphur, and thus prevent the narrow pipes from being choked with sublimed sulphur. The sulphur is placed on an iron ladle, which is thrust into the oven after the sulphur is ignited. From time to time the door of the oven is opened to add a fresh supply of sulphur. If the sulphur does not burn well, the combustion may be accelerated by placing a red-hot iron upon it. In some systems air is forced over the sulphur by an air compressor, while in others an injector in the discharge pipe sucks off the gases and causes a draught of fresh air over the burning sulphur. It is very important that the air passing over the sulphur be dried by quicklime in a special air-dryer, since moist air promotes the formation of sulphuric acid from the sulphurous acid and oxygen of the air, which corrodes the oven and the pipes.

The damage done by neglecting to dry the air may be shown by the analyses of crusts and scales in the lead and iron discharge pipes of sulphur ovens in which the air has been allowed to enter undried.

SCALES IN THE TUBES OF A SULPHUR OVEN

Constituents.	Scales from Tubes.	Scales from Tubes.	Scales from an Oven.
Ferrous sulphate	—	—	68·30
Ferric sulphate	35·00	58·15	trace
Copper sulphate	25·00	—	—
Lead sulphate	0·90	—	—
Iron sulphide	—	—	1·10
Arsenious acid	1·20	—	—
Free sulphur	5·80	12·72	20·77
Free sulphuric acid	—	12·20	1·25
Iron oxide	4·70	—	—
Water of crystallization	7·20	14·78	—
Hygroscopic moisture	20·20		6·64

Drying the Air.—The air is dried by passing it through a box provided with trays filled with lime. As soon as the lime is saturated with water on one of the trays it is removed and replaced by another one containing fresh lime, so as always to have an abundant supply of hygroscopic material in the current

of the air. When the air is forced over the sulphur by means of an air compressor, it is advisable to have this compressor between the dryer and the sulphur oven, for if the dryer is between the compressor and the oven, the fire will become extinguished whenever the drying box is opened for changing the trays of lime, as the air supply to the oven is then stopped.

Composition of Sulphur Fumes.—The sulphurous acid content of the gas passing out of the oven cannot theoretically exceed 21 per cent., but this figure is never attained, the gas usually containing 14 per cent. of sulphurous acid ; sometimes it may rise to 16 per cent. or fall as low as 12 per cent. before the fire goes out.

Sulphites.—Instead of sulphurous acid, acid sulphites have been used for clarifying cane juice. In Icery's process sulphurous acid was combined with lime in wooden troughs, and the resulting solution of calcium bisulphite used as a clarifying agent. Later on, sodium bisulphite was occasionally tried and also magnesium bisulphite, but these agents have never come into regular use, being discarded after a few preliminary trials.

C.—PHOSPHORIC ACID AND ITS PREPARATIONS

Analyses of a sample of commercial crude phosphoric acid and also of samples of two acid calcium phosphates, are as follows :—

		I. Soluble Phosphoric Acid.	II. Acid Calcium Phosphate.	III. Acid Calcium Phosphate.
Mono-calcium phosphate, $Ca H_4P_2O_8$	soluble	2·27	28·19	38·18
Mono-ferric phosphate, $Fe_2(H_4P_2O_8)_3$	soluble	7·75	12·74	2·63
Phosphoric Acid, H_3PO_4	soluble	45·91	18·53	15·89
Calcium Sulphate, $CaSO_4$	soluble	—	4·93	1·53
Tricalcium phosphate, $Ca_3P_2O_8$ ..	insoluble	—	4·44	2·73
Ferric phosphate, $Fe_2P_2O_8$ ..	insoluble	—	2·90	6·91
Sand	insoluble	—	0·73	1·10
Moisture	insoluble	44·07	27·54	31·03
		100·00	100·00	100·00
Free and half-combined P_2O_5 ..		36·65	29·93	27·90

The acid calcium phosphates are known under various names, such as "Ehrmannite," "Newlanite," "Claryphos," "Clarine," etc., etc. They are dry or pasty preparations, the latter having a varying moisture content.

In more recent times these preparations have been supplanted by pure mono-calcium phosphate ($CaH_4P_2O_8$), which is supplied in the shape of a dry powder. Further, extensive use is made of mixtures consisting of acid phosphates with infusorial earth of "Filtercel." "Phospho-gelose" consists of from 25 to 50 per cent. of kieselguhr, and from 75 to 100 per cent. of technically pure insoluble dicalcium phosphate ($Ca_2H_2P_2O_8$). According to information obtained from the inventor, the proportion of 50 per cent. of either constituent has proved the most favourable for use.

An authentic sample analysed by the author showed this composition :—

Dicalcium phosphate, $Ca_2H_2P_2O_8$	44·50
Ferric diphosphate, $Fe_2P_2O_8$	1·50
Calcium sulphate, $CaSO_4$	2·40
Kieselguhr	44·90
Moisture	6·70
	100·00

Another preparation called "Suma-phos" contains about 36 per cent. of water-soluble phosphoric acid and 15-20 per cent. of fine kieselguhr, and is used for the same purpose, viz., to cause a voluminous precipitate which entrains and removes colloidal matter.

IV.—Filtration

In cane sugar factories we meet with two different kinds of filters, viz., juice filters for the filtration of juice, and scum filters for use when the juice is separated from the scum.

1.—JUICE FILTERS

The simplest form of juice filter consists of a bag made of cloth, into which the juice is poured, the clear juice passing through and the impurities being kept back. Such are the Puvrez filters, consisting of a cylindrical bag, of which one end is closed and the other tied round a pipe supplying the juice to be filtered, which enters into the bag under a certain pressure. The impurities are kept back, and the filtered juice passes through the cloth and flows

into an iron gutter to which the bag is fixed, and so is conveyed to the suction tank of the evaporators. A great inconvenience of this filtration from inside to outside is that the finer impurities also are driven between the pores of the cloth and choke them up, so that the bag does not filter for long before becoming full of scum, and then has to be changed. Consequently filters of this description have now been generally abandoned, and other systems have been adopted in which filtration takes place from outside to inside, among which may be mentioned the Danek, Kasalowsky, Philippe filters, etc.

In these systems the filter consists of a square iron vessel on which an iron plate is screwed as a cover. In this vessel are a certain number of chambers lined with cloths, kept apart by perforated plates or metal spirals fitted in the chambers. Each chamber is therefore shut off by cloth at the sides and at the bottom, and the upper edges are pressed tightly together, thus preventing the juice, with which the filter is filled, from entering the chamber without passing through the cloth. Each chamber has a spout for the discharge of filtered juice, and a large cock at the bottom of the filter enables the workman to empty the whole of the contents when it has to be cleaned. When starting, the chambers are supplied with new or clean cloths, the filter is closed with the cover, the screws are tightened, and the hot juice is admitted under a head of three to six feet, avoiding great variations. The juice passes through the cloth, leaving behind the suspended particles, which sink to the bottom of the filter and do not interfere with the filtration through the cloth. The latter can therefore be used for a long time, but when it is dirty it is changed for a new cloth, and as soon as the contents of the entire filter have become too muddy owing to the impurities deposited, it is emptied by means of the cock at the bottom ; the juice is conveyed to the scum tank, while the filter is washed and made ready for use again.

In many cases, the floating impurities are so fine or gummy that filter-cloth is unable to retain them, though much may be done in this direction by keeping the juice hot and maintaining a good pressure in the filters. On this account juice filters have been discarded in most factories, and manufacturers prefer to evaporate their subsided juices unfiltered.

However, there is no doubt that juice, even if allowed to subside for a long time, always contains many floating and suspended particles which deposit on the tubes.

It is not inconceivable that a copious addition of infusorial earth or finely-powdered carbon to the juice during defecation would remove so much colloidal matter that the portion of juice syphoned off could be filtered through cloth.

Zerban*, who mixed juice with about 0·5 per cent. of kieselguhr and raised its temperature to boiling point, found that filtration could be conducted without difficulty, the liquid obtained after this operation being brilliant,

* Louisiana Bulletin, No. 173.

though not very greatly improved in colour. Analysis showed that the treatment with kieselguhr raised the quotient of purity as much as sulphitation generally does, viz., about 0·4 degrees.

Helderman and Khainowsky* treated raw juice with 2 to 3 per cent. of fine carbon, and in this way obtained a juice which filtered easily.

These are, however, laboratory experiments only, and up to now filtration of subsided defecated juice through cloth has not made great strides in practice.

Neither has filtration through bagasse, palm fibre, and similar material survived the trial stage.

Sand Filters.—Instead of bagasse or the above fibre, inventors have suggested pulverized cork, but the dirt is not retained by this material. Better results are reported of the sand-filters of the Danek and Abraham types. In both these systems, the juice is forced through a layer of quartz sand, having a large filtering area, which can, therefore, do more work than cloth. When the filter becomes clogged by a layer of dirt, a new filter-bed may be obtained by simply agitating the uppermost layer of sand. Only when the whole of the sand is saturated with dirt is the juice let off and the sand washed by a strong jet of water. The first washings are transferred to the scum tank, while the later and diluted ones are thrown away.

The sand retains a considerable quantity of slimy material and very fine bagasse fibre in a semi-colloidal state, and, therefore, diminishes the formation of scale in the first vessels of the multiple evaporation plant.

Composition of Dirt retained by Sand Filters.—The composition of a sample of slime scraped from a dirty sand-filter is as follows, calculated on the dry substance :—

	Per cent.
Cane fibre	69·1
Albuminoids	6·9
Wax	7·7
Ash	16·3
	100·00

The ash consists of :—

	Per cent
Calcium silicate	5
Calcium phosphate	8·8
Calcium carbonate	2·2
Undetermined	0·3
	16·3

* "Archief voor de Java Suikerindustrie," 1921, 1235.

A defect of sand-filters is that they require a special kind of sand which is rather expensive, and although the material may be used again, after washing, there is always a certain amount of waste. It is not advisable to use other kinds of sand, which may possibly differ in efficiency and thus spoil the work.

Bronze Gauze.—The subsided juice may also be filtered through fine bronze wire-gauze, such as is used for " bolting " flour. This gauze contains about 2,000 meshes per square centimetre, the diameter of the wire being 0·093 mm., the width of the meshes 0·137 mm., and the composition 93 per cent. copper and 7 per cent. tin. It does not require any intricate system of filtering chambers, but can be used as a strainer through which the decanted subsided juice is caused to pass. The juice passes very rapidly through this gauze and is received in a tank underneath, leaving a thick layer of fine bagasse fibre and slime on the strainer, which is scraped off from time to time. As the gauze is very delicate and liable to break during cleaning, it is advisable to place the strainer in an inclined position with a strip of copper plate at the lower end, and also to protect the fine gauze by a piece of " bird's cage " gauze, so that the scraper cannot touch the former. The dirt gradually sinks towards the strip of copper plate below, and may be removed from this without damaging the soft fine gauze.

A portion of dirt retained by the wire gauze gave this analysis after being washed and dried :—

Gummy and fibrous substance		95·60
Ash		4·40
The ash contained	Silica	2·54
	Calcium phosphate	1·30
	Calcium carbonate	0·56

Cane fibre generally contains 2·50 per cent. of ash, of which 1·75 is silica, 0·20 calcium phosphate, and 0·10 per cent. calcium carbonate, so that the dirt retained by the wire gauze also contains an appreciable quantity of inorganic substances, which would otherwise have caused scale in the evaporating vessels. As this strainer is easily fitted up and inexpensive, it is advisable to use one for straining the whole of the juice before it enters the evaporating vessels.

Another way is to have a wire cylinder coated with bronze gauze, and made to revolve round a hollow axle in a tank, into which the juice to be filtered is pumped.

The juice on its way out through the axle has to pass the wire gauze, on which it leaves its floating impurities ; these drop to the bottom and may be removed from time to time by a cock or drain. The use of centrifugals for separating the dirt has already been mentioned on page 149.

2.—SCUM FILTERS

Bag Filters.—The most primitive method of treating scums was by pressing them in closed linen bags, which were placed in a box provided with a perforated bottom, the pressure being assisted by a cover loaded with stones. A part of the clear juice was thus forced through the cloth of the bags, after which the remainder was expressed in a press. This operation demanded much labour, was dirty, and also unsightly.

Taylor Filters.—Taylor filters are a great improvement, and consist of a number of long narrow bags, closed at the lower end, suspended in an iron chamber furnished with a receiver for the scum. Into the bottom of this receiver are screwed several rows of nipples, to which the above-mentioned bags are fixed. The receiver being filled with the scum, the latter runs into the bags, which retain the impurities, while the clear juice is filtered off and flows into the lower part of the apparatus, from which it is conducted by a gutter to the suction tank of the evaporators.

The front of the apparatus is provided with a door to allow the bags to be changed, but as this work proved troublesome in the hot, narrow chambers, the upper receiver has been made movable in later patterns, so that it may be hoisted with all the attached bags by means of a travelling conveyor and carried outside the factory. The hot bags can thus be changed out of doors, and while this is being done a second receiver fitted with clean bags is placed in the box, so that the filtering can go on without much interruption.

When the bags are full of mud they are detached from the nipples tied round the top, pressed, emptied and washed. Sometimes their contents are washed out by pouring water into the bags, but this operation is generally omitted because the water penetrates the slimy dirt very slowly.

As the filtration proceeds from the inside outwards, the same inconvenience is met with as in the Puvrez filters, though not to so great an extent, as the pressure is much less and the scum more compact. The disadvantage that the juice cools very rapidly owing to the great surface of bags exposed to the air, and hence does not filter so quickly, or even becomes acid, can be remedied by blowing a constant jet of exhaust steam into the chamber, which keeps up the temperature, prevents sourness, and increases the capacity by 20 per cent.

Filter-presses.—In place of these filters most factories now use *filter-presses*, which consist of metal chambers lined with cloth, and screwed tightly one against the other. A pump or monte-jus, or air compressor, fills them with the hot scum. The juice passes through the cloth and escapes through suitable channels, while the solid matter remains behind in the form of hard cakes.

As every chamber has its own discharge pipe, which can be closed by a cock, any of them can be stopped working if the filtered juice should become turbid owing to a hole or leak in the filter-cloth.

Each chamber has an opening through which water or steam can be applied to wash and steam out the scums. The water or the condensed steam penetrates through the cake of scum and replaces the juice still contained therein, which flows off through a drain-pipe that can be shut off by a cock.

The oldest filter-presses consisted of plates having a central opening through which the juice entered the press and which at the same time served for fastening the cloths. This system was abandoned partly because the continuous fastening and loosening of the screws occupied too much time, but chiefly because the cloths wore away very rapidly at that spot and had to be continually mended. Therefore the inlets for juice, for the wash-water, and for steam were all placed on one side of the plates, but then the holes in the cloths were all on the same side, and leakages could not be prevented. Kroog constructed filter-presses in which the inlets for juice, water, and steam, were situated in the upper corners of the plates, and were thus outside the actual filtering surface. These inlets were joined by india-rubber rings or by pieces of cloth provided with a circular opening. Beeg's filters are distinguished by the special construction of the frames (with fine corrugations), rendering superfluous the sieve plates which are to be found in other systems. The cloths are fastened by bronze springs, which grip as soon as the press is closed, the grip being greater the tighter the press is screwed.

The scum should enter the filter-press as hot as possible, having an alkaline reaction, and at a pressure of from 1½ to 2 atmospheres. When the chambers are full of scum-cake, as indicated by the fact of no more juice flowing off, the supply of scum should be stopped, the cakes washed with water or steam, the press opened, and the cakes finally removed. The cloths are changed if they have become too dirty, after which the press is again closed and is ready for further use.

Difficulties in getting Hard Cakes.—It sometimes occurs that scums, even when filtered hot, under sufficient pressure, and with a distinctly alkaline reaction, do not yield hard cakes, filling the whole chamber ; but soon after the filters have started, filtration stops and the chambers are found to be filled with a thick mud, containing much juice, which goes to waste. This may be caused by wax deposited from the hot scums upon the cold cloths, which become clogged with that substance. In order to avoid this, it is advisable to steam the press before introducing the scums.

The scums may also be too slimy, and this is best remedied by using a coarser strainer for the juice, which allows more of the fine bagasse to pass into the juice and renders the scums more porous and capable of being pressed dry. It is, however, advisable not to go too far in this direction since every addition

of fibre increases the amount of scums and the loss of juice in them, while the capacity of the presses decreases accordingly.

Since the fine bagasse imparts colouring matter to alkaline juices it is, in our opinion, much better to use an insoluble and neutral substance, if it is thought necessary to increase the porosity of the mud.

Zerban* recommends the use of kieselguhr or " Filtercel " for that purpose : the well-strained juice from the mills goes to whole juice tanks, where " Filtercel " or kieselguhr is added, after which it is run through juice-heaters at 100°-102° C., and then to a sump before running to the press feed tanks. Two juice tanks are provided to feed the presses. One is used as a main pressure feed tank, and the other as a low pressure feed tank for " pre-coating " or filming the press-cloths with kieselguhr. The whole juice from the juice-heaters is run into both the main feed tank and the pre-coating tank. When a new press is to be cut in, the charge in the pre-coating tank has added to it an extra quantity of kieselguhr to supply the pre-coating. The quantity of kieselguhr necessary to film a 750 sq. ft. press is about 75 pounds ; this may be cut down at times to less than 50 pounds, or about 5 to 10 pounds of " Filtercel " per 100 sq. ft. of filtering area. The pre-coating tank should be so located that the heavy " Filtercel " liquor can be run into the press under a 15-20 foot gravity head or placed below the filters, in which case a centrifugal pump is so arranged that the pressure will not go above 15 pounds. After this liquor has been run into the press and has covered the cloths with a uniform coating of kieselguhr, the pressure liquor is then pumped through in the regular way.

Apart from qualities naturally inherent to the character of the juice itself, we know also of an instance† where in a factory filtration suddenly encountered great difficulties, which could not be overcome notwithstanding that all measures imaginable had been applied. The mud remained liquid and slimy, and did not detach from the cloths after the press had been opened. After a lengthy examination, it was found that a growth of *Leuconostoc* had accumulated under the sulphurous acid supply tubes, and had carried such large amounts of suspended gummy matter into the scums that filtration was rendered impossible. After thorough disinfection of tanks, gutters, suction tanks, pumps, etc., with a 1 per cent. solution of ammonium fluoride, the formation of dextran was checked and filtration of the scums went on as usual.

The absolutely neutral attitude of the dextran against the common reagents had allowed its presence to remain undetected for a long time till a special investigation happened to discover it.

* " Bulletin 173, Louisiana Exp. Station."
† W. C. Nieboer, " Archief voor de Java Suikerindustrie," 1914, 1657.

Washing the Scums.—Formerly it was the custom only to steam the mud in the presses in order to drive the residual juice from between the particles of the cake, and this did not dilute the juice. The effect of this procedure was not a marked one, as filter-press cakes having a sucrose content of 5 to 9 per cent. were obtained and run to waste.

In recent years, however, much attention has been paid to the recovery of the juice from the mud, and has led to an almost thorough exhaustion of the scums without re-solution of non-saccharine matter.

In many factories the filter-press cakes are taken out of the press in an unwashed state and mixed with a fresh amount in some form or other of macerator, e.g., the Ledocte Malaxeur. This is a revolving drum in which the cakes are stirred with water, after which the diluted mud is again filtered in presses. It was feared that a great deal of precipitated impurities might be re-dissolved, so that the repressed juice would be of a very low quotient, but careful investigations showed that no such contingency was to be feared.

Allen* has lately published the results of his experiments with single and double pressing of scums, and understands by the latter expression the operation in which scums from the clarification of sugar juices are discharged directly from the presses without being in any way washed while in the frames, and then mixed with water and re-pressed. This he compares with the results obtained by sweetening-off the press-cake in the frames with hot boiler feed-water.

The average washing time was slightly over an hour. The results were as follows :—

	Sugar per cent.	Moisture per cent.	Loss in Cake per cent. Cane.	Drop in Purity.	Dilution per cent.
Single pressing ..	3·925	55·15	0·049	4·035	39·37
Double pressing ..	1·233	59·03	0·017	3·161	42·00

The Java Sugar Experiment Station recommends the following instructions for sweetening-off the cakes in a plate-and-frame press.

The indispensable requirements for a good and reliable sweetening-off are a very regular supply of mud-juice under a uniform pressure, so as to allow an even coating of the filter-cloths by an ever-increasing layer of scums. Further, the supply of scum-juice should not be pushed so far as to have the frames completely filled with scums, but an open space has to be left between the two inner surfaces in order to allow, later on, the washing water to act evenly on the entire surfaces on both sides. The current of the washing water runs in the same direction as that of the juice.

* "Int. Sugar Journal, 1924, 30.

When applying the system on a press of the Kroog type, a cross-piece or a double T-piece of at least 1½ inch is placed in the juice conduit directly behind the valve. It is connected with the steam and water conduits by two valves or cocks, which are situated as close as possible to the cross or T-piece.

Hot water should be used for sweetening off, and it is conducted in the same direction through the press as is the juice, and at no higher pressure than was used at the end of the filtration of the scum-juice.

When checking the density of the liquor running out of the press during sweetening-off, it may be observed that it remains constant during a long time at the same level as that of the filtered juice, but, as soon as the washing water has pushed the juice out, the density drops to about the zero point. In practical working the sweetening-off is not conducted so far as that, but is stopped at a density of 2°- 3° Brix, and the liquid still in the cakes is pressed off by means of live steam or compressed air.

The time taken to filter is generally 1½ to 2 hours; that of sweetening-off and steaming 25 to 30 minutes. The sucrose content of the mud may be reduced to under 1 per cent., equivalent to a loss of 0·1 part on 100 of sucrose in juice.

Instead of plate-and-frame presses, leaf-presses have lately come into use.

Kelly Press.—The Kelly press consists of an iron cylinder mounted on an inclined frame. A number of filtering elements made of wire-gauze and coated with filtering cloth are supported on a travelling carriage mounted on wheels inside the cylinder. The back of the cylinder is closed and the front consists of the cover at the end of the carriage. The outlets of the filtering elements protude through that cover. After the elements have been fixed in their proper places, the carriage is pushed into the cylinder and tightly screwed with bolts. Scum juice is introduced under pressure in the cylinder; it passes through the cloth, leaves the scum at the outside of the elements, and escapes filtered through the outlets. When the mud has accumulated in such a way that the crusts nearly touch one another, the cylinder is emptied into a tank by means of compressed air, the filter is filled with water which passes through the mud in the same direction as the juice, thereby replacing it in the scums and leaving them almost free from sugar.

Finally the water is discharged, the cover is opened, the carriage rolls out of the cylinder, and the mud is blown off from the outside of the elements by a jet of steam, which makes them swell and shake off the mud, after which the carriage may be returned into the cylinder again.

Experiments show an average sucrose content of the cakes of 0·52 per cent., and a moisture content of 78·2 per cent., corresponding to a loss of sugar in mud of 0·01 on 100 parts of cane or a quite negligible amount.

Sweetland Press.—In the Sweetland press the filter elements are placed transversely along the axis of the cylinder, and have the shape of a circular disc. When the filter has to be opened, the under part is taken down, allowing the cakes to be discharged and the cloths to be washed.

Vallez Filter.—The Vallez rotary filter has a cylindrical casing and a set of filtering bodies rotatably disposed in the casing. A channel for discharge in the casing is provided with a discharge opening at a point midway between the ends of the entire set of filtering elements. Each element comprises a metal frame, with projections and drainage recesses, and having a perforated plate and a filter-cloth or gauze fixed on each side. Passages in the hub of each element register with apertures in the shaft and allow the filtered juice to pass from the drainage recesses to an outlet pipe.

After the filter is full of mud the juice supply is shut off, water is allowed to wash the adhering crusts, which finally are removed from the elements by steam, water or compressed air.

Amount of Water for Sweetening-off.—Van der Linden* publishes the following table for the amount of sweetening-water used in the various systems of filters, together with the loss of sucrose in the mud :—

System of Filter.	Parts of washing-water for one part of insoluble solids in the mud.	Parts of clarified juice lost on 100 parts of insoluble solids in the mud.
Kroog	4·2	48
Halle	6·2	49
Halle	3·7	76
Kroog	2·6	80
Kroog	6·7	109
Kroog	5·2	119
Halle	4·2	99
Kelly	4·9	52
Kroog	4·0	46
Dehne	1·0	90
Taylor	5·7	291
Kroog	5·2	93

Composition of Filter-press Cakes.—In the first edition of this work, at a time when sweetening-off the cakes was still in its infancy, we quoted the following analysis of the old-time mud :—

* "Archief voor de Java Suikerindustrie," 1923, IV, 160.

Constituents.	Taylor Filter.	Taylor Filter.	Filter-press.	Filter-press.
Moisture	72·14	67·73	69·26	69·72
Sucrose	13·20	10·50	9·96	10·20
Reducing sugar	0·60	—	—	0·71
Ash	—	—	7·59	8·10
Albuminoids	1·93	0·87	1·94	1·80
Wax	—	3·04	5·13	4·12
Fibre	—	11·00	—	5·35
Lime	0·66	0·67	—	1·53
Magnesia	—	0·08	—	0·22
Iron oxide and Alumina	—	3·06	—	1·40
Phosphoric acid	0·57	1·49	1·42	1·39
Silica	—	0·21	—	0·37
Sand and Clay	1·38	1·70	—	2·82

Allen* quotes the composition of filter-mud, obtained by double pressing at Fajardo in Porto Rico, as follows :—

Moisture	60·331
Sucrose	0·647
Reducing sugar	0·011
Wax	7·760
Albumin	5·775
Calcium phosphate	1·073
Sand and Silica	6·506
Iron and Alumina	2·666
Magnesia	0·027
Fibre	10·860

Utilization of Filter-press Cakes.—The filter-press cake is usually employed as a fertilizer, and sometimes as fodder, or even as food for the poorer classes of people. In a few factories it is mixed with trash or fine bagasse and then used as fuel.

Some attempts have been made to extract the wax from dried press-cake on a commercial scale. Wijnberg† patented a process by which the dried and powdered filter-press cake was extracted by benzine and although from time to time reports are heard of the successful manufacture of a hard and fine wax from that source, it has not yet come into general application.

* "Int. Sugar Journal," 1924, 35. † *Ibid.*, 1909, 521.

CHAPTER III

CONCENTRATION OF THE JUICE

Principle.—Up to the stage now reached all the operations the juice has undergone have had for their object the removal of the non-saccharine matters and the clarification of the juice. This being accomplished as completely as possible, the sucrose now exists in a solution containing about 15 per cent. of dry substance, including those impurities which clarification has failed to remove. The purpose of the next operation is, therefore, to convert the sucrose into a solid in order that it may then be separated from the impurities still remaining in solution in the mother-liquor or molasses. This is effected by evaporating the juice until the small quantity of water left can no longer hold all the sucrose in solution ; this latter accordingly crystallizes out and can be separated from the mother-liquor and thus obtained in a pure state.

This withdrawal of water by evaporation must be done at once and without delay, as in a dilute solution the sucrose is very liable to decomposition by the action of micro-organisms, which cause inversion or sourness, and therefore give rise to considerable loss. It is hence necessary to deprive the clarified juice of the great bulk of its water as rapidly as possible, so as to bring it into a condition in which it is no longer liable to such decomposition, i.e., a crystalline mass containing 8 to 10 per cent. of moisture.

Concentration in Vacuo.—Since sucrose is liable to decomposition at high temperatures and also inversion, it is desirable to keep the temperature as low as possible during evaporation, and at the same time to reduce the time during which it remains at that temperature. This is effected by boiling the juice under reduced pressure, by means of which its boiling point falls, hence the present-day practice of boiling the juice *in vacuo*. Powerful pumps are used to draw off the vapours and maintain a vacuum.

Concentration performed in Two Stages.—The concentration of the juice is ordinarily effected in two stages, viz., *evaporation*, which concentrates the juice to syrup containing about 50 per cent. of solid matter, and *boiling*, which concentrates this syrup still further, and transforms it into a crystalline mass, so-called Massecuite, containing only from 8 to 10 per cent. of water.

The sole object of evaporation is to concentrate the juice to syrup as soon as possible, with a minimum waste of steam and sucrose and at a low temperature.

In the boiling process (during which the sucrose first assumes a crystalline form), additional care is necessary in order to obtain the maximum yield of crystallized sucrose in as pure a state as possible, and in such a condition as to allow it to be separated from the mother-liquor with the minimum loss and trouble.

Between these two stages the syrup is in most cases subjected to a second clarification, which is of considerable importance in the manufacture of white sugar, as it is impossible to obtain good white sugar from a turbid syrup.

Decomposition of Sucrose during Evaporation.—Though the evaporating and boiling processes are carried out very rapidly, it is not possible to avoid partial decomposition and caramelization; the juice is liable to become overheated locally, which is seen when a syrup or a massecuite is diluted to the density of the original clarified juice, and the colours compared, the colour of the former always being darker than that of the latter.

According to Gröger's* investigations, the increase in colour is strongest in the first vessel of the evaporators, i.e., that in which the temperature is highest, and it is much less in the subsequent vessels, in which the juice boils at lower temperatures. This point will be considered in greater detail further on.

Calculation of Weight of Water evaporated.—The amount of water to be evaporated may be calculated from the degrees Brix of clarified and concentrated juice by the formula :—

$$W = k_1 \left(1 - \frac{S_1}{S_2} \right)$$

in which

W = weight of water to be evaporated.
k_1 = weight of clarified juice.
S^1 = Brix of clarified juice.
S_2 = Brix of concentrated juice.

I.—Evaporation

Evaporation over Open Fire.—Formerly, the clarified juice was evaporated in shallow pans, known as the "Copper-wall," over an open fire, but this process had the inconvenience of utilizing the fuel very imperfectly, and it was also impossible to prevent local overheating, which caused a portion of the sugar to become decomposed. As the pans were open, and the impurities were constantly skimmed off, the mechanical purification was very good, but this advantage could not counterbalance the enormous losses of sugar and fuel.

* "Oesterr.-Ung. Zeitschr. für Zuckerind. und Landw.," 1898, 790.

Evaporation by Steam under Atmospheric Pressure.—Later the juice was evaporated in " Concretors," i.e., open pans, heated by steam, which secured a more economical utilization of the fuel, and avoided the risk of overheating, but they, too, were soon supplanted by multiple effect apparatus.

Evaporation in Vacuo.—In this system the juice boils *in vacuo*, and therefore at a lower temperature, so that inversion is no longer to be feared ; moreover, the employment of two, three, or even four vessels allows the heat of the steam to be utilized as completely as possible ; the vapour from the first vessel passes into the steam-drum of the second, that from the second heats the juice in the third, and so on, as will be seen from the following description.

The evaporating vessels generally in use consist of large iron cylinders, with dome-shaped heads, fitted with a wide pipe which carries off the vapours. The lower part of the cylinder is divided into three portions by two transverse plates pierced with holes, in which are fitted tubes which bring into communication the upper and the lower portions of the cylinder, together called the juice-chamber. The middle portion is called the steam-drum and is traversed by the tubes, but does not communicate with them. The steam-drum of the first vessel is provided with an inlet pipe for exhaust steam, and in most cases with a smaller one for direct steam, also with an escape pipe for the condensed water. The wide pipe which carries off the vapours from the juice is so constructed as to prevent particles of juice from passing off with the vapours, and communicates with the steam-drum of a second similar vessel, and so on, while an air-pump, attached to the vapour pipe of the last vessel, draws off and condenses the steam. The steam-drums are kept free from condensed water by means of pumps.

Working the Triple Effect.—Each vessel of a triple effect is filled with juice to the level of the upper tube-plate, so that the entire heating surface is covered by the juice to be evaporated.

Steam, having a temperature of over 100°, on being admitted to the steam-drum of the first vessel, causes the juice to boil, and becomes itself condensed. The vapour from the juice enters the steam-drum of the second vessel, and heats the juice contained in the juice chamber, where it is under a certain vacuum, and therefore boils at a lower temperature than that in the first vessel. The vapour formed here is similarly capable of boiling the juice in the third vessel, because this, being in immediate communication with the air-pump and the condenser, boils at a still lower temperature than the juice in the second vessel. The vacuum is therefore greatest in the last vessel, and least in the first, in which on the contrary a certain pressure is maintained ; consequently the juice in the last vessel boils at the lowest temperature.

The removal of the condensed water from the steam-drum of the first vessel was formerly effected by a syphon about 15 feet in height, as measured

from the lowest tube plate, but now that the first vessel is generally worked under pressure, the condensed water is allowed to flow out through a steam trap. The condensed water of the second and third vessels is removed by a pump if it is to be used for feeding the boilers ; otherwise, the steam-drums are simply connected with the air-pump. They are therefore always kept free from water, and as the vapours from the juice in the preceding vessels are immediately condensed by the cold juice, a vacuum is produced in the juice chambers in the first and second vessels.

In order to remove air and gases from the steam-drums, narrow tubes are fitted to each drum, such tubes communicating with the dome of the same evaporating vessel. Though no ammonia escapes from cane juices, these tubes are called " ammonia " tubes from analogy with the corresponding tubes used in beet juice evaporators.

The clarified juice is pumped into a tank, from which it gravitates into the first vessel, or is pumped into it if the first vessel is worked under pressure. It enters at the bottom of the juice chamber, where various arrangements are made to facilitate the mixing of the fresh juice with the juice already present in order to ensure a good circulation. The vessels are connected with one another by means of pipes provided with cocks, by the cautious opening of which the juice is drawn from the first vessel into the second, and from there into the third, thanks to the difference in vacuum of the three vessels ; in this way the clarified juice enters the system, and leaves it with the concentration of " syrup." A proper regulation of the cocks permits the supply of juice to equal the evaporated water so that the concentration goes on steadily without changing the position of the cocks.

Care must be taken to keep only such a quantity of juice in the first vessel for the upper tube-plate to be just covered ; if the hot plate is not constantly moistened by the juice, overheating is to be feared, and if the vessel is too full, there is a risk of losing juice by its passing off with the current of vapour leaving the vessel. These remarks are also applicable to the other vessels, though with them the danger of overheating is not so great. If they are insufficiently filled, they should be supplied from the other vessels ; in the contrary case, the supply is stopped, but the syrup is only drawn off when it has the required specific gravity.

A high temperature being always detrimental, it is advisable to work with as little juice as possible in the vessels and to keep the juice level low in each, though, of course, decomposition cannot be entirely avoided.

Decomposition of Sucrose during Evaporation.—Claassen* calculated from Herzfeld's tables the quantity of sucrose decomposed during evaporation. As these are calculated for slightly alkaline juices and cane juices gradually become acid during evaporation, the figures given are not perhaps quite

* " Archief voor de Java Suikerindustrie," 1894, 185.

applicable here ; but where they are not stated for cane juice by direct experiments, Claassen's actual figures are given.

Claassen's Experiments.—Claassen's experiments were made with a low level of juice in the evaporators and concerned :—

1. Quadruple effect with high-pressure steam.
2. Quadruple effect with low-pressure exhaust steam.
3. Triple effect with juice-heater using steam under pressure of 1 atmosphere.

I.—QUADRUPLE EFFECT WITH HIGH-PRESSURE STEAM.

No.	Period of Evaporation in minutes.	Temperature in °C.	Sucrose in the Juice.	Loss of Sucrose per hour according to Herzfeld.	Loss of Sucrose on 100 Sucrose during Evaporation of the Juice.	Loss of Sucrose on 100 Beet.
First vessel ..	7	112	10	0·1677	0·0195	—
Second ,, ..	8	105	15	0·1162	0·0155	—
Third ,, ..	12	95	25	0·0512	0·0102	—
Fourth ,, ..	27	68	50	0·0100	0·0045	—
	54				0·0497	0·0065

II.—QUADRUPLE EFFECT WITH LOW-PRESSURE EXHAUST STEAM.

No.	Period of Evaporation in minutes.	Temperature of the Juice in °C.	Sucrose in the Juice.	Loss of Sucrose per hour according to Herzfeld.	Loss on 100 Sucrose during Evaporation in the Vessels.	Loss on 100 Beet.
First vessel ..	8	102	10	0·1230	0·0164	—
Second ,, ..	11	96	15	0·0844	0·0155	—
Third ,, ..	17	86	25	0·0342	0·0097	—
Fourth ,, ..	43	65	50	0·0100	0·0071	—
	79				0·0487	0·0063

III.—TRIPLE EFFECT WITH LOW-PRESSURE STEAM AND JUICE HEATER.

No.	Period of Evaporation in minutes.	Temperature of the Juice in °C.	Sucrose in the Juice.	Loss of Sucrose per hour according to Herzfeld.	Loss on 100 Sucrose during Evaporation in the Vessels.	Loss on 100 Beet.
Juice heater ..	6½	120·8	10	0·330	0·0358	—
First vessel ..	9	106·2	15	0·120	0·0180	—
Second ,, ...	14	86·1	25	0·055	0·0128	—
Third ,, ...	27	67·7	50	0·010	0·0045	—
	56½				0·0711	0·0092

Provided the dimensions of the vessels are proportional, and the amount of juice is kept small, the loss of sugar by decomposition remains under 0·01 per cent. on the raw material, even in the most unfavourable case.

The most remarkable fact resulting from these experiments is that high pressure steam in the first vessel does not cause more loss than low pressure steam. This is due to the fact that up to a temperature of 115° C. sucrose decomposes very slowly. As the decomposition of sucrose which occurs with the rise of temperature increases, so the time during which the juice remains in the vessel is shortened. This is owing to the increased transmission of heat at the higher temperature, under which condition the heating surface and also the contents of the vessel may be decreased. Owing to this shortened stay in the vessels, the harmful influence of the higher temperature is neutralized, and therefore temperatures of some degrees above 100° may safely be employed in the first vessels or in the juice-heaters.

Kestner Apparatus.—That this is true is seen in the Kestner evaporator, where the juice to be concentrated only remains for one or two minutes in the tubes, but at a steam temperature of 130° C. in the first vessel and of 110° C. in the second, without any notable charring or decomposition of the sugar or other constituents of the juice. Owing to the high temperature and the very small amount of juice in the vessel, it climbs very rapidly in a thin film upwards into the upper part, from which it is carried into the second vessel, and so forth till the syrup is delivered by a barometric column. As the first quantity of syrup that is discharged is often not of the density required, a pipe is coupled to the juice inlet, so that this first liquor can be returned through the apparatus.

Instead of complete evaporating plants with multiple effects after Kestner, we often see one shell working as a pre-evaporator. Hot juice is introduced into the pre-evaporator, is concentrated to some 20° Brix and from there passes into the triple effect, while the juice vapour is used partly for heating the first vessel and partly for the vacuum pans or juice-heaters.

Since such a pre-evaporator only contains very little juice, there is some danger of the latter drying up and charring if by mishap the juice supply is stopped for a moment, without the steam having been shut off. In order to prevent this, juice from the first vessel may be pumped back constantly through the pre-evaporator, so that both form, so to say, one single body.

Mechanical Losses.—Apart from chemical losses due to decomposition or over-heating, mechanical loss also arises either from the vapours carrying over the juice as a spray (entrainment), or by the juice actually boiling over.

Sugar present in Condensed Water.—When the juice boils over in the first vessel, the condensed water in the steam-drums of the next vessel will contain sugar, which causes corrosion of the boiler-plates if the water be used for feeding the boilers. At the high temperature prevailing in the boilers, sucrose becomes decomposed into acids, which attack the plates and thus give rise to leakages.

Testing the Condensed Water.—Although an unpleasant smell in the steam at once reveals the presence of sugar in the feed-water, it is necessary to test the condensed water for sugar from time to time, because this condition need not arise solely from the juice boiling over, but may also be due to leakages in the tubes of the evaporators, which allow the juice to pass into the steam-drum as soon as the vacuum is temporarily destroyed by some circumstance or other.

The examination of the condensed water can be made by evaporating 100 c.c. to a volume of 10 c.c., boiling it for a few moments with a few drops of hydrochloric acid, then neutralizing with caustic soda, and heating it again after addition of a little Fehling's solution, in which case a precipitate of red cuprous oxide reveals the presence of sugars. The reaction with an alcoholic α-naphthol solution* in which the sample assumes a violet coloration in case sugar is present, is too sensitive a test, as it detects traces of sugar which are practically insignificant.

Separator.—In order to avoid losses of syrup from the last vessel, a " separator " is usually placed between it and the air-pump. This is a cylindrical

*Langguth Steuerwald gives in " Archief voor de Java Suikerindustrie " 1919, 656, the following prescription for that test: A test tube is filled with the water to be examined and emptied, so that only about 0·2 c.c. remains adhering to the glass. Add one or two drops of a 4 per cent. alpha-naphthol solution and 15 to 20 drops of concentrated sulphuric acid. A violet coloration within half-a-minute shows contamination of the water with sucrose.

vessel provided with perforated diaphragms, which reduce the speed of the vapour and compel it to part with the drops of syrup it carries with it. The separated syrup collects on the bottom and flows back to the last vessel by means of a discharge pipe.

In many cases where the sugar manufacturer had full confidence in the excellent action of the separator, which was constantly controlled by the analysis of the waste water from the condenser, enormous losses of sucrose were nevertheless recorded, the source of which could not be detected. We have found many a time several per cent. of the juice to be lost during concentration which must have been entrained by the vapours and rushed through the separator into the waste-water. Yet even careful examination with alpha-naphthol had in every case failed to detect any appreciable amount of sucrose in that water.

It is very probable that the heavy drops or streams of syrup do not mix with the water, but collect on the bottom of the pipes and leave the sugar-house underneath the flow of water, thus escaping unnoticed even if the upper layer of the current is constantly tested with great care.

Entrainment.—Langguth Steuerwald* recommends small test pipes to be fixed at the bottom of the vapour pipes, by which the lowest layer of the liquid passing through them may be sampled and tested. When sucrose is found to be present there, the separation may be improved by adopting centrifugal screens or other contrivance to try and reduce entrainment in some way or other.

Nieboer† quotes an instance where after the shifting of the "save-all," the unaccountable loss in the sugar factory decreased at once from 3·59 and 4·55 per cent. in two consecutive years before the improvement to 2·65 immediately after.

De Haan‡ mentions that in several Java factories where separators of the Hodek type had been discarded and replaced by an improved apparatus, using centrifugal screens for the separation of the spray and keeping the level of the liquid in the several elements of the multiple effect below a certain height, results were obtained showing that the loss due to entrainment had been diminished to a negligible amount.

Whereas previously, the unaccountable losses in three factories had been 2·45, 3·39 and 3·12 parts on 100 parts sucrose in cane, they fell immediately to 1·57, 1·17 and 0·78 parts respectively.

According to Claassen,§ forcing the work of the evaporator beyond its normal capacity may have helped to induce a high loss by entrainment. It is an elementary principle that the juice during boiling must not exceed a certain height, the best separator being a lofty evaporating chamber. But

* "Archief voor de Java Suikerindustrie, 1920, 262. † *Ibid.*, 1919, 651. ‡ *Ibid.*, 1920, 223.
§ "Die Zuckerfabrikation," 4 Druck, 173.

so long as a powerful air-pump sucks away vapours with hurricane-like velocity, entrainment from the last vessel may always be expected, even when using a low level and a most perfect separator. Therefore it would be a good thing if the idea of evaporation under pressure, which has been the object of serious experiments (broken off by the war), were to be investigated once more.

Evaporation under Pressure.—In this system the multiple effect is worked just as in the Kestner apparatus, with a small quantity of juice rushing at a high speed through the evaporator and therefore offering very little chance of being overheated.

In the first vessel, the juice boils with a temperature of 128° C., which gradually decreases in the subsequent ones to 102° C. in the last vessel. The final vapour, having a temperature of over 100° C., is used for heating juice-heaters, vacuum pans, etc.

Not only does this system mean a great saving of steam by the elimination of the air-pump and of the cooling water, which now leaves the condenser charged with valuable heat, but it does away also with the great velocity of the vapour from the last vessel and thereby removes one of the greatest factors of entrainment. Besides, if some juice does get entrained, the condensed water is still there and does not escape unnoticed, so that its sugar content may at any moment be accurately ascertained.

Acid Condensed Water.—From time to time it is also necessary to test the reaction of the condensed water, as it sometimes becomes acid. When sulphitation is carried too far and gives rise to bisulphites, these decompose in the evaporating vessel, forming normal sulphite and sulphurous acid, whfch latter is carried over by the vapours, and is thus found in the feed-water ior the boilers. Acid water attacks the boiler tubes and plates, causing them to leak.

Sulphur.—The writer once found sulphur in the condensed water, this being probably due to reduction of the sulphurous acid by the metal of the steam-drums.

Red Colour of Boiler Water.—It often occurs that, although the water is free from sugar and acids, it assumes a red coloration in the boilers and primes violently. This is due to the water being too soft and pure, and rusting the boiler plates, and can be readily remedied by feeding the boilers with calcareous water for a few days, so that the metal becomes covered with a thin scale, which prevents it from being subsequently attacked by the soft water.

II.—Incrustations in Evaporators

Nature of the Incrustations.—As the result of the concentration of the cane juice, various substances which have been dissolved in the thin-juice are separated in an insoluble state ; they partly remain in the juice, rendering it turbid, and are partly deposited as a hard crust on the tubes, chiefly on those of the last vessel, and interfere with the conduction of heat, and hence with the evaporation. It is evident that such an insulating coating on the tubes interferes with the evaporation, and consequently decreases the capacity of the plant. It is therefore desirable, first to avoid the formation of scale as far as possible ; and, second, to remove the scale before it becomes so thick as to retard the evaporation.

Formation of Scale unavoidable.—As even the clearest and best clarified juice is certain to deposit constituents which are insoluble in the concentrated syrup, it is impossible to prevent the formation of scale, so that the only course is to remove it as frequently as possible.

The composition of the scale depends, first, on the nature of the clarification, and, second, on the degree of concentration, in other words, on the particular vessel of the multiple effect in which it has been deposited.

Varying Composition of Scale.—For example, the scale in the first vessel chiefly contains constituents which were present in the juice in the state of colloids or in suspension, while that in the other vessels consists principally of substances which were dissolved in the dilute juice, but became insoluble during concentration, either because of the diminished quantity of solvent or by their being less soluble in concentrated sugar solutions. The first vessel, therefore, does not get much scaled when the methods of clarification permit filtration of the whole of the juice and therefore remove the colloid impurities. On the other hand, the first vessel becomes heavily incrusted when the juice is not filtered, but merely decanted or syphoned off. Moreover, when dead or over-ripe canes are being crushed, yielding juice containing much suspended cane fibre, the first vessel may thereby become choked, the fibre being converted into hard masses by the inorganic suspended matter deposited upon it.

Deposit of Cane Fibre.—An analysis of such a deposit, although not strictly an incrustation, gave the following figures calculated on dry substance :—

	Per cent.
Cane fibre	22·0
Silica	44·4
Calcium carbonate and phosphate	33·6
	100·00

In factories working with the defecation process, where, therefore, the greater part of the juice is evaporated without being filtered, the first vessel of the multiple effect scales much more than in factories where the juice is

clarified by carbonatation and consequently is deprived of all colloid matters by filtration.

In the incrustations from the former juice we find calcium phosphate, the quantity of which decreases as evaporation proceeds; also silica, which on the contrary, increases as evaporation proceeds. In the last vessel the scale contains calcium oxalate.

In the case of carbonated juices, the first vessel contains a little calcium carbonate, probably formed by decomposition of bicarbonate and also some silica. In the other vessels the silica content increases, and in the last calcium oxalate prevails.

Some analyses of incrustations met with in practice follow here, the results being calculated on the dry substance (free from traces of metal from the tubes).

Constituents.	I.				II.				III.			IV.			
	Defecation.				Defecation.				Defecation and Sulphitation.			Defecation and Sulphitation.			
	I.	II.	III.	IV.	I.	II.	III.	IV.	I.	II.	III.	I.	II.	III.	IV.
Loss on ignition ..	22·28	25·97	31·62	39·91	29·10	23·97	33·15	35·50	24·35	30·63	33·58	19·80	20·40	21·36	20·11
Silica	5·64	14·26	41·75	18·45	2·08	1·75	12·35	24·80	2·14	2·83	40·32	4·30	13·26	31·66	57·27
Phosphoric acid ..	29·25	22·12	9·83	2·70	21·87	25·40	6·12	0·97	23·12	21·65	7·51	22·30	22·00	10·34	0·36
Sulphuric acid ..	1·90	2·31	0·45	0·26	4·84	5·87	1·10	0·94	7·92	5·48	2·36	2·01	1·29	0·84	0·41
Sulphurous acid ..	—	—	—	—	—	—	—	—	0·50	0·36	—	—	—	—	—
Carbonic acid ..	0·57	0·55	0·38	11·50	2·12	5·17	1·84	1·35	0·25	0·47	—	4·11	3·37	3·62	3·56
Oxalic acid ..	—	—	1·37	5·00	—	1·10	26·94	24·61	—	—	2·77	—	—	—	—
Iron oxide ..	1·47	2·20	0·76	1·69	3·50	3·10	2·84	1·50	8·11	7·44	0·51	18·79	6·16	11·55	6·50
Alumina	0·30	0·39	0·13	0·81	0·31	—	0·12	0·70	—	—	—	—	—	—	—
Lime	39·13	31·96	13·97	23·42	39·17	31·83	30·00	18·79	31·90	29·35	13·09	29·33	32·63	21·70	15·12

The loss on ignition is the measure of the organic substance, but includes the water of crystallization of the gypsum. The sum of the constituents sometimes exceeds 100 per cent., because the weight of carbonic acid liberated from the calcium oxalate on combustion is recorded under the figure for loss on ignition. In addition to oxalic acid, the organic constituents of the scales include gummy and albuminoid substances which are deposited together with other gelatinous bodies, bagasse fibre already mentioned, and a considerable proportion of caramel, the latter being due to overheating of the juice, which penetrates the incrustations and becomes partly charred.

Origin of different Constituents.—Iron oxide is formed by the solvent action of the juice on the iron plates of the steam chamber; the other constituents, were derived from the juice, some of which were dissolved therein, whilst others occurred in the state of colloids. Among these latter, calcium phosphate occupies the most prominent place, and this explains why the scale in the first vessel chiefly consists of calcium phosphate (with defecated juice), whilst this constituent is almost always absent from the scale formed from carbonatation juice which is filtered previous to evaporation.

It is true that calcium carbonate, sulphite, and oxalate (found in the scale) are insoluble in water, but they dissolve to some extent in sugar solutions, the solubility depending on the concentration, temperature, and reaction of the sugar solutions.

Calcium Carbonate.—Calcium carbonate is only slightly soluble in sugar solutions, and when this substance occurs in appreciable proportion in the scale, it must either have been formed from quicklime by absorption of carbonic acid in the defecation process, or have been dissolved in the form of bicarbonate during the second saturation of the carbonatation process, and subsequently decomposed into normal carbonate and carbonic acid during evaporation, and, in consequence, only found in the scale from the first vessels.

Calcium Sulphite.—Calcium sulphite is much less soluble in sugar solutions at high than at low temperatures, and is also much less soluble in neutral than in alkaline and acid solution. The reason why this substance is sometimes found in scale, and in large quantity, may therefore be due to an insufficiently high temperature during sulphitation, or to an improper reaction or to the concentration of hydrogen ions in the tempered juice.

We discussed on page 158 the formation of incrustation in juice-heaters occasioned by these two causes, and referred to both of them as factors contributing to the scaling of evaporators as well. These remarks bear on instances where no great mistakes have been made during sulphitation, but when excessive gassing has occurred incrustation by calcium sulphite may become very troublesome.

If, owing to carelessness, sulphitation had been pushed so far that part of the already precipitated calcium sulphite has become redissolved as bisulphite, the juice may enter the evaporators, charged with calcium bisulphite. During evaporation this substance is decomposed with the formation of sulphurous acid, which escapes as vapour, and the formation of the normal sulphite, which, at the high temperature, crystallizes out, covering the inside of the tubes with a hard, tenaciously adhering scale.

In such a case, the largest portion of the sulphite is found in the scale of the first vessel, where the bisulphite is decomposed, the normal sulphite being so insoluble that insufficient is left in solution to reappear in the incrustations formed in the other vessels where by evaporation the concentration is increased. It is evident, however, that when care is taken during sulphitation to keep the juice faintly alkaline, that is to say of a pH of slightly over 7, no bisulphite can possibly be formed, and no sulphite can crystallize out owing to the decomposition of that body. If also the temperature during sulphitation is high enough, and is not allowed to fall very much during subsiding, the amount of the normal sulphite dissolved in the clarified juice may be kept so low that serious trouble caused by the incrustation of the tubes by calcium sulphite need not be feared.

The author had the opportunity of analysing scales from first vessels of an evaporating plant, which chiefly, or rather solely, consisted of beautifully crystallized strata of calcium sulphite, which had formed from dissolved bisulphite during heating. As an equivalent quantity of sulphurous acid passed over into the condensed water, the latter became acid, and had a detrimental effect on the boiler plates.

According to Geese* the solubility of calcium sulphite in a 10 per cent. sucrose solution, at the different temperatures, is as follows :—

20°	30°	40°	50°	60°	70°	80°	90°	100°	
0·183	0·144	0·14	0·075	0·035	0·033	0·012	0·010	0·006	per cent.

The solubility is still less in alkaline juices, especially at high temperatures, so that calcium sulphite can only occur in scale if the temperature during sulphitation has been too low, or if the juice has been allowed to become acid.

Calcium Oxalate.—Calcium oxalate is soluble in sucrose solutions, the solubility increasing in inverse ratio to the temperature and (within certain limits) to the concentration of the solution. A 25 per cent. sucrose solution rendered strongly alkaline dissolves 0·05 per cent. of calcium oxalate, but this figure decreases greatly at higher concentrations and smaller alkalinities. There are, therefore, three factors which render calcium oxalate insoluble during evaporation, viz., the decrease in the amount of solvent, the increased concentration of the sugar solution, and the lowering of the temperature. For a given alkalinity, the solubility of calcium oxalate falls very considerably between the concentration of 30-50 per cent., so that we may expect the heaviest deposit of this substance in the last vessels, which actually occurs. Indeed, the scale of the fourth vessel of a quadruple effect sometimes consists exclusively of calcium oxalate.

Gypsum.—According to Stolle† the solubility of calcium sulphate in sucrose solutions falls in proportion as the concentration and temperature rise. At low temperatures a sucrose solution dissolves more gypsum than corresponds with its water content, and this is also the case at higher temperatures in low concentrations. But it dissolves less than corresponds with the water content at higher concentrations.

Calcium Silicate and Aluminate.—Calcium silicate and aluminate also dissolve in sugar solutions, especially when the solution is strongly alkaline. After long continued heating, lime is withdrawn from these compounds, so that silica and alumina become insoluble and are deposited as scale. Besides the silica occurring in this form, there is no doubt that it is also present as gelatinous silica, suspended in the juice and deposited during evaporation. Otherwise, the fact that acid juices (free from calcium silicate) deposit silica during evaporation could not be explained.

* "Zeitschr. Rübenzuckerind.," 48, 103.
† *Ibid.*, 50, 321.

Alumina.—Alumina is sometimes found in scale, although this constituent occurs in but small quantity in cane juice. Being specially noticeable in scale from juice treated by carbonatation, we are justified in concluding that it is derived from the lime used for the clarification, which sometimes contains as much as 1 per cent. alumina.

Nitrogen.—The nitrogen content of the dry scale is due to coagulated albuminoids, and never amounts to more than 0·20 per cent., and in most cases is less.

Cane Wax.—The scale in the first vessel sometimes contains a relatively large proportion of cane wax which was suspended in the juice. Some deposits contain so much wax that the sight-glasses of the evaporators become covered with a greasy, dark-coloured film. Owing to its low specific gravity, such a film floats on the surface of the juice, and therefore wax is not a notable constituent of hard scale.

The following is a typical analysis :—

Cane wax	40·2
Calcium phosphate	48·7
Silica	5·7
Undetermined organic matter	5·4
	100·00

It goes without saying that thorough subsidation, or, if possible, filtration of the juice, will help to prevent incrustation by suspended matter, and further that a neutral juice will dissolve less lime-salt than an acid or an alkaline one, and, therefore, will be less liable to deposit scale.

As juices are very seldom exactly neutral, they all deposit incrustations, and all that can be done is to restrict their formation as much as possible, and to remove them as soon as they have formed.

Composition of Scale before and after Treatment.—The analytical figures given below relate to the washed and dried scales after treatment with caustic soda, and are from the same factories as the samples I to IV of Series I, mentioned on page 193, which were analysed before boiling with caustic soda. From the insignificant difference in composition before and after boiling with caustic soda, we see that this reagent has a loosening effect rather than a solvent action.

Removal of the Scale.—Some twenty-five years ago, iron spirals were suspended in the tubes of the evaporating vessels, with the object of inducing the scale to deposit on these, rather than on the tubes. About the same time wooden staves ($\frac{1}{8}$ inch less in diameter than the tubes) were similarly used, with the additional advantage of diminishing the volume of juice, and thus increasing

the heating surface. The rough surface of the staves was also more favourable for the deposit of scale than the smooth surfaces of the brass tubes. Though this novelty was at first regarded very favourably, the staves have since disappeared.

Such appliances do not obviate the cleaning of the vessels from time to time. In countries where the mill stops on Sundays, that day can be utilized for this purpose, while in places where the work continues all seven days of the week without rest, it is well to clean and scrape the evaporators, say, once a fortnight. The practice, sometimes followed, of continuing evaporation without stoppage until the evaporators are excessively scaled, is to be condemned, because the capacity of the plant will be considerably reduced during the last days, and also because the layer of scale has then become so thick that it demands a great deal of labour and time to remove it.

Boiling with Caustic Soda.—The best mode of cleaning is to boil caustic soda solution in the vessels, with open valves and with live steam in the steam-drum, so that the liquid boils at ordinary atmospheric pressure, otherwise the temperature is not sufficiently high. After some six or more hours the caustic soda solution is let out, the vessels are washed a couple of times with water, and the scale scraped off with scraping irons. This last operation, being somewhat rough, is not without danger to the brass tubes, as appears from the rather large quantity of brass splinters which were found in every sample of scale examined by the author. Formerly, the vessels in which the soda solution had been boiled were boiled again with dilute hydrochloric acid, after the soda had been let off, and the vessels washed. This was to dissolve the already loosened scale, but the practice is now discontinued. It is true that most of the incrustations dissolve in concentrated hydrochloric acid, but this should not be applied,

	Scale after Treatment with Caustic Soda.			
	I.	II.	III.	IV.
Loss on ignition	26·20	27·60	40·01	35·30
Silica	7·28	8·43	22·49	39·01
Phosphoric acid	20·91	19·85	1·61	0·37
Sulphuric acid	4·87	3·38	0·43	—
Sulphurous acid	—	—	—	—
Carbonic acid	3·97	4·08	3·06	3·81
Oxalic acid	—	—	20·49	12·60
Iron oxide	2·24	3·47	0·69	0·55
Alumina	—	—	—	—
Lime	34·29	31·28	20·15	13·96

since it attacks the iron vessels and results in leakages. Diluted hydrochloric acid dissolves the calcium carbonate found in scale in the first vessels treating carbonatation juice, but it fails to attack the crystallized oxalate and the silica, so that the action of this reagent is very restricted. It also has the disadvantage of attacking the unscaled portions of the iron plates,with the formation of hydrogen, which forms an explosive mixture with air. No workmen should, therefore, enter the vessels with a light after boiling with hydrochloric acid before the latter have been thoroughly washed out with water, and the air drawn off by the air-pump. Hydrochloric acid does not dissolve much of the scale, neither does caustic soda, but this last reagent loosens it, so that it may be more easily scraped off. Some silica becomes dissolved and calcium oxalate is decomposed, but otherwise the composition of a scale is not much changed after being boiled with caustic soda.

Van der Linden* mentions an observation made in a factory, where great trouble had been experienced in the removal of the scale of the third and fourth vessels. On one occasion it was found that after boiling out, the scale from the fourth vessel was present on the tubes as a gelatinous mass, which offered no trouble in its removal. Investigation showed that among the drums of caustic soda which had been used, one containing ammonium fluoride had inadvertently been included. Later on, 15 kilograms of ammonium fluoride were used along with the caustic soda in the cleaning of the first and second vessels and 35 kilograms each in the third and fourth ones ; and in every instance the incrustation was so thoroughly loosened that scraping was very easily accomplished. In a third case, a fourth vessel having 246 square metres of heating surface was boiled out successfully with a solution containing 35 kilograms of caustic soda and 25 kilograms of ammonium fluoride.

Schweizer† says that he cannot imagine what reaction is exerted by the ammonium fluoride on the scale, either on the silica or on the silicates, and he supposes that possibly one of the other halogen salts, sodium chloride or ammonium chloride, might be used.

Schmidt‡ boiled out small quantities of scale in the laboratory with caustic soda and ammonium fluoride in weak concentration, and found that 51 per cent. had gone into solution, of which figure 73 per cent. was silica. Then he repeated the same experiment with caustic soda and sodium chloride, when 50 per cent. went into solution, 71 per cent. of which appeared to be silica. There being little difference between the two results, common salt might be used to replace ammonium fluoride.

Removal of Scale by Fermentation.—A sure but slow means of cleansing the vessels after the close of the grinding season is to fill them with diluted molasses (6-8° Brix) and allow this to ferment. The evolution of gas within the scale itself loosens the particles of which it consists, and after some three weeks the

* "Int. Sugar Journal," 1923, 638. † *Ibid.*, 1923, 639
‡ *Ibid.*, 1923, 640.

fermented wash is run off, and the scale may then be brushed away without scraping. As this method requires time, it cannot, of course, be employed during the grinding season, so that boiling with caustic soda and scraping is then the only remedy.

Deposits in the Steam Chambers.—The deposits so far mentioned are derived from the juice, but another troublesome deposit occurs in the steam chamber of the first vessel, which likewise retards the transmission of heat and evaporation. The steam used for heating the first vessel may contain small quantities of lubricating oils, which collect on coming in contact with the cold tubes and which, in combination with iron or copper oxides, graphite, and similar substances, form a greasy, non-conducting layer which causes much trouble. The author has examined several such deposits, and his analyses, calculated on dry substance, are recorded here. The first sample dates back to 1892, and must have been gradually forming during the ten preceding years. The organic matter chiefly consisted of fatty acids, while in the second and third samples it consisted of mineral lubricating oils.

Constituents.	I.	II.	III.
Oil	28·4 (fatty acids)	28·50	13·9
Iron oxide	39·4	58·80	82·6
Copper oxide	30·0	2·50	—
Lead oxide	1·0	6·20	2·3
Graphite, etc.	1·2	4·0	1·2

Removal of the Greasy Deposit.—Such a layer is sometimes, but not always, found in the steam chambers; and as its presence cannot be detected from outside, it is advisable to inspect the steam chamber every year, and to remove the deposit if present. Mineral oils do not dissolve in soda or in acids, and therefore boiling either with acids or alkalis is of no use. It can be removed by dissolving in petrol (gasoline) or benzine. The fatty deposit is thereby softened and flows off as a greasy mud; after which the spirit is run off and the steam chamber washed free from solvent with water. This method is rather dangerous because of the inflammable vapours escaping from the spirit. It should therefore never be attempted during the grinding season, but only when factory operations have come to a complete standstill.

If this method be considered too dangerous, the only other remedy is to remove all the tubes and scrape them carefully.

III.—Clarification of the Syrup

Though the greater proportion of the substances which become insoluble during evaporation deposit on the walls and tubes of the vessels, yet a part remains in the syrup rendering it turbid.

Composition of Deposit from a Syrup Tank.—The composition of these floating and suspended substances is similar to that of scale, as can be gleaned from an analysis of a deposit from syrup tanks in a factory working with the defecation process.

Loss on ignition	34·33
Silica	26·95
Phosphoric acid	12·25
Sulphuric acid	2·03
Carbonic acid	0·23
Iron oxide and alumina	3·58
Lime	19·45
Undetermined	1·18
	100·00

This sample closely resembles the average of the scales from the different evaporating vessels, and consists of the same elements. It is very important to remove these impurities, because they become enveloped in the sugar crystals during the growth of the latter and impart to them a dark tint which cannot be removed by washing.

Removal of Impurities.—It is not feasible to remove these impurities by filtration through cloth if the syrup has been obtained from defecated juice, since the gummy constituents clog the filtering media. Consequently one is compelled to allow the syrup to subside.

Filtration.—Sand filters, which were greeted with enthusiasm fifteen or twenty years ago, do not seem to have fulfilled early promises, since they have not come into general use for syrup. It seems that the very large quantity of water required to wash out the syrup from the sand when cleaning is the chief obstacle to their employment for this purpose.

Subsidation.—If we abstain from filtering the syrup it can only be clarified by subsidation. The best method is to boil the syrup in eliminating pans after it leaves the evaporating plant, neutralizing it with lime or soda if necessary, or, if too alkaline, with phosphoric acid. Sulphitation may be combined with

this elimination, but a stirring apparatus is then necessary in order to bring the gases into contact with the syrup. It is advisable to keep the temperature of the syrup below 70° C. in order to prevent inversion during sulphitation.

If the precipitate subsides slowly, a little phosphoric acid and lime, soda, or sodium phosphate, may be added during elimination, but great care must be taken not to use too much of these, or the precipitate might become too thick. If possible, the syrup is then filtered. If not, it is run into settling tanks, similar to those described in the chapter on Clarification. A clear supernatant syrup then separates from a layer of mud, the thickness of which depends on the quantity and the flocculence of the precipitate.

It is advisable to decant the subsided syrup as soon as possible, and not to leave it in the tank too long. According to Herzfeld's tables, mentioned on page 20, at a temperature of 90-95° C. such a syrup loses 0·0392 per cent. sucrose per hour, so that during the six hours which syrup generally requires to subside, no less than 0·25 per cent. of the sucrose may become decomposed.

Treatment of the Syrup Scums.—As soon as the syrup has settled, it is drawn off from the precipitate and conveyed to the suction tank of the vacuum pan, while the precipitate itself is dealt with in various ways. Some manufacturers mix it with the molasses coming from the centrifugals, and boil them together to second sugars. There could not be any objection to this plan if the second sugars always found a market, for the fine particles of the precipitate would be incorporated in the crystals of the second sugars. In many cases the second sugars are re-melted in the juice, and the impurities, previously eliminated with care, are thereby returned into process. Moreover, the practice of returning molasses to the pan has suppressed the second sugars, so that the mixing of the syrup scums with first molasses has become impossible. In other factories, the syrup scums are run into the scum tank of the mill juice, which proceeding has the drawback of increasing the work of the scum filters, and thus diminishing the capacity at that stage. Further, this scum is very slimy, and the particles are excessively fine, so that they choke up the pores of the filter-cloths. Finally, these scums contain a highly concentrated sugar solution, and as even the driest press-cakes contain about 50 per cent. of juice, it is not an economical proceeding, since we are here dealing with concentrated juice or syrup, in which a considerable quantity of sugar becomes lost. The best way is to pump the syrup scums into the juice in the defecation pans; the syrup mixes with the great volume of mill juice without any visible increase of concentration, and again undergoes all the operations of clarification and settling, while the fine precipitate is carried down by the flocculent scum of the mill juice, subsides with them, and does not give rise to any increase in the quantity of juice to be filtered by the presses. Moreover, the higher sugar content of this juice is so trifling that the loss of sugar in the filter-press cakes is not perceptibly increased.

Sourness of Syrup by Fermentation.—Sometimes juice which is neutral after defecation yields a very acid syrup, but in this case no attempt should be made to remedy the defect by using more lime in tempering, for, strange as it may appear, this is more apt to increase the acidity of the syrup than to diminish it. The only thing is to neutralize the quantity of syrup in hand, and immediately seek the cause of this acidity.. In every case where a neutral clarified juice becomes very acid during manufacture this is generally due to some ferment or other which attacks sucrose and forms acid from it. As a rule, lime-salts favour these fermentations, hence an excessive use of lime promotes the deterioration. This can be remedied by keeping the amount of lime as low as possible, and by great care in cleaning all pipes, tanks, and pumps, and even disinfecting them with a one per cent. solution of ammonium fluoride. It is, of course, better to prevent such infection by washing out the gutters and settling tanks daily.

IV.—Boiling

Principle.—During the boiling process, the concentration of the clarified syrup is continued up to the point where there is not sufficient water for the sugar to remain dissolved, and it becomes partially crystallized.

Rapidity of Crystallization.—The rapidity with which sugar crystallizes out from a solution depends, other circumstances remaining the same, on the purity of the solution, i.e., on whether much or little foreign substance is present along with the sugar. When the sugar solution is fairly pure, crystallization takes place as soon as the concentration has become too great for all the sugar to remain in solution. When an impure juice such as molasses is evaporated, crystallization takes place only very slowly, and no crystals are visible, even when the concentration is much greater than corresponds with the solubility of sucrose.

The hot concentrated liquid is allowed to cool, and after some time sucrose crystallizes out, partly because it is less soluble in cold water than in hot, but chiefly because the supersaturated state slowly gives place to the normal state of solubility.

Properties of Sucrose in Solution.—In solution in water and during crystallization from such aqueous solutions sucrose behaves quite differently from almost any other substance.

According to Wulff, the differences between sucrose and some salt or other in solution in water may be characterized as follows: Water dissolves a salt, at first very rapidly, later on more slowly, but in every case within a short

time in accordance with its solubility at the given temperature. On dissolving crystallized sucrose, the first stage is just as in case of the salt, but as soon as the water has taken up the major part of the quantity which it can finally hold, further solution proceeds much more slowly, and notwithstanding the continuous stirring of crystals and solvent, the ultimate point of saturation is very hard to obtain.

When, on the other hand, amorphous sucrose, as prepared by the melting and rapid cooling of the crystallized body, is dissolved in water, solution goes on more slowly during the first stage, and only after the material is completely impregnated with liquid does it dissolve with greater rapidity. Actually amorphous sucrose dissolves copiously, the concentration of the solution increasing regularly to the final point of solubility, in fact even surpassing that limit, and the more so the greater the excess of sucrose present. Later, the solution becomes supersaturated, and yet solution proceeds till the moment when crystals make their appearance, after which concentration sinks till the maximum point of stability is reached.

Wulff considers these phenomena as a manifestation of an *intermixture* of two miscible bodies, viz., water and amorphous sucrose, owing to which, contrary to true *solution*, mixtures in every proportion are possible.

Wulff believes sucrose to be present in solution only in the amorphous state, so that it has to pass over from the crystallized state into the amorphous on solution, and back from the amorphous state into the crystallized on crystallization. Since this transformation takes time to complete, this hypothesis explains the delay in solution and crystallization of sucrose in aqueous solution.

Amorphous sucrose is more stable at low than at high temperatures, and heating rather rapidly transfers it into the crystallized state.

Even the most concentrated sucrose solutions may be cooled very far before they crystallize out; and the more the solution is cooled, the longer crystallization is delayed and the greater the interval before its end point is attained.

Van Ginneken* heated some sucrose solutions to 130° C., and others of identical composition and concentration to 110° C., when he found after cooling them all to the same temperature that the time elapsing before the formation of crystals was greater in the case of the liquids that had been raised to the higher temperature, although no decomposition apparently had occurred. He also observed that if sucrose were repeatedly crystallized from aqueous solutions and redissolved, the time taken for the appearance of the first crystal became longer after each operation, the conditions of supersaturation, temperature, intensity of movement and of composition, always being the same, the time required for the re-solution of crystals likewise successively increasing. Other experiments demonstrated that apparently the history of the sucrose is of some importance in regard to its crystallizing properties. Thus he showed that a pure sucrose, obtained by precipitating a concentrated aqueous solution of refined

* "Chemisch Weekblad," 1919, 1210. "Jour. Soc. Chem. Ind.," 1919, 784a.

sugar with alcohol and washing with alcohol and ether, crystallizes more slowly than the refined sugar from which it was prepared. After being kept in a bottle for a few months that same preparation, however, once more became normal in respect of its solubility. Van Ginneken's experiments demonstrated that the optimum concentration for grain formation is 83·5 to 84·5 per cent. at 80° C.; and he further observed that the time necessary for the formation of the first crystal is much greater than that required for the appearance of subsequent ones, temperature and concentration being kept the same. He found also that with a concentration under 80 per cent. the tendency to form new crystals (rather than to grow on those already present) is so small as to be negligible. At 81 per cent. it is greater, but it is not practicable to keep the concentration as low as this when boiling and cooling, so that in sugar-houses the possibility of the formation of false grain under such conditions is difficult to preclude.

In order to explain why the first crystal forms so much more slowly than those appearing later, Van Ginneken suggests two possibilities, namely, (1) the auto-catalystic effect of the already present crystals on the molecules of sucrose in their vicinity ; and (2) the formation of large complexes of sucrose molecules which are driven apart by heating, but on cooling gradually condense until they combine to form crystals, at which point others have approached the same condition and consolidate very soon after.

These phenomena have already been noticed before by other investigators. When carrying out his experiments on the formation of molasses, the author had observed that sucrose crystallized more rapidly when the solution had previously been heated to a relatively low temperature during a short time, than when the liquid had been kept at a higher temperature for a longer period. Van der Linden* showed that a molasses which on cooling readily gave a crop of crystals failed to do so when the grain was re-dissolved and the liquid cooled in exactly the same way as before.

The rapidity of crystallization of sucrose is also greatly influenced by the simultaneous presence of impurities. In the case of the crystallization of a salt solution, it proceeds just as rapidly in the presence of other bodies, even if they are of a viscous nature, as from a pure solution. In the case of sucrose, however, where besides the simple crystallization a transformation from the amorphous into the crystallized state has to be reckoned with, the non-sugars from the juice exert a notable delay in crystallization, as the result of which crystallization of sucrose from impure concentrated syrup and molasses is slower, more difficult and less complete than from pure liquids.

Different Ways of Boiling.—Boiling may therefore be performed in two ways, one of which is followed when boiling syrup, and the second when boiling molasses, but, as will be shown later on, this distinction is not strictly observed.

* "Archief voor de Java Suikerindustrie," 1919, 1517.

Pure juices are concentrated at not too high a temperature until the sucrose commences to crystallize in the pan, after which the crystals grow slowly as evaporation continues and fresh supplies of syrup are added, until ultimately the whole is transformed into a stiff *magma* (massecuite), consisting of a large quantity of crystals and only a little liquid (molasses). This method is called " boiling to grain," and can only be applied to pure syrups. If the solution is too impure for this purpose, it is concentrated at moderate temperatures to such a degree that the concentrated syrup or molasses only deposits sugar on cooling, this mode of working being termed " boiling smooth." In this case, the consistency of the liquid is such that a sample can be drawn out in the form of a thread, when the liquid is said to be boiled to " string proof."

Boiling in Vacuo.—Boiling is always performed *in vacuo* for the reason given under the heading " Evaporation," and for the further reasons that under atmospheric pressure the temperature at which the heavy material parts with its water is higher ; and circulation being less perfect, the risk of local over-heating and consequent decomposition and coloration of the massecuite increases.

Vacuum Pans.—The old vacuum pans were made of copper, but owing to the demand for pans of large capacity they are now almost exclusively made of iron, so that copper pans provided with steam-jackets are the exception. The iron vacuum pans are in many cases lined with copper plates in order to prevent coloration of the sugar by the solution of iron in the acid syrups and molasses.

Heating.—In most cases pans are heated by means of copper coils, to which either direct or exhaust steam is admitted.

Steam-jackets.—As already mentioned, the earliest pans had steam-jackets in addition to coils, but these were subsequently dispensed with in order to secure a wide discharge opening, and thus to economize time when discharging the pan. The advantages of a steam-jacket are that the heating surface of the pan is increased by several square feet, and the massecuite in the pan is less exposed to the cooling influence of the external atmosphere. Consequently, steam-jackets have been adopted in many modern pans, even the discharge door being heated in this manner.

Calandrias.—Instead of coils and steam-jackets, many pans have calandrias, similar to those used in multiple effect evaporators, but with tubes of much larger diameter to permit the semi-solid massecuite to circulate. In many cases the circulation is further promoted by a screw revolving in a large central tube whereby the boiling mass is raised in the centre, and thus induced to flow downwards at the sides.

Circulation.—It is of great importance that the contents of the pan be thoroughly mixed; first, in order to prevent local overheating of the massecuite where it is in contact with the hot coils; and, secondly, in order to secure crystals of uniform size. Circulation is especially necessary when molasses (previously separated from crystals) is used for diluting the massecuite in the pan, otherwise the concentrated massecuite settles to the bottom of the pan, and does not mix with the added molasses. Under such conditions, samples taken with the proof-stick give false indications concerning the real state of the contents of the pan. Even when boiling syrup massecuite, the bottom discharge is often choked by a hard mass of sugar which has been accumulating for a considerable time, and has interfered with the circulation. In order to prevent this, and, in general, to promote circulation, many pans are provided with a perforated copper coil, through which dry low-pressure steam is blown through the boiling mass, so as to mix it, the vacuum not being perceptibly lowered. The perforations should be made at the bottom of the coil, in order to prevent them being choked with sugar crystals when the pan is discharged. A second device for promoting circulation is to introduce the syrup and molasses through a bent pipe extending nearly to the bottom of the pan, so that the thin liquids are compelled to force their way upwards through the massecuite and so become thoroughly mixed with it, which is not the case if the syrup is introduced at the top of the massecuite. The same end is sometimes gained by pumping the syrup and molasses through a perforated copper coil at the bottom of the pan. To prevent choking, this coil is likewise perforated on its under side.

The Proof-stick.—In order to examine the contents of the pan without disturbing the vacuum, samples are withdrawn from time to time by means of the apparatus now to be described. A copper tube is fixed in an inclined position within the pan in such a manner that the upper extremity projects through the side of the pan, while the lower extremity terminates at its centre, and, consequently, is immersed in the boiling massecuite. The upper extremity of this tube is open, but the lower extremity is closed by a valve of special construction. When a sample is to be withdrawn from the pan this valve is opened by means of a long brass rod, which is thrust down the tube and then turned as one turns a key in a lock. This rod, called the "proof-stick," is provided at its lower end with a plug, by means of which the above-mentioned valve is opened, and also with a small cavity, which thereby becomes filled with the boiling material. During this operation, the upper opening in the tube is kept air-tight by the closely fitting rod or "proof-stick." The latter is then twisted back to its former position, thereby locking the valve, and then withdrawn from the tube, carrying with it the small sample of massecuite which fills the cavity.

The proof-stick should be of sufficient length that samples may be withdrawn

from the bottom part of the pan, and the cavity sufficiently large to withdraw a fairly big sample at each test.

Fittings of the Pans.—Chemicals are drawn into the pan through an iron pipe fitted at the side, whilst a cock near the top of the pan (called the butter-cock) may be used for introducing colouring agents, such as ultramarine, etc.

The pan is connected with an air-pump and injector for removing and condensing the vapours, and the resulting vacuum permits of syrup, molasses, chemicals, etc., being sucked into the pan without being pumped, as is the case in open evaporators. Between the pan and the air-pumps is fixed a kind of separator called the " save-all," which keeps back the spray rising from the boiling liquid and returns it to the pan.

Boiling to Grain.—When boiling to grain, syrup is drawn into the pan as soon as the vacuum permits, and when the bottom coil is covered, exhaust steam is turned on. As the level of the liquid rises by the constant inflow of syrup, steam is similarly admitted to the second and, if necessary, also to the third coil, the proper charge of syrup being found by experience ; the supply of syrup is then stopped and concentration commences. The formation of grain commences at about 80° Brix, and is accelerated in one of two ways. The syrup is either concentrated until it begins to crystallize, or additional syrup is suddenly added to the already supersaturated syrup, causing such an agitation throughout the mass that the sugar (present in a supersaturated state) is deposited as fine glittering crystals. This addition of dilute syrup suddenly reduces the boiling point and causes a brisk ebullition and thereby agitation of the whole liquid, which promotes crystallization. The temperature in the pan is usually 50° C. (122° F.), and the vacuum 28 inches (71 cm.).

When the pan is heated by a calandria instead of by coils, it is necessary first to draw in enough syrup to immerse the whole heating surface, after which the concentration of the syrup up to the graining point is carried out as before. But it may be necessary to start with less syrup than will completely cover the steam chamber, so that coils are to be preferred unless it is practicable to transfer part of the contents of one pan to another, after the grain has been formed, and to continue boiling what is left.

Determination of the Graining Point.—Although every pan is provided with thermometer and vacuum gauges, and the boiling can be perfectly regulated by means of these instruments, most native pan-boilers prefer to judge the concentration of the syrup from the appearance of the drops of liquid spattering against the sight-glasses.

Although this test seems very crude and uncertain, experienced pan-boilers know pretty well what to expect of the juice with which they have to deal, and when the syrup does not grain at the point they are accustomed to, they

will go on concentrating before attempting to " grain " by the sudden addition of fresh syrup.

As cane juice grains very easily, the artifices sometimes employed in the beet sugar industry to promote crystallization are superfluous. On the contrary, it is sometimes difficult to prevent premature formation of grain.

Composition of Syrups at Graining Point.—The analyses of a great number of cane syrups at their graining point are set forth here.

No.	Brix.	Polarization.	Reducing Sugars.	Water.	Quotient of Purity.	Glucose Ratio.	Sucrose per 100 Water	Sucrose crystallized out on 100 Massecuite at 60° C.	Coefficient of Supersaturation at 60° C.
1	79·21	71·70	3·34	21·59	90·52	4·66	332·1	9·67	1·15
2	80·32	72·23	3·63	20·88	89·93	5·07	345·9	12·23	1·21
3	79·73	69·00	5·16	21·27	89·65	6·25	325·8	7·88	1·13
4	77·65	70·81	3·97	23·65	89·17	5·57	299·4	2·86	1·04
5	78·43	71·11	3·91	22·67	88·68	5·60	326·9	5·98	1·14
6	80·62	71·55	3·99	20·08	88·62	5·58	356·3	13·86	1·24
7	81·72	71·87	5·04	19·58	87·95	7·01	367·1	13·70	1·28
8	78·59	68·95	4·52	22·61	87·45	7·14	304·9	3·99	1·06
9	83·11	72·48	3·40	17·99	87·21	4·7	402·9	20·80	1·40
10	79·26	69·00	5·16	21·74	87·06	7·48	317·4	5·54	1·10
11	78·47	67·49	4·82	22·73	87·04	7·14	297·0	2·19	1·03
12	83·50	72·52	4·18	17·40	86·85	5·76	417·8	23·53	1·46
13	78·51	68·15	5·59	22·39	86·81	8·2	304·4	3·82	1·06
14	79·55	67·54	5·64	21·45	84·89	8·35	314·9	5·91	1·10
15	79·75	66·49	6·71	21·49	83·37	10·09	309·4	4·75	1·08
16	75·95	63·14	7·60	25·19	83·13	12·0	250·6	—	0·90
17	79·10	65·67	3·85	21·60	83·02	5·86	304·0	3·61	1·06
18	81·05	66·77	3·71	19·75	82·38	5·55	338·1	10·03	1·18
19	84·73	69·52	4·97	17·67	82·05	7·15	393·4	18·75	1·37
20	85·56	70·19	4·70	15·54	82·03	6·7	451·7	25·54	1·57
21	81·60	66·9	8·26	19·43	82·00	12·3	344·3	11·08	1·20
22	78·74	64·13	4·11	22·46	81·44	6·41	285·5	—	1·00
23	83·83	68·24	5·43	19·54	81·37	8·0	354·6	12·10	1·23
24	80·36	65·0	7·78	20·75	80·90	12·0	313·2	5·79	1·09
25	82·08	66·3	7·81	18·17	80·79	11·78	364·9	14·10	1·27
26	85·29	68·48	5·34	17·02	80·29	7·78	402·3	19·58	1·40
27	80·28	64·34	8·08	20·89	80·14	12·56	308·9	4·32	1·07
28	83·20	66·40	8·26	18·07	80·00	12·4	367·4	14·48	1·28
29	77·07	59·24	8·28	26·21	76·87	13·97	226·0	—	0·79
30	85·87	65·21	8·24	16·68	75·94	12·64	391·6	17·29	1·36

In this table the sucrose content is assumed to be identical with the polarization, on account of the relatively low percentages of reducing sugar. Further, we have assumed that the solubility of sucrose in the water of the syrup is the

same as in pure water,* and that the sugar is grained at 45° C. According to Herzfeld's table on page 4, the solubility of sucrose in water at 45° C. is 71·32 parts of sucrose in 26·68 parts of water, or 241·7 parts of sucrose in 100 parts of water. When calculating from these values the amount of sucrose actually dissolved in the syrup, we can find by difference the amount of sucrose present in a supersaturated form, which could crystallize out at the given temperature if the syrup were strongly agitated.

Amount Crystallizing Out on Graining.—The quantities of sugar capable of crystallizing differ very considerably, and in Nos. 16 and 29 the graining point was very probably missed and the pan-boiler compelled to concentrate again before obtaining sufficient grain. It is evident that, for a given concentration, the supersaturation is greater in pure than in impure syrups, and therefore the former forms grain more readily than the latter, partly because a pure syrup contains more sucrose per 100 parts of dry substance, and partly because this sucrose crystallizes out more easily from pure than from impure solutions.

A few examples taken from the foregoing table will show how the crystallizable sugar varies with the purity of the syrup, although in reality this difference is somewhat compensated for by the lower solubility of sucrose in impure syrups.

Water.	Quotient of Purity.	Sucrose in Supersaturated Solution.	Water.	Quotient of Purity.	Sucrose in Supersaturated Solution.
19·58	87·95	13·70	21·59	90·5	9·67
19·54	81·37	12·10	21·74	87·06	5·54
20·88	89·93	12·23	21·60	83·02	3·61
20·89	80·14	4·32	22·67	88·68	5·98
21·45	84·89	5·91	22·61	87·45	3·99
21·49	83·37	4·75	22·73	87·40	2·19

The graining point is, therefore, not only determined by the density, but also by the purity of the syrups, and it is therefore not surprising when an experienced pan-boiler sometimes fails to grain a pan of syrup at the first attempt, only succeeding after a few trials.

* This may be done for syrup, but in molasses the solubility of sucrose is less.

Boiling.—Care should be taken, at the outset, to form as large a number of crystals as will be present in a full-grown state in the finished massecuite. When making a coarse grained sugar, fewer crystals will be required than when making a fine grained sugar. In the former case it is customary to grain "low down," that is to say, to start with only sufficient syrup to form the required quantity of grain, which is then allowed to grow by constantly drawing in syrup. In this case, we cannot make use of all the steam-coils, because steam may only be admitted to those which are covered by the syrup; consequently, boiling proceeds very slowly. But when making fine grained sugar, we may at once employ the whole heating surface, because the graining is performed "high up," when all the coils are immersed. The pan may, therefore, be filled at once and steam admitted to all the coils, and the whole mass be concentrated to the graining point. But when, owing to evaporation, the volume of syrup diminishes to such an extent that the topmost coils are exposed, steam must be shut off from these. It is evident that when graining high up, the boiling goes on more rapidly than when graining low down, although in this latter case the operation may be much accelerated by graining sufficient syrup in one pan to supply two pans with grain, the grained syrup being then divided between two pans with the same result as if each had been grained "low down." In this way, coarse grained sugar may be obtained in a pan heated by a steam chamber, which is not otherwise feasible.

By spreading a proof-stick sample upon a piece of glass plate, an experienced pan-boiler can judge whether the number of crystals in the grained syrup is sufficient for the kind of sugar required. If not, he re-dissolves the crystals by the addition of fresh syrup and by increasing the temperature, and tries again after he has concentrated a little further.

False Grain.—By the careful addition of more syrup, the grain is allowed to grow regularly, care being taken that the concentration of the liquid (in which the crystals float) does not become as high as before graining, since this might give rise to a secondary crystallization (i.e., formation of minute crystals between those already formed). When this happens, it is detected by the turbid appearance of the mother liquor in the samples from the proof-stick, and these crystals, known as "false grain," must be re-dissolved. To this end a large quantity of syrup is drawn into the pan, the injection diminished, and more steam turned on in order to raise the temperature. The fine crystals are thereby dissolved, the original crystals being somewhat diminshed in size; but, by causing the temperature to fall slowly, the latter commence to grow again. Syrup is drawn into the pan either in a continuous stream or at intervals until the pan is full. The pan-boiler must not force the concentration too much towards the end of the operation, because if false grain is now formed it can no longer be dissolved owing to imperfect circulation. We are then compelled to leave it in the massecuite, the consequences of which will be discussed further on.

Finishing.—When the pan is full, the supply of syrup is stopped and the contents concentrated to a Brix of 92-93° (a water content of 9-11 per cent.), usually at a temperature of 65° C. (149° F.) and a vacuum of 730 mm. (28·7 inches). The temperature and vacuum may be read from the gauges, but the exact point at which the concentration should be arrested, i.e., the striking point, has to be found by experience, and is determined by the firmness of a sample (taken by means of the proof-stick), when suddenly cooled in a pail of water.

The Brasmoscope.—Some years ago, Curin* constructed an instrument which he called a " brasmoscope," which indicates the density of the saccharine liquid boiling *in vacuo*. He started from the observation that between the limits of 57½-48½ cm. vacuum (22·6-19·1 inches), and 65-74°C. (149-165·2° F.) every cm. difference in vacuum (mercury pressure) corresponds to a difference of one degree Centigrade in the boiling point of pure water.

Therefore, if a barometer divided in cm. and a Centigrade thermometer be together immersed in water boiling *in vacuo*, the mercury will rise the same amount in both instruments within the limits mentioned above. This is shown by the following table of the tension of water vapour according to Regnault and Bloch.

Temperature in °C.	Tension of the Vapour in cm. Mercury.	Vacuum in cm. Mercury.	Temperature in °C.	Tension of the Vapour in cm. Mercury.	Vacuum in cm. Mercury.
64	17·89	58·11	70	23·33	52·67
65	18·71	57·29	71	24·36	51·64
66	19·57	56·43	72	25·43	50·57
67	20·46	55·54	73	26·54	49·46
68	21·38	54·62	74	27·69	48·31
69	22·34	53·66	75	28·88	47·12

The agreement is very close in the case of boiling water, but less so with syrup, especially when concentrated. But, when once the difference is ascertained empirically, the specific gravity of the boiling syrup can be determined from the readings of the thermometer and vacuum gauge. In Curin's apparatus a scale of degrees Brix is calculated for the differences between temperature and vacuum, enabling the density of the boiling liquid to be read off from a sliding scale.

* " Oest.-Ung. Zeitschr. f. Zuckerind. und Landw.," 1894, 756.

The brasmoscope is only used for syrups or molasses free from grain ; when grain is present it only shows the density of the mother liquor in which the crystals float. When the massecuite contains much grain, the temperature varies so much in different parts of the mass (owing to imperfect circulation) that the readings by no means correspond with the average temperature of the massecuite.

Claassen's Apparatus.—Claassen* found as the result of a great number of experiments that the crystallization of sucrose from boiling syrups and molasses proceeds most favourably when these liquids possess a coefficient of supersaturation proper to their quotient of purity. The coefficient of supersaturation is defined as the ratio of the normal solubility, at a given temperature, to the solubility of the supersaturated solution at the same temperature. If the coefficient becomes too high, sucrose crystallizes out too rapidly and forms separate crystals instead of adhering to the pre-existent ones. If it is too low, crystallization takes place too slowly, occasioning loss of time. The optimum coefficient of supersaturation is the same for syrups of the same quotient of purity, but rises in proportion as the purity of the juice sinks ; and since, during boiling, sucrose crystallizes out, and accordingly the purity of the mother-liquor falls, the coefficient at the end of the process will have to be higher than at the outset.

Claassen determined the most favourable coefficients for beet juices and molasses of every range of purity and found that whilst boiling a syrup massecuite it should not exceed 1·20 ; only at the end, when the purity of the mother-liquor is low, may the supersaturation be raised to 1·30.

The coefficient for after-products massecuites has to be kept higher than for massecuites from syrup, as has been shown by Claassen, who gave data for the proper coefficient for liquors of every quotient of purity.

Claassen constructed an apparatus by which the water content, and consequently the coefficient of supersaturation of the mother-liquor in the pan, can be constantly recorded during the boiling process ; this is drawn up on the same lines as Curin's, only is much more accurate.

He found the following data for beet juices :—

WATER CONTENT

Temperature of the boiling massecuite.	Purity of the mother-liquor.		
	80°	75°	70°
70	17·3	15·2	13·2
75	16·4	14·5	12·5
80	15·6	13·8	11·9
85	14·7	13·0	11·2
90	13·8	12·2	10·5
95	12·9	11·4	9·7

* "Zeitschr. Rübenzuckerind.," 1907, 1232.

During boiling the water content of the mother-liquor is at the temperature affixed.

Temperature.	During the first period.	During the second period.	During the last period.	When finishing.
70	12·5	12·0	11·5	11·0
75	12·0	11·5	11·0	10·5
80	11·5	11·0	10·5	10·0
85	11·0	10·8	10·0	9·3
90	10·0	10·5	9·5	8·7
95	9·5	9·0	8·5	8·1
100	—	—	—	7·7

Rules for boiling Syrup Massecuites.—Up to the present no data have been collected relating to cane sugar juices and molasses; in fact neither Curin's nor Claassen's apparatus seems to have found any application in cane sugar houses. Pan-boilers prefer to work by rule-of-thumb methods, which, it is true, have, up to now, given excellent results in experienced hands. In order to form a basis for that work we give here the general rules on the subject prepared by G. Bartsch* :—

1. The syrup should be clear, and 5° C. hotter than the boiling temperature prevailing in the vacuum pan.

2. The thinner the syrup the larger will the crystals become, and *vice versa*.

3. The bolder and the more regular the crystals required, the weaker the thread and the lower the final concentration. When making fine-grained sugar the massecuite should be strongly concentrated before every fresh portion of syrup is drawn in.

4. When making large crystals, the syrup should be drawn in in large portions at a time, and when making fine grain the portions should be small but frequent. In no case, however, should the supply of syrup be so large as to re-dissolve existing crystals.

5. The more regular and the larger the crystals desired, the slower and the quieter has boiling to proceed, while a brisk boiling is favourable to the formation of small crystals.

6. In the case of large crystals, the final concentration may be a high one, since large grain facilitates the spinning-off of the molasses in the centrifugal; great care ought, however, to be bestowed on boiling-off, as coarse-grain massecuites are liable to form false grain between the crystals.

* "Zeitschr. Rübenzuckerind.," 1921, 285.

7. When graining, the calandria or the heated coil ought to be kept some inches under the level of the syrup ; and, according to the size of the crystals required, the amount of syrup present in the pan at the moment of formation of the grain should range from 25 to 40 per cent. of the total quantity of syrup required for the boiling.

8. When graining, the coefficient of supersaturation, as shown by the appearance of the thread, should be such that after no more than one additional supply of syrup the necessary number of grains is present. This is a cardinal point for obtaining a regular crystal in the product. If in order to secure the required number of grains, syrup has to be drawn in more than once, the sugar will become of irregular appearance.

9. The boiling mass should be kept thin at the outset. If it becomes too thick, there is danger of false grain being formed. When the boiling is half completed, it should be still so thin that it runs easily out of the hollow of the proof-stick.

10. The lighter the colour of the sugar intended, the lower the boiling temperature should be kept.

11. False grain should be re-dissolved as soon as the stage of the boiling permits. In the second half of the boiling process no re-solution in water should be attempted for fear of entrainment. When re-dissolving the false grain by raising the temperature, the transition from a high to a low vacuum should be a slow and cautious one, after which a fresh supply of syrup should at once be drawn into the pan.

Discharging the Pan.—As soon as the massecuite is ready for discharging, the steam is shut off, the air-pump and injector stopped, and air admitted to the pan by opening a cock in the top of same. The discharge door can then be opened and the massecuite falls from the pan into suitable receivers.

Cutting.—When the grain is very small and a larger grain is wanted, only half the contents of the pan are discharged, and after being again closed fresh syrup is added and boiled, so as to permit the crystals to grow larger, a process which bears the name of " cutting."

Steaming.—When the pan is empty, a certain amount of massecuite always remains attached to the coils and to the walls of the pan, which, if allowed to stay, would become charred by overheating and produce dark-coloured lumps in the sugar of the next strike. Formerly, those crusts were removed by steaming out the pan, when they became loose and fell through the discharge opening. The steamings were kept separate, so that those from several operations might be cured together, or they were dissolved in the cane juice. But later, the discharge opening of the vacuum pan was frequently connected direct

with the crystallizers, rendering the separate collection of the steamings impossible. This difficulty sometimes induced pan-boilers to abandon steaming out their pans, with a deleterious effect on the appearance of the sugar. The best remedy is to draw clarified juice into the pan as soon as this is empty and ready to receive a fresh charge of syrup. This juice enters the pan through a pipe of 1 or 2 inches diameter, carrying a perforated nozzle at the top of the pan. The juice falls in a gentle shower against the coils and walls of the pan, detaching the crusts of sugar and dissolving them completely, while a fresh supply of syrup is being concentrated in the pan.

Composition of a Massecuite and its Molasses.—One of a series of analyses of massecuite and of the molasses occurring therein, between the crystals, is given below. In this example, a pure syrup was concentrated, grained, and boiled, and from time to time samples were taken from the pan and divided into two portions. One of these was analysed, and the other cured in a hand-centrifugal to separate the molasses, which was also analysed. The following results indicate the composition of the massecuite, and of the contained molasses at several stages of the boiling process.

Nature of the Sample and Stage of the Process.	Dry Substance.	Brix.	Polarization.	Sucrose (Clerget).	Reducing Sugar.	Ash.	True Purity.	Apparent Purity.	Temperature.	Vacuum.
Concentrated syrup at the graining point..	80·43	81·61	69·0	70·21	5·20	1·80	87·3	84·6	51	70
Mother-liquor separated after cooling ..	71·00	72·50	57·5	58·4	6·50	2·40	82·3	79·2	28	—
Massecuite (pan ¼ full)	90·50	91·7	77·3	79·0	5·50	2·09	87·3	84·3	57	71
,, (pan ½ full)	90·57	91·8	78·2	79·9	5·40	2·10	88·2	85·2	61	71
,, (pan ¾ full)	91·37	92·57	77·4	78·9	5·40	2·15	86·3	84·7	62	71
,, (pan quite full)	91·37	92·6	79·4	81·0	5·40	2·12	88·6	85·7	62	71
Molasses (separated hot)	78·97	82·6	52·0	55·8	12·50	5·93	70·7	63·0	62	—
,, (after cooling and filtration) ..	78·63	82·25	51·8	55·6	12·41	5·95	70·7	62·9	28	—

Solubility before and after Graining.—The concentrated syrup therefore contained 19·57 per cent. of water and 70·21 per cent. of sucrose at the graining point, or 358·8 parts of sucrose per 100 parts of water. At the temperature of 51° C. sucrose dissolves in water in proportion of 72·44 : 27·56. When this syrup had completely crystallized, showing the normal solubility of sucrose in the water present, the said water would have been able to dissolve $\frac{19{\cdot}57 \times 72{\cdot}44}{27{\cdot}56} = 51{\cdot}44$ per cent. of sucrose at 51° C., and 70·21 − 51·44 = 18·77 per cent. would crystallize out. In reality the figure for the solubility of sucrose is much less than the theoretical solubility, as is seen from the analysis of the mother-liquor, which was obtained later at a temperature of

28° C In this liquor, 29 parts of water contained 58·4 parts of sucrose, or 201·4 parts of sucrose per 100 parts of water. At that temperature, 100 parts of pure water can hold 215·3 parts of sucrose in solution. After complete cooling, only 39·41 per cent. sucrose is retained in solution, and 30·80 per cent. has crystallized out.

The syrup used in this experiment being of constant composition, the analyses of the contents of the pan do not show much variation at different periods. The molasses separated hot from the last sample contained 55·8 parts of sucrose on 21·03 parts of water, or 265·3 parts of sucrose per 100 parts of water at 62° C. The solubility of sucrose in pure water at that temperature is as 25·42 : 74·58, or as 100 : 293·4. Again the solubility of sucrose is less in the impure water of the molasses than in pure water. The cooled molasses was rendered turbid by minute sugar crystals, which separated during cooling, and which were so small that they could not be separated by filtration through asbestos or glass-wool, but passed into the filtrate. Consequently, the analyses of the filtered molasses gave almost the same results as the molasses before filtration. In this impure liquid the influence of temperature on the solubility of sucrose was the same as in pure water. On 100 parts of water $293{\cdot}4 - 215{\cdot}3 = 78{\cdot}1$ parts of sucrose must have crystallized out, or $\frac{78{\cdot}1 \times 21{\cdot}03}{100} = 16{\cdot}43$ on 100 parts of molasses. The extremely small size of the crystals prevented their direct estimation, so that it is impossible to say whether this figure was actually obtained, but the quantity did not appear to exceed a few units per cent. Hence, in these solutions the ratio of sucrose to water is not only influenced by the normal solubility of pure sucrose in pure water, but also by other circumstances of a much more complicated nature.

II.—MASSECUITE YIELDING WHITE SUGAR.

Nature of the Sample and Stage of the Boiling Process.	Dry Substance.	Brix.	Polarization.	Sucrose (Clerget).	Reducing Sugar.	Ash.	True Purity.	Apparent Purity.
Concentrated syrup at the graining point ..	80·5	81·5	72·1	72·18	4·93	1·50	89·66	88·3
Cold molasses after crystallization	70·68	72·4	60·87	61·10	5·74	1·84	86·45	84·0
Massecuite, pan ¼ full	89·36	90·49	80·00	80·16	5·43	1·63	89·70	88·4
Molasses contained therein	77·62	80·07	59·85	60·42	8·15	3·16	77·84	75·0
Massecuite, pan ½ full	89·18	90·13	79·92	80·10	5·34	1·65	89·98	88·6
Molasses contained therein	75·41	77·63	57·65	58·99	8·40	3·15	78·23	75·5
Massecuite, pan ¾ full	90·21	90·82	82·42	82·60	5·01	1·44	91·56	90·9
Molasses contained therein	78·21	90·55	61·54	63·03	9·00	3·25	80·59	76·4
Massecuite (pan full)	88·63	89·91	80·56	80·75	5·35	1·48	90·50	89·3
Molasses separated hot	78·76	80·74	58·96	59·18	10·63	3·56	75·14	73·0

Amount of Sucrose crystallizing in the Pan.—It is a fact that the quantity of sugar crystallizing from a massecuite increases as the water content diminishes, although this increase is not proportional to the increase in concentration. The more a massecuite is concentrated, the greater the quantity of sugar that crystallizes, but in practical working certain circumstances prevent the concentration proceeding beyond a certain limit, viz., about 6 to 8 per cent. of water, causing the composition of a first massecuite, boiled from average juice, to be :—

Sucrose	81·10
Reducing sugars	6·93
Ash	1·12
Water	8·66
Undetermined	2·19
	100·00

A massecuite from impure juice may give the following figures :—

Sucrose	74·10
Reducing sugars	11·07
Ash	1·54
Water	9·02
Undetermined	4·27
	100·00

But, though it may appear absurd, the quantity of crystallized sugar does not vary very much in the two cases, provided that the amount of water is approximately the same, as sugar is much more soluble in the liquid portion contained in a pure massecuite than in that of an impure one.

We may assume that in a pure massecuite, after being completely cooled, one part of water holds two parts of sucrose in solution, so that in the massecuite containing 8·66 parts of water, 17·32 parts of sucrose are in a state of solution, and 81·10 — 17·32 = 63·78 as crystals.

Limits of the Concentration.—If we could concentrate this massecuite further, every part of water which is evaporated should cause two parts of sucrose to crystallize, so that in a massecuite containing 3 per cent. of water, only 6 per cent. of sugar would remain dissolved and the balance be present in the crystallized form. It is, however, impossible to reach such high concentrations unless some radical changes are made in the *modus operandi*, because the last portions of sucrose to crystallize would not be deposited on the crystals already formed, but between them, as " false grain," owing to the total cessation of circulation in so stiff a mass. We have already remarked that perfect

circulation is an essential condition of crystalline growth. Moreover, in the absence of circulation, those portions of the massecuite which are in actual contact with the steam coils, or other heating surfaces, would become overheated and caramelized, and heat would cease to be transmitted to the more distant portions of the massecuite. Finally, it is not possible to get such a stiff massecuite out of the pan, and for these several reasons one is compelled to leave so much water in the massecuites that 12 to 18 per cent. of the sugar remains dissolved and is removed in the molasses during curing. But, as an appreciable portion of that sugar could be recovered as first sugar (owing to the fact that it passes over into the molasses as syrup), many devices have been suggested for increasing the fluidity of massecuites and the circulation in the pan, so as to induce a part of the dissolved sugar to deposit on the crystals already formed, and thereby be recovered in the first sugars.

It is, of course, quite as impossible to recover all this dissolved sugar, as it is to clarify juices so thoroughly that a pure sugar solution results. The juices will always contain certain impurities which have to be separated from the sugar crystals, and this separation is brought about by converting the sucrose into a solid (crystal) whilst the impurities remain in solution.

A part of these non-sugars has combined with sucrose to form syrupy combinations, while a second, not unimportant, part remains dissolved in that thick material adhering to the crystals. On increasing concentration, we very soon reach the point of 81° Brix, mentioned by Van Ginneken, beyond which the sucrose crystallizing out has a strong inclination to form minute grains instead of depositing on already existing crystals. This inclination would be strengthened by any defective mobility of the sugar crystals in the thick mass, such as hampers circulation and keeps off the sucrose molecules ready to crystallize out from the planes of those already present, thereby forcing them to form false grain. Practically, therefore, there is a limit to concentration, so that simple evaporation of the water from the syrup is not sufficient to cause all the available sucrose to crystallize out in one operation.

Increase of Mobility by Addition of Molasses.—It is, however, possible to restore the mobility of the growing crystals so necessary for their development in every sense, by adding molasses, obtained on curing a previous massecuite, to the already well-concentrated syrup massecuite in the pan. This molasses, saturated with sucrose as it is, does not dissolve sugar from the crystals, but dilutes the sticky mother-liquor adhering to them, and allows them to separate and so reassume their mobility. By this means they are enabled to attract the crystallizing sugar molecules to their surfaces during the subsequent evaporation and to grow regularly until at the end of the evaporation the vacuum pan is full of a mass of sucrose crystals swimming in a bath of a thick mixture, or a combination of sucrose, glucose, fructose, caramel, salts of organic and inorganic acids, which on further concentration do not give up any more

crystallized sucrose. As, in the case of impure solutions, the presence of a large amount of crystal surface is necessary to prevent formation of false grain, the proper crystallization is the more certain the smaller the crystals are, and therefore it is necessary for the highest recovery of sugar crystals from impure solutions that the grain be small and fine.

But even then crystallization from such impure solutions as are considered here is much too slow to be completed during the short time of boiling in the vacuum pan.

After the massecuite has been struck, crystallization goes on, even at high temperature, owing to the transformation of sucrose proceeding from the amorphous into the crystallized state, and later on, though to a much smaller degree, by the lesser solubility at lower temperatures. Owing to the great bulk of the liquid portion of the massecuite (sometimes amounting to 60 per cent. of its weight) which is necessary to ensure proper circulation, this after-crystallization yields an important amount of sugar, so that it is worth while to induce that sucrose to adhere to the pre-existent crystals and not to form minute crystals of its own account, which are liable to go to waste in the final molasses.

Practical Disadvantages of Complete Crystallization.—But in order to attain this end, a very copious addition of molasses to the syrup massecuite is necessary, and the capacity of the vacuum pan is diminished in proportion to the amount of this uncrystallizable material. Moreover, as much time and storage capacity are required to allow the last portions of dissolved sucrose to crystallize out from the hot mother-liquor, this method of effecting complete crystallization of first massecuites has had to be abandoned and other methods substituted for it.

Various Methods of adding Molasses.—In the first attempts, the concentrated syrup massecuite was mixed with about half its volume of hot molasses of about 80° Brix, and the mixture boiled for a quarter of an hour, and subsequently cooled in motion. As the molasses obtained on curing was of the same composition and purity as the molasses which had been added, this last had not lost any sugar, and only served for dilution. The advantages ascribed to this system were that a diluted massecuite can be discharged from the pan much more rapidly than a stiff one, and that steaming out is obviated by the absence of hard crusts on the coils, and adhering to the walls of the pan. A better yield of sugar is obtained on curing, and of better quality than from undiluted massecuite. But, as the purity of the molasses added to the massecuite was the same as that yielded by it (viz., from 55° to 60°), the increased yield was not as great as the inventors claimed.

Concentration of the Mixed Massecuite.—A great improvement was effected by concentrating the diluted massecuite to 93-94° Brix, which yielded molasses

of 50° purity. Some of the dissolved sucrose in the added molasses then crystallized out, thereby increasing the yield of the total massecuite. This improved yield necessitated a decreased yield of second sugar, but as first sugar fetched a higher price, the advantage was perceptible. In any case, there still remained a second product, because the volume of diluting molasses and the required storage capacity would become too great if the dilution were pushed so far that the finished massecuite yielded first sugar and exhausted molasses. For if the quotient of purity of the finished massecuite had to be reduced as low as 60° by diluting massecuite of 86° purity with molasses of 45° purity, only one-third of the pan capacity would be available for boiling syrup (undiluted).

Complete Crystallization in Two Stages.—In order to avoid these inconveniences, and yet obtain only first sugar and exhausted molasses, crystallization is now effected in two stages. The syrup massecuite is diluted in the pan with molasses of 45° purity, so that the purity of the mixture amounts to 75°, after which it is concentrated to 93° Brix. It is cured hot (before being completely crystallized) and the great bulk of the sugar is thus recovered as first product without loss of time or resorting to coolers. The molasses obtained on curing has also a purity of 45°, so that the added molasses only serves for dilution without being deprived of any sugar. In addition to the molasses added to the massecuite, recovered unchanged in composition and purity, we also obtain molasses of similar composition originating from the syrup in the massecuite, so that after the separation of the sugar, the total amount of molasses of 45° purity is increased. This is re-boiled and crystallized, so as to yield exhausted molasses of 30° purity once or twice a day, depending on the quantity of molasses and the capacity of the vacuum pans. To this end, a small quantity of syrup is boiled to massecuite, and mixed with as much molasses of 45° purity as will reduce the purity of the mixture to 60°. The mixture is concentrated to 95° Brix, or even higher, discharged into coolers, and cooled slowly in motion down to 45° C. After two or three days' cooling in motion, this massecuite is cured and then yields sugar of similar quality and appearance as the first product, and exhausted molasses of 30° quotient, which passes out of the manufacture. When the work is well conducted, as much exhausted molasses of 30° purity is removed every day as daily enters into the factory in the juice in the form of impurities or non-saccharine matter, and the quantity of molasses of 45° purity which circulates in the factory is just sufficient for the necessary dilutions and admixtures.

Decomposition of Molasses by Repeated Concentrations.—Consequently, in this process, a part of the molasses never leaves the factory, but is repeatedly returned to the pans, and being partially decomposed by repeated heating and concentration, it becomes acid and gummy, and contains so many impurities

that it is no longer possible to reduce the purity of the exhausted molasses to the desired point, as at the commencement of the operations. Moreover, the cured sugar develops an unpleasant smell owing to the adhering sour molasses, and might deteriorate during storage or transport. When such molasses was boiled separately, and the process started anew with fresh molasses, the purity of the exhausted molasses at once dropped to the desired figure of 30°, and this induced manufacturers to avoid keeping molasses in circulation for too long a time.

Avoiding Decomposition of Molasses.—This is accomplished in the following manner: a massecuite boiled from syrup is cooled and cured, yielding 30 per cent. of its weight of molasses of, say, 70° purity. This molasses is added to a second syrup massecuite, the mixture concentrated, cooled, and cured, yielding molasses of about 60° purity. This is again mixed with a syrup massecuite until the resulting molasses reaches the purity of exhausted molasses (about 30°). Now all the molasses in circulation is added to syrup massecuite, so that the mixture has a purity of 60°. The molasses obtained on curing this is exhausted, and can be removed and the process started anew with fresh syrup. In this way, all the available sugar may be obtained as first product after 4 or 5 days' work, leaving an exhausted molasses, the quantity of molasses remaining in circulation being small, so that overheating, sourness, and accumulation of decomposition products are avoided.

"Absolute Recovery" Process.—These operations have been further modified and led to the so-called "absolute recovery" process, now to be described in detail. In order to reduce the purity of the first molasses, that of the syrup may be reduced to 80° by mixing it with first molasses of, say, 60° purity. The resulting massecuites of 80° purity will yield molasses of about 60° purity. When the syrup is not higher than 80°, this mixing with molasses is omitted, but in the case of adding the concentrated molasses to the syrup care must be taken not to concentrate the latter to the same degree as when no molasses is mixed with it, for the concentrated molasses would raise the density of the mixture so high that too small a grain would result. The massecuite, having a purity of 80°, is cured hot, and yields first sugar and molasses of the same purity as that used for mixing with syrup, viz., 58-60°. Sufficient of this molasses is drawn into a second massecuite (of mixed syrup and first molasses of 80° purity) that the purity of the mixture becomes 70°; this is also concentrated and cured without cooling, and yields first sugar and a second molasses of 48-50° purity. Finally this is added to a third massecuite of 80°, until the mixture has a purity of 60°. This is highly concentrated, cooled gradually, and, on curing, yields sugar and exhausted molasses.

General Rules for the Series of Boilings.—These systems have undergone every kind of variation and change, according to the purity of the syrup, to the assortment of sugar to be made, to the capacity of the existing plant, etc. On pages 265, 266 and 267, we give a series of schemes in use in the different countries of production from which it will be seen that the general features common to all of them are :—

1. All massecuites are boiled to grain either with seed from syrup or from molasses sugar.

2. The first strikes are derived from syrup, either in combination with a seeding by molasses sugar or not, while no more molasses is added than is needed to dilute the stiff mass and allow it to run speedily out of the vacuum pan.

3. The second strikes are derived from a seed of syrup and the molasses from the first strike.

4. The third strikes, yielding exhausted molasses, are derived from seed of syrup and the molasses from the second strike.

5. The first and second strikes are centrifugalled hot without awaiting the after-crystallization.

6. The final strikes, yielding the exhausted molasses, have an apparent purity of about 60°. They are boiled slowly to a very high concentration, and cooled in motion over a long period (even a week or more) in order to get as much as possible the full effect of the after-crystallization.

Particulars of the Process.—The following general rules should be observed in executing the work :—

Before returning the molasses to the pan, it is heated with steam to a higher temperature than that of the already formed massecuite in the pan, because, otherwise, it would not mix properly with the massecuite, and by suddenly cooling it, a part of the dissolved sucrose would be precipitated as a "false grain." The molasses is steamed before being drawn into the pan, and the froth removed by means of wooden ladles. Steaming renders the molasses more dilute and easier to handle, it dissolves the fine grain usually present in it, and thereby eliminates the possible formation of a secondary crystallization, which causes much trouble in the centrifugals by choking the linings.

In some factories the molasses is diluted with water, but in most cases the steaming causes sufficient dilution. The steaming pipe must be perforated on its under surface, otherwise it may get choked by subsiding impurities when the steaming is temporarily stopped. As the heavy impurities remain in the molasses, such steaming and skimming operations are not very effective,

and it would be far better if the molasses could be completely clarified and thus be rid of all suspended impurities. As cane molasses is much too gummy for subsidation or filtration, the author suggests experiments with a high-speed centrifugal separator (2,000 revolutions per minute), by means of which the insoluble impurities might be deposited against the interior of the drum and the clear molasses passed out.

Calculation of Massecuite from Syrup and from Molasses.—The quantity of syrup massecuite which should be in the pan before molasses is drawn in, that is to say, the ratio between the massecuite boiled from syrup and from molasses, will depend on the purities of these two constituents and on the use made of the resulting molasses.

Pasma* gives the following relations between the purity of mixed masse cuites and the molasses obtained on curing without previous cooling :—

Massecuite of	90	purity	yields a	molasses of	75	quotient.
,,	88	,,	,,	,,	71	,,
,,	85	,,	,,	,,	65	,,
,,	82	,,	,,	,,	62	,,
,,	80	,,	,,	,,	60	,,
,,	70	,,	,,	,,	50	,,
,,	65	,,	,,	,,	45	,,

When it is necessary to add a molasses of 50° purity to a subsequent masse-cuite, the massecuite yielding such molasses must have a purity of 70°, and so on. But, if it is customary to use molasses of a fixed purity for all kinds of massecuite, the purity of the juice or syrup will decide the quantity of syrup massecuite which must be present in the pan when the molasses is added.

For example, supposing that molasses of 50° purity is usually added to the second massecuite, the mixed massecuite should then have a purity of 70°, and we get this by mixing x parts of syrup massecuite of purity a with $100 - x$ parts of molasses massecuite of 50° purity. We see that the value for x depends on that for a (purity of the syrup) and that the percentage of syrup massecuite that must be present in the pan before the molasses is drawn in, to form a mixed massecuite of 70° quotient, may be calculated from the formula :—

$$a \times x + 50\,(100 - x) = 100 \times 70$$
$$a \times x - 50\,x = 7000 - 5000$$
$$x = \frac{2000}{a - 50.}$$

This percentage becomes $\frac{2000}{85-50} = 57{\cdot}2$, for a purity of the syrup $= 85$;

and $\frac{2000}{80-50} = 66{\cdot}7$, for one of 80.

* "Archief voor de Java Suikerindustrie," 1904, 624.

This means that with syrup of 85° purity, 57·2 per cent. of the mixed contents of the pan must consist of syrup, and the balance of hot molasses. With syrup of 80° purity, this percentage becomes 66·7, and so forth.

Where the same pans are always used, it is advisable to calculate once for all the height to which each must be filled with syrup massecuite for different purities of syrup and of mixed massecuite, and to mark the outside of the pan, so that the pan-boiler may fill the pan with syrup massecuite to a certain mark in every instance before drawing in molasses.

Massecuites yielding "Green" Molasses are cured hot.—When molasses have to be re-boiled in the course of manufacture, the massecuites yielding such molasses need not be completely crystallized, and are therefore usually cured hot to save time and storage space. The purity of the molasses so obtained without previous cooling is known by experience, and serves as a basis for calculating the proportions between the two components.

Massecuites yielding Exhausted Molasses are cooled.—This, however, is not the case with massecuites yielding exhausted molasses, where every effort should be made to induce as much sucrose to crystallize as possible, and in the most favourable form to be recovered, because any sugar which fails to crystallize or which crystallizes as minute crystals which cannot be separated in the centrifugals, disappears in the exhausted molasses and is lost. The boiling, cooling, and curing of the molasses massecuites, therefore, require much attention and skill, also suitable plant, since any losses which occur cannot be rectified.

Shape of the Pan for boiling Molasses.—The vacuum pan used for boiling final massecuites should not be deep, the proof-stick should be fixed as low as possible, the sight-glasses extended to the bottom, and a good circulation maintained. These conditions are necessary to prevent the concentrated massecuite settling to the bottom, instead of mixing with the molasses, and thus frustrating the object aimed at. Pans provided with steam chambers, so-called calandria pans, are not suitable for boiling these massecuites, owing to the difficulty of gauging the quantity of syrup massecuite in the pan before introducing the molasses.

Method of boiling Final Massecuites.—When boiling a molasses massecuite, we first calculate the proportion of syrup massecuite and molasses to be added to yield a mixture of 60° purity. Usually the proportion of syrup massecuite is from one-quarter to one-third of the total volume. Such a massecuite must be kept free from false grain, and care must be taken to form sufficient grain as will ensure a proper proportion of fully developed crystals in the finished massecuite. The crystals increase in size during boiling, but their number should not increase. It is therefore advisable to grain high up, so that after two or three additions

of syrup and concentration, sufficient massecuite is in hand, and the introduction of molasses commences. The molasses is drawn in slowly and regularly, in not too large quantities at a time. The mixed massecuite is boiled at a rather high temperature, viz., 66° C. (150·8° F.), at a vacuum of 70 c.m. (27·5 inches), and concentrated to about 96° Brix. Just before discharging, the massecuite is warmed to 70° C. (158° F.) in order to facilitate its discharge from the pan, by slackening the speed of the air-pump and diminishing the injection. The steam supply being then turned off, and the air-pump and injector stopped, air is admitted to the pan and the hot massecuite discharged into coolers by opening the bottom door of the pan.

Composition of Massecuites and their Molasses.—A number of analyses of massecuites and the contained molasses at various stages of the boiling process are recorded here. They illustrate the composition of the contents of the pan and the gradual exhaustion of the mother-liquor during boiling.

I.—MASSECUITES MIXED WITH MOLASSES YIELDING "GREEN" MOLASSES.

No.	Nature of the Sample and Stage of the Process.	Brix.	Dry Substance.	Polarization.	Sucrose (Clerget).	Reducing Sugar.	Ash.	Apparent Purity.	True Purity.
1	Syrup	59·96	59·87	54·4	54·61	2·92	0·82	90·72	91·42
2	The same at the graining point	82·06	81·35	75·42	75·23	3·65	1·00	91·90	92·41
3	The same more concentrated	88·28	88·09	80·96	80·96	3·97	1·16	91·70	91·90
4	At point where molasses should be added	90·67	90·03	83·76	83·90	4·85	1·32	92·37	93·19
5	Mother-liquor	79·47	77·24	60·22	61·01	8·57	3·09	75·47	79·13
6	Molasses added	75·82	72·88	56·16	57·06	9·86	2·66	74·07	78·29
7	Mixture after two additions of molasses	91·0	89·22	79·8	79·2	5·50	1·61	87·71	88·77
8	Mother-liquor	77·09	73·56	54·51	55·62	8·30	3·33	70·71	75·61
9	Molasses added	75·74	70·76	55·22	56·12	9·34	2·85	73·17	79·31
10	Mixture after three additions of molasses	91·88	89·23	77·40	77·72	8·84	1·96	84·23	87·20
11	Mother-liquor	84·2	77·49	49·99	51·39	10·81	4·11	59·37	66·31
12	Massecuite finished	91·22	72·88	75·83	57·06	9·74	2·01	83·13	84·46
13	Molasses contained in it	82·13	77·37	50·19	51·65	10·92	4·23	60·94	66·76
	II.								
1	Concentrated syrup at the graining point	81·7	80·01	70·34	71·12	4·70	1·91	86·1	88·9
2	The same concentrated	90·3	88·9	77·6	78·7	5·2	2·12	85·9	88·6
3	Mother-liquor	78·7	75·2	48·6	49·1	11·4	4·80	61·7	65·3
4	Molasses used for mixing	84·2	80·39	52·0	52·7	11·0	4·90	61·7	65·1
5	Mixture after one addition	91·3	89·45	74·2	76·4	6·76	2·90	81·3	85·4
6	Mother-liquor	82·0	78·5	48·0	51·1	14·5	5·19	58·5	65·0
7	Massecuite finished	91·4	89·54	64·0	67·3	6·4	2·81	70·0	75·2
8	Molasses contained in it	82·1	78·49	39·0	43·9	14·3	5·18	47·5	55·9

We notice here a constant lowering of the quotient of purity of the mother-liquor in proportion as the evaporation of water proceeds, such evaporation being favoured by the better circulation after each addition of molasses.

The molasses separated from the above massecuites are mixed with syrup massecuites in other pans to form the following mixtures :—

II.—MASSECUITES YIELDING EXHAUSTED MOLASSES.

Nature of the Sample and Stage of the Process.	Dry Substance.	Brix.	Polarization.	Sucrose (Clerget).	Reducing Sugar.	Ash.	True Purity.	Conventional Purity.	Temperature.	Vacuum.
I.										
Concentrated primary massecuite ..	87·9	89·6	73·6	75·9	5·75	2·52	89·8	82·2	—	—
Molasses used for mixing	79·3	84·1	38·2	43·3	21·3	7·30	54·6	45·4	—	—
Mixed massecuite after 1 addition ..	91·0	93·6	64·8	69·6	12·5	4·28	76·5	69·2	48	71
Mother-liquor	81·4	88·9	36·6	42·3	21·3	7·12	51·9	42·6	48	—
Mixed massecuite after 2 additions ..	92·0	95·0	61·4	66·7	14·7	4·91	72·5	64·6	56	71
Mother-liquor	83·6	88·5	33·2	40·3	23·8	7·93	48·2	37·5	56	—
Mixed massecuite after 4 additions ..	91·9	94·7	62·0	66·8	13·5	4·37	72·7	65·4	60	71
Mother-liquor	84·8	89·9	31·8	39·5	25·6	8·28	46·6	35·3	60	—
Mixed massecuite finished	92·7	95·4	67·2	70·2	12·5	4·39	75·7	70·4	66	71
Mother-liquor	82·4	87·6	33·6	40·3	25·6	8·23	48·9	38·4	66	—
Molasses separated after cooling ..	82·0	88·3	25·0	31·8	30·1	10·2	38·8	28·3	40	—
II.										
Concentrated primary massecuite ..	89·6	91·2	75·4	77·9	6·0	2·50	87·0	82·7	—	—
Molasses used for mixing	77·8	81·6	42·2	44·8	16·1	6·23	57·6	51·7	—	—
Mixed massecuite after 1 addition ..	92·5	94·9	72·2	73·7	9·6	3·65	79·7	76·1	54	73
Mother-liquor	81·6	86·5	39·6	44·0	20·9	7·95	53·9	45·8	54	—
Mixed massecuite after 3 additions ..	92·8	95·3	67·4	69·6	10·9	4·21	75·2	70·7	58	71
Mother-liquor	84·2	89·6	36·2	42·0	22·8	8·69	49·9	40·4	58	—
Mixed massecuite after 5 additions ..	92·6	95·3	63·0	65·5	12·5	4·77	70·7	66·1	66	71
Mother-liquor	86·3	91·7	33·4	39·8	23·3	8·87	46·1	36·4	66	—
Mixed massecuite finished	94·1	97·4	57·6	61·6	13·9	5·28	65·5	59·2	68	71
Mother-liquor	84·7	90·7	30·6	37·3	24·4	9·55	44·0	33·7	68	—
Molasses separated after cooling ..	83·2	89·5	24·0	31·6	25·0	10·05	38·0	27·0	40	—
III.										
Concentrated primary massecuite ..	88·3	90·0	72·2	74·8	8·2	2·79	84·7	80·2	—	—
Molasses used for mixing	74·0	78·8	37·6	42·4	16·9	7·65	57·3	47·8	—	—
Mixed massecuite after 1 addition ..	92·4	94·8	68·8	72·2	7·46	3·92	78·2	72·6	52	71
Mother-liquor	83·8	88·6	40·3	45·2	14·4	7·67	53·9	45·5	52	—
Mixed massecuite after 3 additions ..	92·5	95·5	61·8	66·2	9·1	4·81	71·6	64·7	56	71
Mother-liquor	82·2	87·8	36·2	42·6	20·0	8·49	51·8	41·2	56	—
Mixed massecuite after 5 additions ..	92·2	95·7	61·1	65·4	11·1	5·39	70·9	63·7	59	71
Mother-liquor	86·0	92·0	33·0	40·1	22·5	9·51	46·6	35·9	59	—
Mixed massecuite finished	91·4	95·9	56·3	61·4	15·6	7·02	67·2	58·7	66	71
Mother-liquor	85·3	91·4	30·6	37·8	22·4	9·43	44·5	33·4	66	—
Molasses separated after cooling ..	80·04	86·2	23·8	32·0	23·8	9·65	40·0	27·5	40	—

IV.										
Concentrated primary massecuite ..	88·6	90·2	74·2	76·6	5·88	2·61	84·7	80·2	—	—
Molasses used for mixing	77·6	81·5	38·1	44·0	15·4	6·40	56·7	49·1	—	—
Mixed massecuite after 1 addition ..	91·4	93·8	65·6	69·8	9·4	3·87	76·5	69·9	58	70
Mother-liquor	81·6	87·44	37·2	43·6	17·8	7·20	53·4	42·5	58	—
Mixed massecuite after 2 additions ..	89·95	92·8	59·4	64·1	11·6	4·71	71·2	64·0	60	71
Mother-liquor	80·10	83·8	33·3	40·7	14·8	5·98	50·3	39·7	60	—
Mixed massecuite after 4 additions ..	89·3	92·7	56·8	61·4	12·8	5·10	68·7	61·2	66	71
Mother-liquor	84·4	89·2	33·2	40·5	20·0	7·98	48·0	37·2	66	—
Mixed massecuite finished	90·9	94·5	54·4	59·4	14·3	5·81	65·3	57·9	70	71
Mother-liquor	84·0	90·1	29·6	37·7	22·5	9·6	46·1	32·8	70	—
Molasses separated after cooling ..	83·2	89·4	24·6	31·6	24·1	9·9	40·4	27·5	40	—

The massecuites were discharged hot, and this explains why in some cases the purity of the mother-liquor of the finished massecuites is higher than at a previous stage of the boiling. We again notice a constant lowering of the purity of the mother-liquor as crystallization proceeds. The advantages of cooling are shown by the purity of the molasses obtained after cooling, being about ten points lower than that of the mother-liquor present in the hot massecuites.

Crystallization of Sucrose during Cooling.—This decrease in purity is really due to crystallization of sucrose and not to sourness or inversion during cooling, as is proved by the relation between the figures for reducing sugar and ash in the last mother-liquor and in the exhausted molasses. If the molasses contained more reducing sugar for the same amount of ash than the final mother-liquor, this would prove that the former had increased at the expense of the sucrose content and quotient of purity. In the examples cited above, these ratios are as under :—

	I.	II.	III.	IV.
Final mother-liquor ..	3·01	2·56	2·28	2·34
Exhausted molasses ..	2·95	2·49	2·46	2·43

and we are justified in concluding that as the ratios are not perceptibly disturbed no appreciable inversion or decomposition has taken place during the cooling process, and that the decrease in quotient of purity is not due to decomposition or inversion but to crystallization of sugar.

The four analyses mentioned above are examples of practical working, and show us how the mother-liquors become gradually exhausted with the formation of completely exhausted molasses.

The hot mother-liquors deposited sugar in the sample bottles, but the crystals were too fine to be determined quantitatively.

Ratio of Sucrose to Water in Final Molasses.—The ratio of sucrose to water in the mother-liquors and final molasses cannot serve as a measure of the solubility of sucrose in the water present in those liquids, since they chiefly consist of hydrated combinations of sugars and salts, in which the amount of sucrose depends on the amount of reducing sugars simultaneously present, and the amount of water on the degree of concentration of the finished massecuite. It is obvious that if concentration had been carried further, a few more per cents. of water would have been driven off from the saline combination, and although no sugar crystallizes out or becomes dissolved, the ratio of sucrose to water would be completely changed.

The above examples from practice show that the concentrations, etc., referred to attain the desired result, since an appreciably lower apparent purity of the molasses than 23° will hardly be possible.

Calculation of Massecuite from Sucrose.—Pasma* calculated the total quantity of massecuite obtained on working up the syrup by various methods, for different purities of syrup, viz., 90, 88, 85, and 82°, and on a basis of 100 parts of dry substance in syrup and in massecuites, so that the water present in the different products may be ignored. He classifies the methods of working as follows :—

A. Original method of adding the molasses in two operations (page 220).

a. With massecuites of 65° quotient, yielding green molasses and
,, ,, 60° ,, ,, final ,,

b. ,, ,, 70° ,, ,, green ,, and
,, ,, 60° ,, ,, final ,,

B. The more modern method of boiling with a minimum quantity of molasses in circulation (page 221).

With massecuites of 80° and 70° quotient, yielding green molasses and
,, ,, 60° ,, ,, final ,,

C. "Absolute recovery" process without circulating molasses (page 221).

With massecuites of 90°, 88° and 70°, at a purity of 90°
,, ,, 88°, 80° ,, 70°, ,, 88°
,, ,, 85° ,, 70°, ,, 85°
,, ,, 82° ,, 70°, ,, 82°
all yielding green molasses and
One massecuite yielding final molasses and possessing a purity of 60°.

All calculations are based on the figures for dry substance as determined by the degrees Brix, and on purity as calculated from the degrees Brix and polarization. The purity of the raw sugar is taken as a fixed value 97·5° (apparent). In the following formulæ we make use of these abbreviations :—

* "Archief voor de Java Suikerindustrie," 1904, 616.

m.c. = dry substance in mixed massecuite.
s = dry substance in syrup.
ret. m. = dry substance in returned molasses.
cent. m. = dry substance in centrifugalled molasses.
R = dry substance in sugar.

Further, we will suppose that, without cooling, a massecuite of
90 purity yields a centrifugalled molasses of 75 purity.
one of 88 ,, ,, ,, ,, ,, 71 ,,
85 ,, ,, ,, ,, ,, 65 ,,
82 ,, ,, ,, ,, ,, 62 ,,
80 ,, ,, ,, ,, ,, 60 ,,
70 ,, ,, ,, ,, ,, 50 ,,
65 ,, ,, ,, ,, ,, 45 ,,

Fundamental Formulæ.—The following formulæ may be used for calculating the proportion of dry substance from syrup occurring in a mixed massecuite of a given purity, which is formed by the mixing of that syrup and a molasses of a given purity.

$$x \times \text{quot. s} + (100 - x) \times \text{quot. ret. m.} = 100 \times \text{quot. m.c.}$$

$$x \text{ or s} = \frac{\text{quot. m.c.} - \text{quot. ret. mol.}}{\text{quot. s} - \text{quot. ret. mol.}} \times 100.$$

The quantity of returned molasses is therefore $100 - x$ or $100 - s$. The amount of sugar which may be obtained from a given massecuite can be calculated as follows :—

$$x \text{ or R} = \frac{\text{quot. m.c.} - \text{quot, cent. mol.}}{97{\cdot}5 - \text{quot. cent. mol.}} \times 100$$

and the amount of molasses obtained on curing becomes $100 - R$.

Calculation.—The calculation is for a purity of 90° in the syrup for the various methods described above.

A. *a.* 100 m.c. of 65 yield .. .. .. 61·9 molasses of 45.
For 100 ,, 65 is required .. .. 55·6 ,, 45.
100 m.c. of 65 thus yield a surplus of 6·3 ,, 45.

100 m.c. of 60° require 66·7 molasses of 45°, which are furnished by $\frac{66{\cdot}7 \times 100}{6{\cdot}3} = 1059$ m.c. of 65°.

1059 m.c. of 65 are derived from 470·2 m.c.
100 ,, 60 ,, ,, 33·3 ,,
1159 503·5 ,,

In this case 100 parts of syrup massecuite yield a total of $\frac{1159}{503 \cdot 5} \times 100 = 230 \cdot 2$ parts of mixed massecuite.

A. *b*. 100 m.c. of 70 yield .. .. 58 mol. of 50.
For 100 ,, 70 are required .. .. .. 50 ,, 50.
100 ,, 70 thus yield a surplus of .. 8 ,, 50.

100 m.c. of 60° require 75 molasses of 50°, which are derived from $\frac{75 \times 100}{8} = 938$ m.c. of 70°.

938 m.c. of 70 are derived from 469 m.c.
100 ,, 60 ,, ,, 25 ,,
1038 494 ,,

In this case 100 parts of syrup massecuite yield a total of $\frac{1038}{494} \times 100 = 210 \cdot 1$ parts of mixed massecuite.

B. 100 m.c. of 80 yield .. .. 46·7 mol. of 60.
For 100 ,, 80 are required .. .. .. 33·3 ,, 60.
100 ,, 80 thus yield a surplus of .. .. 13·4 ,, 60.

We saw from A (*b*) that

100 m.c. of 70 yield .. .. .. 58 molasses of 50 and
for 100 ,, 70 one requires .. .. 67 ,, 60 ,,
100 ,, 60 .. .. 75 ,, 50.
100 ,, 60 require .. .. .. 75 ,, 50 and
100 ,, 70 yield .. .. .. 58 ,, 50;

so that in order to supply sufficient molasses for 100 m.c. of 60°, there are required $\frac{75 \times 100}{58} = 129$ m.c. of 70°. For 100 m.c. of 70° are required $\frac{67 \times 100}{13 \cdot 4} = 500$ m.c. of 80°, or for 129 m.c. of 70° $\frac{129 \times 500}{100} = 645$ m.c. of 80.

100 m.c. of 60 are derived from 25 m.c.
130 ,, 70 ,, ,, 43 ,,
645 ,, 80 ,, ,, 432 ,,
875 mixed m.c. is derived from 500 mixed m.c.

so that 100 parts of original m.c. from syrup have yielded $\frac{875}{500} \times 100 = 175$ mixed m.c.

C.	100	m.c.	of	90	yield	..	..	..	33·3	molasses of 75.
	100	,,		80	,,	..	..	..	46·7	,, 60.
	100	,,		70	,,	..	..	..	58·0	,, 50.
For	100	,,		70	there are required			..	66·7	,, 75.
,,	100	,,		70	,,	,,	..	..	67·0	,, 60.
,,	100	,,		60	,,	,,	..	..	75·0	,, 50.

For 100 m.c. of 60° the molasses is furnished by 129 m.c. of 70°, for which the molasses is furnished by $\frac{129 \times 67}{46{\cdot}2} = 186$ m.c. of 80°. The molasses for this portion is again furnished by $\frac{186 \times 66{\cdot}7}{33{\cdot}3} = 372$ m.c. of 90°.

100	m.c.	of	60	are derived	from	25	original m.c.
129	,,		70	,,	,,	43	,,
186	,,		80	,,	,,	62	,,
372	,,		90	,,	,,	372	,,
787	mixed m.c.			are derived	from	502	original m.c ; or 100 parts of

original m.c. have yielded $\frac{787 \times 100}{502} = 157$ parts of mixed massecuite.

The figures for the purities of syrup of 88°, 85° and 82° may be calculated in the same way.

In order to make the calculation complete, the author calculated how much total massecuite would have come from 100 original m.c. if no molasses were returned, but had been boiled to string-proof as second products. In our instance of 90° and 88° purity we reckon 1 m.c. of first sugar for second, 1 for third, and 1 for last sugar; at a purity of 85° and 82°, only first, second, and last sugars are made, while at a purity of the syrup of 75°, only first and last sugars are made.

100	m.c.	of	90	yield	..	..	..	33·3	molasses of	75.
100	,,		75	,,	..	..	..	60	,,	60.
100	,,		60	,,	..	..	..	71	,,	45.
100	,,		45	,,	..	..	..	100	last m.c.	

The total amount is, therefore,

100	first m.c.	= 100
$\frac{100 \times 33{\cdot}3}{100}$	second m.c.	= 33·3
$\frac{33{\cdot}3 \times 60}{100}$	third m.c.	= 20·0
$\frac{20 \times 71}{100}$	last m.c.	= 14·2
		167·5

total amount of m.c. for 100 original m.c. from syrup.

In the following table the amounts of total dry massecuite are recorded, calculated on 100 parts of dry substance in syrup for the various quotients of purities and for the various methods of boiling.

Method.	Quotient of the Original Syrup.									
	90°		88°		85°		82°		75°	
Without returning Molasses..	167·5	100	173·0	103	163·5	98	180·0	108	153·0	91
A (a)	230·0	138	221·0	132	208·0	124	195·0	116	145·0	87
A (b)	210·0	125	203·0	121	195·0	116	180·0	108	154·0	92
B	175·0	105	170·0	102	—	—	—	—	—	—
C	167·5	100	160·0	95	155·0	93	158·0	95	151·0	90

With syrups of high purities, the differences in the total quantity of massecuite for the different methods are considerable, but they decrease with the quotient of purity and become imperceptible at purities of about 70°. We see from the table that with usually high purities of cane juice, the methods B and C yield much less massecuite than either of the methods A, but this advantage diminishes when impurer juices have to be worked up.

The required pan capacity does not bear any direct relation to the quantity of massecuite expected, because the duration of the boiling process is not identical for each method. It is evident that it takes more time to concentrate a massecuite consisting only of syrup than one consisting largely of concentrated molasses.

Syrup massecuites take longer to boil than those to which molasses are added, but actual figures cannot be given because account must also be taken of the time required for filling and emptying the pan. A pan which can be filled and emptied quickly may be charged once more in the same period than another in which those operations require considerable time, so that as every pan has its peculiarities the time required to boil any particular massecuite cannot be definitely stated. A single example may suffice however.

Supposing that a syrup massecuite requires 8 hours, then one consisting of half syrup and half molasses will require 7½ hours, a molasses massecuite, 6, etc., or on the average 7 hours (filling and discharging being included). If, as is the case in many places, the molasses massecuites are being boiled much slower and, e.g., occupy the pan for 12 hours, the average duration becomes more, but we shall stick here to the example cited.

Calculation of the Pan Capacity.—To calculate the pan capacity required for a factory crushing 1000 tons of cane daily, we proceed as follows :—

1,000 tons of cane yield 800 tons of normal juice containing 18 per cent. dry substance, or 144 tons of dry massecuite, or 158·2 tons of moist massecuite

containing 90 per cent. of dry substance. Assuming the purity of the syrup to be 85°, and treating this as in scheme C, we have 155 parts of mixed massecuite for every 100 parts of syrup massecuite, and for 158·2 tons of the latter, $\frac{158{\cdot}2 \times 155}{100} = 245{\cdot}2$ tons of mixed massecuite. The specific gravity being 1·513, these 245·2 tons occupy a space of 5724 cubic feet, and as the massecuites generally remain in the pans for 7 hours, the pan capacity for 1,000 tons of cane per 24 hours will be $\frac{5724 \times 7}{24} = 1670$ cubic feet.

Boiling String-proof.—Although at present most syrups and molasses are grained, yet sometimes molasses are boiled smooth or "string-proof"; in other words, they are so concentrated as to become supersaturated solutions at the temperature prevailing in the vacuum pan. The hot massecuite is then discharged into tanks or crystallizers, wherein the sugar is allowed to crystallize whilst the massecuite cools. The boiling is conducted as follows:—A vacuum having been created in the pan, molasses is drawn in until the coils are quite covered. Steam is then admitted, and concentration commenced, molasses being drawn in from time to time, so as to keep the coils constantly covered. Now and again a proof-stick sample is taken from the pan and drawn out between thumb and finger, forming a thread which breaks at a certain length. If the thread breaks too soon, the concentration has not been carried far enough; if it does not break at all, it has gone too far. The author is of opinion that the refractometer will prove a valuable guide as to the exact degree of concentration of the massecuite. At present the skill and experience of the pan-boiler alone determines this, partly by the "breaking length" of the thread, and partly by the appearance of the sample. Generally speaking, first molasses may be more highly concentrated than second or third, while the temperature at which molasses are boiled is rather high.

Whereas, in boiling syrup massecuites, the object is to induce sugar to crystallize in the pan, and to boil at a relatively low temperature, the object now in view is to concentrate the massecuite as much as possible without forming crystals in the pan, in order to obtain subsequently the maximum crystallization in the cooling tanks. The higher the temperature of such a molasses massecuite, the more sugar can it hold in a supersaturated state, and deposit as crystals on cooling. In order to avoid decomposing the reducing sugar, it is advisable not to exceed 70° C. during the boiling. When the massecuite is sufficiently concentrated, the steam and molasses supply are shut off, the air-pump stopped, and air admitted to the pan. The contents of the pan are then run into tanks or waggons placed under the discharge door.

In the chapter on massecuites boiled "smooth" further particulars will be given concerning the density and other properties of low-grade massecuites.

Concrete Sugar.—What is known as " concrete sugar " is sometimes made in several countries for direct consumption by concentrating syrup, until after cooling, it becomes a hard crystalline mass. The final point for the concentration of the clarified juice is found by taking up a portion of the boiling sugar from the pan and cooling it by blowing upon it when, if sufficiently concentrated, the sample hardens and becomes brittle. The concentrated mass is taken out of the pan, stirred with a pestle till it crystallizes, and then run immediately into moulds where it hardens and forms hard blocks. In other cases the hot concentrated syrup is led into drying trays, where it is spread evenly with a spade, and is pushed backwards and forwards over the tray until it thickens, which happens in about three minutes. After a quarter of an hour the crystals will have formed and have absorbed the molasses, and on being turned over and raked, the whole becomes changed into a soft, yellow crystalline powder free from lumps.

This method of making sugar has become obsolete, since the production of centrifugal sugar has made so much headway. Yet one does not want to omit all reference to the procedure of its manufacture from these pages, and here we mention briefly the different apparatuses that have been in use.

Fryer's Concretor.—In the year 1868 Fryer constructed an apparatus, which he called a " concretor." It consisted of a series of shallow trays, set at a slight angle and fitted with transverse partitions, forming baffle-plates, by means of which the juice was caused to flow across each tray six times and to traverse a total heating surface of 400 square feet. After leaving the series of trays with a density of 55°–60° Brix the syrup was further concentrated in a rotating drum, fitted internally with curved blades over which the liquor flowed, exposing a large surface to the heated air drawn through it by means of a fan or blower. After about twenty minutes the concentrated liquor was run out of the drum, when at a temperature of about 200° F. (93·5° C.), and of such a consistency that it set to a solid mass on cooling.

Other devices to the same end have been a triple effect, the action of which could be reversed by introducing the clarified juice into the third vessel and drawing off the concentrated syrup from the first, or hottest one, where crystallization did not take place. Or a series of jacketed pans, standing over a furnace, which pans were filled with paraffin oil having a high boiling point, which transmitted the heat of the fire to the concentrated syrup in such a manner that the latter boiled regularly without danger of being overheated or charred.

Finally, it is also possible to evaporate the syrup very rapidly in a Kestner single-effect evaporator, where syrup of 50° Brix can be concentrated to 2 per cent. of water in a few minutes by steam at 135° C. (275° F.) and afterwards cooled to about 100° C. (212° F.)

But, as we have said above, these processes now have lost almost all of their importance.

Solidified Molasses.—In many Java factories it is customary to concentrate the final molasses to a hard mass, called solidified molasses, which preparation can be shipped in bamboo baskets without any trouble.

For the manufacture of this product the final molasses is steamed, by which it becomes diluted; insoluble impurities rise to the surface, forming a thick layer of froth, which is skimmed off, a clear molasses of about 70° Brix resulting. This is concentrated in a vacuum pan as far as possible till all of the water is driven out and a thick liquid of melted molasses remains in the vacuum pan.

Concentration should be driven so far that a sample from the proof-stick cooled in water is so hard that it cannot be impressed by touching it between thumb and finger, and so brittle that it breaks when dropped on a concrete floor. In order to prevent decomposition and overheating as much as possible, the temperature in the vacuum pan should be low and regular, and the vacuum should be high. Accordingly, as the molasses froths violently during concentration, it is advisable to fill the pan no further than three-quarters of its capacity in order to leave sufficient room for frothing. Boiling over not only means direct loss, but also a great deal of trouble, as the tough mass may choke the vapour pipes and the save-all. The time of boiling should not exceed three hours, and after the proper point of concentration has been attained the mass should be cooled rapidly. To this end the steam supply is stopped, but the vacuum is maintained by the air-pump. This latter has a twofold signification: firstly to drive off the last traces of moisture, and secondly to remove the gaseous decomposition products escaping from the hot impure mass. Notwithstanding every precaution, the sticky mass resting on the hot coils undergoes some decomposition and gives rise to the formation of gases, which have to be sucked off constantly.

A thorough cooling may be attained by pumping a current of cold water through the coils, by which operation it is feasible to reduce the temperature rapidly to 60° C. or thereabouts. The air-pump is stopped, air is admitted into the pan, the discharge valve is opened and the thick mass oozes out slowly from the pan into baskets placed underneath. The current of this mass is so slow that one may hold it back with one's hands for a moment while the baskets are shifted, though the hands should be well moistened to prevent the hot molasses from adhering to the skin and scalding it. The bamboo baskets are lined with mats and with a paper covering.

It is advisable to fill half of a basket first and to stir the contents with a stick till the mass solidifies; after the first layer has become hard, the basket may be filled up and closed with a bamboo cover.

Usually the weight of the basket is 140-160 pounds nett.

Loss of Sucrose during Boiling.—Losses of sugar may occur during the boiling process, and may be brought about either by decomposition or by boiling over, both of which causes are more common in the case of molasses massecuites than with syrup massecuites.

It is true that in the case of syrup massecuites the boiling temperature is, as a rule, very low, but local overheating sometimes occurs, so that sucrose is decomposed. The author has several times detected notable percentages of caramelized substances in scale from the coils, indicating decomposition of sugar by overheating.

Scale on Coils.—A few analyses of such scale calculated to 100 parts of dry substance here follow :—

	Scale from Coils in 1st product vacuum pans.	Scale from Coils in 2nd product vacuum pans.
Loss on ignition	22·70	26·40
Silica	38·12	37·46
Phosphoric acid	—	1·65
Sulphuric acid	8·20	4·74
Carbonic acid	0·64	—
Iron oxide	13·29	23·34
Alumina	2·10	3·26
Lime	6·78	4·88

Boiling Over.—The mechanical losses during the boiling of syrup massecuites are unimportant, the risk of boiling over being small because, when the pan is full, the contents are no longer fluid.

Bartsch* gives the following rules for the prevention of boiling over of syrup massecuites : Boiling over may be occasioned by rapid changes in temperature and vacuum and by a too rapid rate of boiling. The syrup should be introduced into the pan slowly and preferably in a constant current. Its temperature should be only 5° C. higher than the one prevailing in the pan. The pan should not be filled above the level indicated by the design, and a space of two or three feet should remain empty. Every leak should be carefully avoided, and the air inlet should not dip into the massecuite.

In molasses massecuites, however, mechanical losses may be heavier since decomposition products sometimes accumulate to such a degree that spontaneous decomposition may be brought about by local overheating. The temperature then suddenly rises, with the evolution of gases, so that the whole mass begins to foam violently and passes upwards through the "save all" and air-pump into the condensed water gutter, leaving only a small quantity of massecuite in the pan.

Although the author is unable to give figures, he is convinced that the mysterious losses of sucrose sometimes experienced when boiling molasses massecuites are solely due to spontaneous decomposition of primary decomposition products of sugar. This decomposition is brought about by local overheating, and when once started cannot be stopped. As remarked above, the effects are inversion and loss by decomposition, owing to the sudden rise in temperature. This phenomenon will be further discussed on page 262, under the heading Froth Fermentation.

* "Zeitschr. Rübenzuckerind.," 1921, 285

CHAPTER IV

CURING AND FINISHING

I.—Curing of Massecuites Boiled to Grain

Principle.—The operations now to be described have for their object the mechanical separation of the massecuite into crystallized sugar and molasses, the so-called " curing " process. It is evident that the most advantageous way to effect this separation is that in which the maximum of crystallized sugar is obtained at once as first product, because if portions of it become again mixed with the molasses, they can only be recovered after much trouble and expense (and not even then entirely), in the less valuable after-products. It has been proved that curing is most easily effected, and with a minimum of loss, when the sugar crystals are well developed, and the mother-liquor limpid and free from so-called " false " grain.

All the preceding operations have co-operated to this end. The mills have extracted as much juice as possible from the raw material, clarification has removed matters liable to make the juice turbid, besides which a greater or less amount of the colloids has been precipitated, and the use of a moderate amount of lime has minimized the formation of viscous dark-coloured lime salts. Filtration, if applied, has rid the juice of all suspended particles. Concentration *in vacuo* has prevented overheating and decomposition of sugar ; and, finally, care has been taken during the boiling process to form large regular crystals, free from false grain. Notwithstanding all this, careless working after the massecuite leaves the pan may nullify all the advantages thus gained.

Crystallization on Cooling.—When discharged from the pan, the hot massecuite contains more sugar in solution than it can hold after it has been cooled down. In massecuites containing much mother-liquor, as in the case of syrup massecuites which have been diluted with molasses, the quantity of dissolved sugar is much greater than in undiluted massecuites. Not only is the amount of liquid larger, but owing to the slow crystallization of sucrose from impure liquids all the sugar capable of crystallizing has not yet assumed that state in the pan.

Crystallization at Rest.—We have already seen that the sugar crystals are in constant movement in the pan and come into contact with all parts of the liquid from which the sugar is crystallizing out, the crystals growing in size by sugar being deposited on them instead of forming new crystals. When such motion ceases owing to the massecuite being discharged into crystallizing tanks, or in cases where the massecuite is very stiff and no circulation exists, the sugar afterwards crystallizing out will assume the form of false grain.

With pure massecuites, containing but little false grain, the influence is not perceptible, but from impure massecuites (especially when boiled at a high temperature), a great deal of sugar will crystallize out afterwards and, even if the boiling is carefully conducted, will cause the cooled massecuite to contain so much false grain that trouble and loss will arise on curing it.

This is especially the case with massecuites boiled very close and afterwards diluted with a large quantity of hot molasses ; if these be cooled down without certain precautions, a mass would result that could not be separated into crystals and molasses. Pure massecuites can safely be cooled down in the crystallizers and then give a maximum yield on curing, but very impure ones should either be cured hot (in which case the sugar crystallizing afterwards cannot interfere with the curing) or they should be cooled in motion, which operation is indispensable for massecuites mixed with molasses.

When first molasses is to be mixed with a subsequent massecuite, the dissolved sugar will find opportunity to crystallize in the pan, and it is therefore superfluous to waste time and cooling space. Those massecuites may be cured hot, but as there is some danger of forming false grain when the whole of a massecuite cannot be cured off at once, and becomes cooled, it is advisable to provide the massecuite tank with a stirring apparatus, or to discharge the massecuites into a large tank in which an Archimedean screw revolves, and keeps them in motion.

Crystallization-in-Motion.—When, however, it is desired to obtain exhausted molasses from the cured massecuites, it is necessary to cool and stir previous to curing, so as to get all the dissolved sugar to crystallize in such a form that it may be easily recovered, that is to say, to induce it to deposit on the crystals already present. To this end, it is necessary to maintain a gentle movement and circulation throughout the whole mass, which, at the same time, is allowed to cool gradually. This so-called crystallization-in-motion does not increase the quantity of sugar crystallizing out (as has been repeatedly asserted), but imparts to the newly crystallizing sugar the same form as that which crystallized in the pan, and therefore yields a mixture of homogeneous sugar crystals and a limpid molasses which can be separated rapidly and without loss.

Apparatus for Crystallization-in-Motion.—The apparatus for crystallization-in-motion may be divided into two classes, viz., open and closed. Both consist of cylindrical vessels, sometimes provided with a " jacket " into which hot or cold water can be introduced, and fitted with a shaft with dashers arranged in spiral form, which, by revolving, keep the contents in regular and gentle motion.

The open vessels are uncovered, and the massecuite runs into them through an open trough. On the other hand, the closed vessels have an air-tight connection with the discharge outlet of the pan and with a mixer placed above the centrifugals, and are worked under a vacuum. An air-compressor enables them to be discharged by means of compressed air. When the highly con-

centrated massecuite, diluted with the necessary amount of hot molasses, is to be transferred from the pan to the crystallizer, steam is turned off from the pan, the air-cock opened, also the discharge valve, and air pumped out from the crystallizer. The diluted massecuite is drawn into the latter with great rapidity, when the pan is again ready for use. The crystallizer being now closed, the massecuite is slowly cooled in constant motion, after which compressed air is admitted and the discharge door opened, causing the cold massecuite to pass into the mixer of the centrifugals. In the open systems, the massecuite runs through an open gutter into the vessel, is stirred, and afterwards discharged into a suction-tank, from which it is conveyed to the centrifugals by means of a chain-pump or an elevator. Before the massecuite is run into the crystallizer, the apparatus should be heated in order to prevent the hot massecuite from depositing false grain on coming in contact with the cold plates. The shaft should already be in motion for, if started when the vessel is full, there is risk of breakage. During the stirring and cooling, the massecuite becomes harder, and frequently more hot molasses has to be added in order to prevent the dashers breaking. After some time, water is introduced into the jacket (in such apparatus as are furnished with one), and it is necessary to allow the air to escape from the jacket through a vent-cock, as otherwise the cooling is very irregular. In order to promote rapid cooling, the inner walls of the jackets must be occasionally cleaned to remove sediment deposited from the cooling water.

Progress of Crystallization during Cooling-in-Motion.—The following tables show the gradual decrease in sucrose content in the mother-liquor during the cooling process. A little massecuite was taken from the crystallizer from time to time, the molasses separated by filtration, and analysed in the usual way.

I. Composition of the Massecuite when discharged from the pan :—
Brix, 93·96 ; Pol., 68·4 ; Purity, 72·8°.

Composition of Mother-Liquor at different Stages of the Work.	Temperature in °C.	Brix.	Pol.	Purity.	Per cent. Crystals in m.c.
After 1 hour's cooling ..	65	90·36	40·1	44·3	48·13
,, 3 hours' cooling	63	90·0	39·4	43·76	48·50
,, 5 ,, ...	61	89·76	39·0	43·4	48·79
,, 7 ,, ...	60·5	89·62	38·6	43·0	49·15
,, 9 ,, ...	59·5	89·76	38·2	42·57	49·44
,, 11 ,, ...	58	89·76	38·0	42·33	49·54
,, 13 ,, ...	57	89·84	37·6	41·85	50·0
,, 15 ,, ...	56	90·0	37·7	41·89	50·10
,, 17 ,, ...	56	90·0	37·2	41·33	50·29

II. Composition of the Massecuite, Br. 91·32, Pol. 68·4, Purity 74·9°

Composition of Mother-Liquor at different Stages of the Work.	Temperature in °C.	Brix.	Pol.	Purity.	Per cent. Crystals in m.c.
Before cooling	69	87·86	43·0	49·0	46·38
After 2 hours' cooling	67·5	87·6	41·6	47·49	47·66
,, 4 ,, ...	66	87·3	41·0	46·97	48·02
,, 6 ,, ...	64	87·96	40·2	45·0	49·77
,, 8 ,, ...	62·5	87·62	39·9	45·5	49·27
,, 12 ,, ...	60	87·62	38·8	44·28	50·12
,, 16 ,, ...	57	87·62	38·2	43·6	50·49
,, 20 ,, ...	54·5	86·9	37·2	42·8	51·22
,, 24 ,, ...	52	87·76	37·0	42·17	51·67
,, 26 ,, ...	51·5	87·76	36·6	41·7	52·01
Separated molasses	—	83·56	36·4	43·58	50·51

III. Composition of the Massecuite, Br. 97·70, Pol. 61·80, Purity 63·2°

Composition of Mother-Liquor at different Stages of the Work.	Temperature in °C.	Brix.	Pol.	Purity.	Per cent. Crystals in m.c.
Before cooling	70	94·1	35·80	38·0	40·5
After 6 hours' cooling	60	93·7	33·83	36·1	42·2
,, 12 ,, ...	52	93·6	32·50	34·7	43·4
,, 18 ,, ...	47	93·2	31·33	33·6	44·3
,, 24 ,, ...	44	93·1	30·73	33·0	44·8
,, 30 ,, ...	30	93·0	30·30	32·5	45·1
Separated molasses	—	—	—	34·2	—

IV. Composition of the Massecuite, Br. 97·70, Pol. 61·30, Purity 62·7°

Composition of Mother-Liquor at different Stages of the Work.	Temperature in °C.	Brix.	Pol.	Purity.	Per cent. Crystals in m.c.
Before cooling	71	95·4	35·2	36·8	40·2
After 8 hours' cooling	61	94·1	30·9	32·8	43·9
,, 16 ,, ...	53	92·1	28·7	31·1	45·7
,, 24 ,, ...	45	91·7	27·0	29·4	46·9
Separated molasses	—	—	—	33·3	—

It will be seen that crystallization progresses continuously during cooling, the increase per cent. massecuite being respectively 2·16, 5·63, 4·6, and 6·7, on 52, 53·5, 59·5 and 59·8 per cent. of mother-liquor originally present, corresponding to an increase in crystals of 4·1, 10·5, 7·7, and 11·1 per cent. on molasses. As these figures are calculated from the degrees Brix and polarization, they do not strictly represent the dry substance and sucrose. As the true figures are given in the tables on pages 226 and 227, together with the apparent values, and as the decrease in purity during cooling is the same as in the above instances, we make use of those figures in the table given here.

Constituents.	I.		II.		III.		IV.	
	Last Sample.	Molasses.	Last Sample.	Molasses.	Last Sample.	Molasses.	Last Sample.	Molasses.
Dry substance	82·4	82·0	84·7	83·2	85·3	80·0	84·0	83·2
Sucrose	40·3	31·8	37·3	31·6	38·8	32·0	38·7	33·6
Water	17·6	18·0	15·3	16·8	14·7	20·0	16·0	16·8
Actual purity	48·9	38·8	44·0	38·0	45·5	40·0	46·1	40·4
Apparent purity	38·4	28·3	33·7	27·0	33·4	27·5	32·8	27·5
Sucrose on 100 water	229	177	244	188	264	161	242	194
Decrease in purity (actual)	10·1		6·0		5·5		6·5	
Decrease in purity (apparent)	10·1		6·7		5·9		5·3	
Calculated sucrose crystallized out ..	13·6		8·2		7·8		8·0	
Decrease in sucrose on 100 water ..	52		56		103		48	

Effect of adding Diluted Molasses.—About 9 per cent. sucrose on 100 parts of molasses has, therefore, crystallized out during cooling, which greatly reduces the amount of sugar dissolved in 100 parts of water. According to the figures for dry substance, massecuites Nos. I., II., and IV. have not been diluted with water or molasses during cooling, but No. III. has undoubtedly received an addition of diluted molasses, as seen from its originally high concentration. The ratio of sucrose on 100 of water is consequently affected, and does not represent the effect of after-crystallization. Attention may be drawn to the fact that, notwithstanding such dilution, the decrease in purity is not less than in the other cases, so that dilution has not caused sucrose to be dissolved, and has not even hindered its crystallization.

Experiments have been made on the crystallization-in-motion of molasses massecuite boiled to grain. First molasses was diluted, steamed and skimmed off, boiled to grain, and discharged into crystallizers and cooled in motion. As the fine grain did not subside rapidly, it was not necessary to agitate constantly. It was quite sufficient to rotate the stirrer axle of the cooler thrice in 24 hours for an hour. After two or three days the cooled second massecuite

could be cured. The following examples refer to molasses treated in the indicated manner. When cured, the sugar was purged with water, for which reason the purity of the molasses is in most cases higher than that of the mother-liquor in the massecuite.

Molasses of 66·7° purity was grained, highly concentrated, and cooled in motion during 2 × 24 hours. The analysis of the massecuite gave Brix 96; polarization 64·0; reducing sugar 11·1; purity 66·7°. The composition of the mother-liquor during the different phases of cooling was as follows :—

Sample taken.	Temperature in °C	Brix.	Polarization.	Reducing Sugar.	Purity.	Sucrose crystallized.
When discharged	73	93·2	40·0	18·2	42·9	40·0
After 12 hours cooling ..	65·5	91·9	35·6	19·2	38·7	43·8
,, 48 ,, ,, ..	51	91·4	32·8	20·2	35·9	46·1
Molasses obtained on curing	—	85·8	33·6	19·4	39·2	43·4

This massecuite was very stiff and had to be diluted with water, and yet the molasses adhered so firmly to the sugar that a good deal of water was required in purging, causing the purity of the resulting molasses to rise excessively.

A second molasses was boiled in the same way, but less concentrated; it was, however, stirred for a longer time and cooled to a lower temperature.

Analysis of the massecuite: Brix 92·6, Pol. 59·2, Reducing sugar 12·25, Purity 63·9°.

Sample taken.	Temperature.	Brix.	Polarization.	Reducing Sugar.	Purity.	Sucrose crystallized.
On striking ..	72	91·3	45·0	16·12	49·28	25·6
After 24 hours ..	60	89·7	36·8	18·31	41·02	35·2
,, 48 ,, ..	51	88·9	34·0	19·22	38·24	37·0
,, 72 ,, ..	44	88·6	32·8	20·83	37·02	38·9
,, 96 ,, ..	39·5	88·3	31·2	21·27	35·33	39·8
Separated molasses	—	87·4	34·0	19·84	38·90	38·0

This table shows that the crystallization during cooling follows the same course as was noticed in the case of first massecuites.

Rate of Temperature Fall during Cooling.—The temperature falls regularly during the cooling process, but of course more rapidly at first. In crystallizers provided with jackets, the contents cool more rapidly than in those which cool by contact with the air, but as the cooling must proceed slowly in order to obtain a regular crystallization, and be continued down to the desired temperature, nothing is gained by accelerating or retarding the rate of cooling.

Sax* mentions that in open crystallizers, without jackets, the temperature of the contents decreased 14-15° C. in the first 12 hours in some factories, and 10-12° C. in others, while in a jacketed crystallizer the decrease amounted to 2° C. per hour. It is evident that when the difference in the temperature between the massecuite and the atmosphere or cooling water is greatest, as at the outset, the decrease in temperature per unit of time is also greatest.

Mixing Massecuites with Diluted Molasses.—In many cases the massecuite is so concentrated (to 100° Brix, or 94 per cent. dry substance) that the stirring apparatus fails to keep the mass in motion, and dilution becomes necessary, as also for the subsequent curing operation. Having previously noted that a very high concentration of the massecuite is indispensable for a maximum yield of crystals and a well exhausted mother liquor, it seems strange that such a mass can be diluted with impunity, yet a massecuite concentrated to 100° Brix can safely be diluted to 96° Brix without dissolving the crystallized sugar.

First of all, the molasses surrounding the crystals is very probably more or less supersaturated and unable to dissolve sucrose even after a relatively large dilution by water. When water is added, the molasses will start by reaching its stable coefficient of saturation and therefore is not in a condition to dissolve any of the crystallized sucrose. And even if this point should be surpassed, the rate of solution in such a highly concentrated and such an impure medium is so small that during the short time in which the crystals are in contact with the molasses, the danger of a pronounced re-dissolution is not great. Such additions of water cannot be made by merely pouring water upon the concentrated massecuite, because these would not mix and the water would then dissolve some of the whole massecuite instead of only diluting the concentrated mother-liquor throughout the whole bulk of massecuite.

Therefore the water is added to the massecuite either through a perforated pipe in the bottom of the crystallizer, or hot diluted molasses of 80° Brix is poured on top of the massecuite. The hot molasses readily mixes with the highly concentrated molasses surrounding the crystals, so that the mixture becomes more fluid and in proper condition for the subsequent curing in the centrifugals.

* "Archief voor de Java Suikerindustrie," 1899, 246.

In Example III., on page 241, where molasses was diluted to 86 Brix, the purity of the molasses obtained on curing did not rise above 27·3, while, in another instance when a massecuite was concentrated to 99·5° Brix and diluted with hot molasses in the crystallizers, the resulting molasses gave 86·1° Brix and 26·8 apparent purity. A comparison of the ratios of ash to reducing sugar in the massecuite and the molasses showed that the low quotient of purity was really due to crystallization and not to decomposition or inversion.

Although it is not possible to state limits which will apply in every case, we may say that a highly concentrated mixed massecuite may be diluted down to 96° Brix; the limit of dilution allowed will not then be attained, still less exceeded.

Van der Linden* does not trust to the chance that no re-solution of already crystallized sugar will take place during the stay of the mixture of concentrated massecuite and diluted final molasses in the coolers. He recommends delaying the mixing of massecuite with molasses diluted to 85° Brix to the very moment of curing, and practising this addition in a gutter just previous to centrifugalling.

He advises one to boil to a high concentration, viz., 95–98·5° Brix, to cool rapidly in motion with the aid of both cooling by vacuum and by water circulation to the temperature of the atmosphere, and to cure after two or three days' cooling. Just before curing, the massecuite is mixed with sufficient molasses diluted to 85° Brix to facilitate the spinning off of the mother-liquor.

In a series of experiments in which six massecuites were treated in this way, the average (apparent) quotient of purity of the molasses spun off was 31·3°, with a minimum of 29·9 and a maximum of 33·2, while the average purity of another series of six similar massecuites treated in the old way by mixing in the coolers, amounted to 35° or 4° more. The cooling by vacuum had raised the concentration and therefore achieved the work begun in the vacuum pan.

Searby† mentions a number of *modus operandi* in use in sugar factories in the Hawaiian Islands, from which we quote here a few of the most characteristic, and observe that in those islands the cooling time is continued much further than in the instances from Java:

1. Base of the No. 1 molasses and syrup, if necessary grained, and finished up with molasses to 96° Brix, stirred 7 days in crystallizers, and a little water added to prevent supersaturation.

2. No. 1 molasses, 52° purity, boiled to grain and built up to 97°–97·5° Brix, stirred 8 days in crystallizers, and water added after 4 days to bring down Brix to 94°.

* "Archief voor de Java Suikerindustrie," 1922, III., 142; 1923, IV., 208.
† "Int. Sugar Journal," 1923, 309.

3. All No. 1 molasses boiled to grain, dropped at 99° Brix, stirred during 12 days in crystallizers, and water added after fifth day, the Brix falling to 95·5°.

4. No. 1 molasses, 54–62° apparent purity, boiled to proof; powdered sugar added, allowed to stand, cut and built up; cooled 3 to 5 days in crystallizers, and 4 to 6 days in large cooling tanks, dilution by steam or by heated molasses to 91° Brix. Na_2CO_3 added till low grade is alkaline to litmus.

5. No. 1 molasses, 50–53° apparent purity, grained and built up to 97–99° Brix, stirred during 7–10 days in crystallizers; addition of heated waste molasses to 92° Brix.

Curing in Centrifugals.—The separation of crystals from the molasses is effected in centrifugal drums with perforated walls, which are made to revolve at a high speed. The massecuite is poured into the open top of the drum, which is then set in motion, whereby the massecuite is forced against the perforated walls of the drums which retain the crystals, but allow the molasses to percolate through. Around the revolving drum is an iron casing, in which the molasses is caught and from which it escapes to a gutter.

Pug-mill.—Massecuite cooled in any apparatus for crystallization-in-motion is ready for curing, but a massecuite cooled at rest must first be broken up into loose crystals in a pug-mill, where it is mixed with molasses and reduced to a homogeneous mass, thus obviating the risk of charging the centrifugals irregularly.

The mixture of molasses-sugar and syrup or purge-syrup used as seed for the starting of fresh massecuite is also prepared in similar pug-mills.

Shape of Crystal an Important Factor.—The way in which a massecuite is boiled and cooled has considerable influence on the yield from the centrifugals. When the crystals are regular and well formed, and the molasses limpid and not turbid from minute crystals, the separation is effected very easily and without loss. But if the crystals are irregular in size, or if the massecuite contains false grain, a considerable part of the crystallized sugar passes away with the molasses and is lost as first product. The minute crystals form an emulsion with the molasses, which firmly adheres to the large crystals and can only be removed by a copious application of water. Apart from the minute crystals which escape with the molasses through the holes of the centrifugal gauze, the surfaces of the larger crystals are dissolved in the water and increase the loss. Finally, the molasses obtained is thereby diluted and can re-dissolve sugar from fresh quantities of massecuite with which it is mixed in the pug-mill or mixer.

But even if the massecuite on being struck from the vacuum pan is quite sound and exempt from false grain, it may show a great deal of minute crystals

in case during cooling a secondary crystallization takes place which spoils the good effect of an irreproachably conducted boiling. For this reason the cooling down of the massecuites previous to their being centrifugalled is of great importance and it is advisable to cool every one of them in motion.

With pure massecuites the difference between cooling at rest and in motion is small, as more sugar crystallizes in the pan and less on cooling, and the latter has a tendency to form larger crystals than is the case with impure syrups. As modern methods involve the introduction of molasses or syrups into the pan in one way or another, after-crystallization of first massecuites is becoming customary. Even if first massecuites are cured direct, it is still advisable to keep them in motion before curing, because if this latter operation be postponed owing to some accident, and the massecuites be allowed to cool at rest, an opportunity for the formation of false grain is given with all the inconvenience attached thereto.

A good example of the influence of cooling-in-motion on the form of the crystals, and hence upon the yield obtained on curing, is afforded by the following analyses of four massecuites, boiled in the same way from rather impure syrups, but of which three were cooled at rest, and one in motion.

Cooling.	Massecuite.					Molasses.					Sugar Crystallized.	Amount of Crystals obtained on Curing.	Loss of Crystals on Curing per [100 of Sugar.
	Sucrose.	Reducing Sugar.	Ash.	Water.	Quotient of Purity.	Sucrose.	Reducing Sugar.	Ash.	Water.	Quotient of Purity.			
At rest ..	74·1	11·07	1·54	9·02	81·44	32·9	28·59	3·91	23·37	42·86	61·62	51·51	16·40
,, ..	78·9	8·99	1·16	7·76	85·32	33·7	28·12	3·65	25·40	44·63	68·22	58·81	13·79
,, ..	79·0	8·47	1·15	7·82	85·72	32·9	27·52	3·72	26·12	44·67	69·09	55·28	19·88
In movement	77·0	9·62	2·51	7·49	83·45	35·3	26·54	6·70	22·94	46·40	64·00	64·00	—

It will be seen that the quantity of crystallized sugar did not differ much in all four cases, neither did the chemical analyses of the massecuites nor of the molasses; but there were marked differences in the quantity of sugar obtained on curing. While all the crystallized sugar in the massecuite cooled in motion could be obtained on curing, in the other cases, where the after-crystallization had taken place at rest, so much false grain had been formed that no less than from 13 to 20 per cent. of the crystallized sugar re-dissolved. This table therefore shows the great influence of the shape of the crystals on the yield obtained, and how important it is to pay close attention to the boiling of first sugar, where, for want of care, more loss can be suffered than at any other stage of the manufacture. Apart from improving the shape of the crystals, crystallization-in-motion has the further advantage of being a cleanly operation and free from mechanical losses.

Difficulties met with in Curing.—The curing of first massecuites, boiled without addition of molasses and derived from pure juice, does not present any difficulties, but it sometimes happens that the molasses present in the massecuite is so gummy that it does not permeate the crystalline layer, but remains inside, and cannot be removed by washing. The only improvement is to stop the centrifugal after it has run for a certain time, mix the sugar with the layer of molasses, and then centrifugal again ; but as the loosened mass does not distribute itself evenly against the walls of the drum, this device is not very satisfactory.

Sometimes massecuites are so stiff that the curing takes too long, and one bad strike of massecuite retards the whole curing plant for hours, and occasions a stoppage in the factory routine. In such a case, it should be ascertained that the centrifugals are running the prescribed number of revolutions, *e.g.*, 1200 per minute, as (owing to the slipping of the belts) a reduction of a couple of hundred revolutions per minute may remain unnoticed, but is sufficient to retard the curing.

It is also desirable to steam the centrifugal liners once every day and thus keep the meshes clear of sugar crystals which are apt to choke them.

Regulation of the Curing Work.—Assuming that the centrifugal plant is in good order, one centrifugal is charged with the massecuite which has to be cured. If this sample cures without any difficulty, the whole of the massecuite is transferred to the mixer of the centrifugals, and cured. But, if the trial sample cures badly, the massecuite is mixed with hot molasses and another trial made, If this shows no improvement, the massecuite may be added in small portions to larger quantities of easily cured massecuite and the resulting mixture cured with as little delay as possible. But if the quantity be too large to mix with other massecuites, it is better to store it in a tank for a fortnight, as the most obstinate massecuite is more easily cured after such a period of rest.

Crystals in the Molasses Gutter.—Sometimes sugar crystals are found in the molasses gutter which are much too large to have passed through the mesh of the centrifugal liners, and which indicate that the liners have been torn or otherwise damaged. Of course, it is impossible to obtain exhausted molasses with such a defect, however much care and trouble be bestowed on the boiling and cooling. It is well to point out this source of loss here, and to advise the daily inspection of the centrifugal liners, so as to ensure replacing the damaged ones before too much crystallized sugar has passed through them.

Purging.—It is not feasible to separate all the molasses from the sugar crystals in the centrifugals and, consequently, the crystals remain covered with a thin film of molasses after that operation. When making refining crystals from pure syrup massecuites, this film consists of neutral molasses.

of 60-70° purity, and, being harmless, it will not give rise to sourness or deterioration. But when making white or raw sugar from massecuites to which molasses has been added, the adhering film of molasses is of low purity and may cause sourness, so that it is necessary to remove this by pouring water on the sugar while the centrifugal is in motion. The water dilutes the molasses which then escapes through the perforated walls of the drum. This separation is called purging, covering, or washing, and must be performed very cautiously in order to avoid dissolving too much sugar at the same time. Usually, the water is sprayed upon the sugar whilst the centrifugal is in full motion, after allowing sufficient time for the molasses to pass out of the drum, leaving the sugar fairly dry. Owing to the rapid motion, every portion of the sugar receives its share of the water, which, after passing through the crystals, escapes through the liner. Various appliances have been devised to spread the water in a fine spray over the sugar in the drum, and so to moisten a large surface with a small quantity of water ; or a nearly saturated solution of less valuable sugar is used for covering, which, while it removes the molasses, cannot dissolve any crystallized sugar. Finally, steam is used for covering, or a jet of steam which carries air along with it ; the steam becomes condensed on the crystals, forming water, which dissolves the thin layer of molasses and is separated with this.

However carefully this work be done, it is impossible to prevent some sugar being dissolved during purging ; even when purging with sugar syrup much pure sugar will pass over into the molasses. When curing raw sugar, the molasses from which is to be returned to the pan, this dissolving of sugar is of minor importance, because it returns to the pan in a product of the same composition as that from which it came. But when curing a massecuite yielding exhausted molasses, any dissolving of the sugar in the purging water is to be avoided, since this would raise the sucrose content and purity of this waste product, and result in a palpable loss of sugar. Similarly, when making white sugar, the mixing of so-called "green" molasses with the purging syrup is irrational, because much water or steam is required to remove the last trace of colour from the crystals, and consequently so much sugar is dissolved that a large amount of covering syrup is obtained, having a higher purity than the original syrup, and which, when mixed with the molasses, reduces the yield of first sugar considerably.

Separation of Green Molasses and Covering Syrup.—When curing first massecuites for white sugar and mixed massecuites yielding exhausted molasses, it is necessary to keep the molasses quite separate from the purging water, and to collect them in separate receptacles. This has the further advantage that one may use a free amount of purging water, because the sugar dissolved therein returns to the pan in the form of a product of the same purity as that from which the sugar was derived, instead of passing into an inferior product (molasses).

The simplest way of performing this separation is to provide two gutters behind the centrifugals, one for the molasses proper, the other for the purer washings. A movable outlet from the centrifugal discharges the molasses into that gutter where it belongs by simply inclining it to one side or the other. This, however, is not quite sufficient, as the viscid molasses has not entirely left the outer drum when purging has started, so that a partial mixing of the two in the outer drum cannot be avoided. Moreover, the outlet is not shifted automatically, and there is consequently risk of this operation being sometimes omitted. Another method is an arrangement in the outer casing of the centrifugal, which necessitates the centrifugal being able to rotate in opposite directions by means of an alteration in the driving gear.

A third device is a centrifugal having two casings, one inside the other; each of which is provided with a discharge outlet. One casing is fixed, as in the ordinary type, while the inner casing may be shifted up or down by means of a lever. Whilst the true molasses is being separated, the movable casing is raised, so that the molasses is caught in the fixed one, and passes through its discharge pipe into the molasses receiver. As soon as purging commences the movable casing is lowered, and catches the purging syrup, which escapes through the other discharge pipe into a separate receiver.

All these appliances are very ingenious and work well when properly handled, but there is a risk that the workmen will sometimes omit to shift the levers when purging begins or ends. So long as there is a chance of doing something wrong, nothing will prevent careless workmen from doing so occasionally. When experiments or trials have been made with such apparatus they always worked admirably, but in practical working it will occasionally happen that the rich purgings will get into the receptacle intended for molasses or *vice versa*. The first eventuality is the more serious, because if the rich purgings are once mixed with the exhausted molasses they are lost, and the loss of sucrose in the waste molasses is unnecessarily increased.

Curing in Two Sets of Centrifugals.—To be on the safe side, the curing may be carried out in two sets of centrifugals. In the first, the poor molasses is separated, and the raw sugar then mixed with purging syrup in a pug-mill, and again cured in the second set of centrifugals. The fluid obtained from this second curing operation is partially used for mixing with the raw sugar, while the remainder is returned to the juice. Usually, the purity of such washings is superior to that of the syrup from which the first massecuites are boiled, and there is therefore no objection to their being returned to the clarified juice. It is not advisable to return them to the syrup, because, being of higher density, they might trouble the pan-boiler who prefers to work with syrup of uniform density, especially when boiling massecuite for white sugar. But when the purging syrup is returned to the clarified juice, it passes through the evaporating plant, and a uniform syrup is supplied to the vacuum pans without danger of loss of sugar.

Instead of employing two sets of centrifugals, it has been proposed to separate the true molasses in a very large centrifugal of special construction and open at bottom. The massecuite enters it from the mixer when the centrifugal is running at full speed, the sugar clinging to the walls of the basket, while the molasses passes through. By slackening the speed of the machine so as to reduce the centrifugal force, the sugar is caused to slip down until it falls through the open base of the basket into a second pug-mill, where it is mixed with purging syrup and then finally cured in the ordinary centrifugals.

Molasses Sugar.—We have noted that white sugars are cured twice, and that raw sugars, from massecuites which yield rich molasses, may be purged in the centrifugals without trouble. It now remains to state what is done with the sugar cured from second or third massecuites which yield exhausted molasses. It is evident that purging would, in this case, cause irreparable loss of sugar, so that curing separates the massecuites into exhausted molasses, and a moist sugar containing a considerable quantity of the same exhausted molasses. In an earlier chapter, we explained that in order to obtain a well-exhausted molasses, the crystals in these last massecuites ought to be small, so as to present a sufficient surface whereon fresh sugar may deposit when assuming the crystallized state. If complete exhaustion of the molasses is aimed at, we have now to deal with a very fine-grained sugar, coated with exhausted molasses, and the problem is how to dispose of it to the best profit.

When there is a demand for such sugar, the problem is solved by selling it, but in many cases such low-grade sugar is not wanted, and the manufacturers have to work it up in some way or other.

Mixing with First Massecuites in the Coolers.—In cases where a fine-grained first sugar is not objected to, the most rational and economic plan is to transform this low-grade sugar into first sugar, by boiling the first massecuites to fine grain and mixing this with the low-grade sugar in the crystallizers. The first massecuites contain crystals and a mother-liquor of about 50° purity and, when mixed with low-grade sugar (consisting of crystals and exhausted molasses) the crystallized sugar from both sources is recovered as a uniform grain, and a mixed molasses separates, the purity of which will be higher than 30° but below 50°. This mixing has the same effect as if the low-grade sugar were mixed with rich syrup in a mingler before being cured a second time, and this operation is therefore equivalent to curing in two sets of centrifugals. After being well mixed, the mixture of massecuite and sugar is cured, the sugar is purged with water, syrup, or steam, and the resulting washings allowed to mix with the molasses, because these will be reboiled to form a second massecuite. In this way, the low-grade sugar is got rid of without undergoing any loss of the adhering exhausted molasses, the latter being replaced by a much purer molasses, which, if necessary, may be removed by purging water, because the

portion of sugar dissolving during that operation is recovered in the form of a product of the same purity as the original massecuite.

It is evident that the purity of the rich mother-liquor, surrounding the crystals of the first massecuite, will be reduced by being mixed with the exhausted molasses introduced in the form of low-grade sugar, and this reduction in purity depends on the purity and quantity of low-grade sugar added.

Starting from a pure juice, the quantity of low-grade sugar will be much less than in the case of an impure juice, and, consequently, the decrease in purity will be much larger in the second case than in the first.

Let us assume an example in which a first massecuite of 80° purity yields molasses for a second mixed massecuite of 70° purity, the molasses from which is re-boiled to a molasses massecuite of 60° purity. We can then calculate how far the purity of the mother-liquors deteriorates by the return of the low-grade sugars to the first massecuites.

We will assume that in one case a low-grade sugar of 90° polarization is returned, and in another case one of 85° polarization, and that they are returned into a massecuite of 80° purity or into one of 70° purity, giving one example of each.

Sugar having 99° Brix and 90° polarization (yielded by a massecuite containing mother-liquor of 30° purity) shows a purity of $\frac{90 \times 100}{99} = 91°$ and contains $\frac{91 - 30}{100 - 30} \times \frac{99}{100} = 86$ per cent. crystallized sucrose and 14 per cent. molasses. 100 parts of molasses massecuites of 60° purity yield $\frac{60 - 30}{91 - 30} \times \frac{95}{99}$ $= 47{\cdot}2$ parts of moist low-grade sugar, which contain $\frac{47{\cdot}2 \times 14}{100} = 6{\cdot}6$ parts of exhausted molasses.

A sugar having 98° Brix and 85° polarization shows a quotient of 86·7°, and contains 79·4 per cent. of crystallized sucrose and 20·6 per cent. of molasses, while 100 parts of molasses massecuites yield 50 parts of moist low-grade sugar containing 10·3 parts of exhausted molasses.

According to Pasma's figures,* and working by Scheme B, we have on every 100 parts of molasses massecuite 130 parts of massecuite at 70°, and 650 at 80°. When all the molasses sugar is returned into the massecuite at 70°, then, in case of a sugar of 90° polarization, 6·6 parts of exhausted molasses are mixed with 130 parts of a massecuite of 70°, or in 100 parts $\frac{6{\cdot}6 \times 100}{130} = 5{\cdot}1$ (For simplifying the calculation the water contents of the different massecuites are assumed to be the same.) We assume all the massecuites to be 95° Brix, and all the molasses to be 90° Brix and that, according to Pasma, a massecuite of 70°

* "Archief voor de Java Suikerindustrie," 1904, 614; and page 223 of this Work.

purity gives molasses of 50° purity, and one of 80° purity gives a molasses of 60° purity.

100 parts of massecuite at 70° purity contain $\frac{70-50}{100-50} \times \frac{95}{100} = 38$ parts of crystals, and 62 parts of mother-liquor of 50°. To these are added 5·1 parts exhausted molasses at 30° purity, causing the purity to decrease as follows :—

$$\begin{array}{rr} 62{\cdot}0 \times 0{\cdot}50 = & 31{\cdot}00 \\ 5{\cdot}1 \times 0{\cdot}30 = & 1{\cdot}53 \\ \hline 67{\cdot}1 & 32{\cdot}53 \end{array}$$

48·5° or a decrease of 1·5°

If the low-grade sugar had polarized 85°, $\frac{10{\cdot}3 \times 100}{130} = 7{\cdot}9$ parts of exhausted molasses would have been mixed with the 62 parts of mother-liquor bringing down the purity by 2·3 degrees as here shown :—

$$\begin{array}{rr} 62{\cdot}0 \times 0{\cdot}50 = & 31{\cdot}00 \\ 7{\cdot}9 \times 0{\cdot}30 = & 2{\cdot}37 \\ \hline 69{\cdot}9 & 33{\cdot}37 \end{array}$$

47·7° or a decrease of 2·3°

If, on the contrary, the low-grade sugar had been mixed with massecuite of 80° purity, the decrease would have been much less, owing to the considerable quantity of that massecuite.

On every 100 parts of molasses massecuite, we have not less than 650 massecuite of 80° purity. 100 parts of this contain $\frac{80-60}{100-60} \times \frac{95}{100} = 47{\cdot}5$ parts of crystals, or 52·5 parts of molasses at 60°.

6·6 parts of exhausted molasses are added to 650 parts of first massecuite, or on 100 parts, $\frac{6{\cdot}6 \times 100}{650} = 1$ part in one case and $\frac{7{\cdot}9 \times 100}{650} = 1{\cdot}2$ parts in the other case. The decrease in purity in the case of sugar of 90° becomes $60 - \frac{52{\cdot}5 \times 0{\cdot}60 + 1 \times 30{\cdot}0}{53{\cdot}5} = 0{\cdot}5°$ and in the case of sugar of 85° $60 - \frac{52{\cdot}5 \times 0{\cdot}60 + 1{\cdot}2 \times 0{\cdot}30}{53{\cdot}7} = 0{\cdot}7°$.

When calculating these figures for other purities of the juice, we obtain the following table for the decrease in purity of the mother-liquor in the first and second massecuites, when all the low-grade sugars are returned to them.

	Quotient of Purity of the Syrup				
	90	88	85	82	75
Parts of 1st m.c. on 100 molasses massecuite ..	650	451	463	162	56
,, 2nd ,, 100 ,, ..	130	127	123	118	118
When returning sugar of 90° Polarization					
Parts of waste molasses on 100 parts of 1st massecuite	1·0	1·4	1·4	4·1	11·8
,, ,, ,, 100 ,, 2nd ,,	5·1	5·2	5·2	5·6	5·6
Decrease in quotient of mother-liquor 1st massecuite	0·5	0·8	0·8	2·4	4·2
,, ,, ,, ,, 2nd ,,	1·5	1·6	1·6	1·6	1·6
When returning sugar of 85° Polarization					
Parts of waste molasses on 100 parts of 1st massecuite	1·6	2·3	2·2	6·4	18·4
,, ,, ,, 100 ,, 2nd ,,	7·9	8·1	8·4	8·8	8·8
Decrease in quotient of mother-liquor 1st massecuite	0·7	1·3	1·2	3·5	5·8
,, ,, ,, ,, 2nd ,,	2·3	2·3	2·4	2·5	2·5

We notice that, when returning low-grade sugar into second massecuite, the purity of the mother-liquor does not decrease more when the original purity of the syrup is high, than when it is low.

On the contrary, this purity exerts a great influence on the quantity of low-grade sugar obtained, which rises considerably when the purity of the syrup falls. This is clearly shown in the following table, where all the figures are calculated on 100 parts of dry substance in syrup.

	Quotient of Purity of the Syrup.				
	90	88	85	82	75
Total amount of massecuites ..	176	160	155	158	151·5
Quantity of molasses-massecuite ..	20	25·4	33	42	60
,, sugar of 90°	9·4	12·	15·6	19·8	28·3
,, ,, 85°	10	12·7	16·5	21	30

Whereas, at a high purity of the syrup, the amount of low-grade sugar to be returned is not excessive and will not cause any trouble, it increases considerably when the purity of the original syrup falls below 85°. Under these conditions, it is not wise to return all this sugar into first or second massecuites, since by doing so the centrifugal work will be increased by 25 per cent.

When, therefore, the juice is impure, and consequently the amount of low-grade sugar becomes excessive, it often happens that the centrifugals fail to do

the required work properly, and that the low-grade sugar still contains an excessive quantity of molasses owing to its being imperfectly cured. By doing so, much more exhausted molasses is returned to the first massecuites than the calculated quantities, so that the purity of the mother-liquors in such massecuites decreases much more than might have been expected. In such cases, when it is seen that the amount of low-grade sugar is getting too large to be properly dealt with, it is best not to force matters, but to cure only so much massecuite as the centrifugal plant can deal with properly, and to store the remainder in tanks until an opportunity of curing it occurs.

Low-grade Sugar as Seed.—This mixing of low-grade sugar into first massecuites can only be done when the size of the grain is of no importance. To obtain a well-exhausted molasses, it is essential that the grain of the low-grade sugar be fine. In order to mix this with first massecuites, it is evident that the grain of this ought to be fine also, otherwise the mixed sugar will contain small and large crystals, and will, moreover, give rise to many difficulties in refining operations.

When a large-grained first sugar is desired (which is generally the case since one has experienced that such a sugar has a better keeping quality than the fine-grained raws), such a mixture is no longer feasible, and the low-grade sugar may then be used as seed in boiling these massecuites as follows :— The sugar, well cured in the centrifugals (but without purging), is mixed with syrup and the mixture drawn into the pan and concentrated. The syrup washes off the adhering molasses leaving the sugar crystals clean and brilliant, just as when graining in the ordinary way. The quantity of grain thus added must be sufficient (as when graining syrup) to yield the requisite number of fully grown crystals in the finished massecuite. The small crystals are allowed to grow by the addition and concentration of more syrup, and the massecuite finished in the usual way. A great advantage of this method is that the hot syrup dissolves all the smaller grains from the added sugar, so that ultimately the seed consists exclusively of well-shaped crystals.

The calculation of the amount of molasses returned into the massecuite is the same as in the case of returning these sugars into the crystallizers.

If the quantity to be returned is relatively small, this process has no disadvantages, but in case of a low purity of the syrup the same impediments arise as have just been pointed out above.

Van Welie advises one to cure the molasses sugar separately in a centrifugal to a purity of about 97°, and to mix the white finely-grained sugar obtained with syrup as seed for the first massecuites. The impure purging syrup obtained on this operation, having a purity of 48-50°, is kept apart and drawn into a vacuum pan in which a final massecuite is being boiled. The impurities of that liquid do not therefore interfere with the graining, while the sugar contained therein serves to build up the crystals already present in the massecuite.

In cases where the amount of molasses sugar is too large to be disposed of in one or other of the ways indicated above, we are compelled to melt them in the juice, and boil them into first sugar, an operation that cannot be performed without loss, which in this case is unavoidable.

The following table shows the composition of a number of sugars intended for re-melting, all of which are taken from practical working. The percentages of crystallized sucrose and of adhering molasses are calculated from the reducing sugar content of the second sugars and the molasses separated from them.

No.	Kind of Sugar.	Polarization.	Reducing Sugar in		Per cent. Crystal.	Molasses.
			Sugar.	Molasses.		
1	1st Molasses sugar ..	86·8	3·93	14·86	73·6	26·4
2	1st ,, ,, ..	77·2	4·07	7·04	43·0	57·0
3	2nd ,, ,, ..	84·8	5·5	23·9	77·0	23·0
4	3rd ,, ,, ..	80·5	8·09	30·5	73·5	26·5
5	3rd ,, ,, ..	76·7	9·1	29·0	68·6	31·4
6	3rd ,, ,, ..	74·0	10·0	25·0	60·0	40·0
7	3rd ,, ,, ..	70·3	10·0	22·9	56·3	43·7

When a product, containing a great percentage of exhausted molasses, is to be returned, the question arises as to which stage of the manufacture is most suitable. Some melt the second sugars in the heated mill-juice, by allowing the latter to flow over a sieve on which this sugar is piled ; others melt it in the subsided clarified juice, while others run off the syrup at a relatively low density and bring it to its proper concentration by dissolving the second sugar in it. All these methods have their advocates and opponents. No loss is to be feared from dissolving these sugars in the syrup, but then the mistake is made of mixing the syrup that has been carefully clarified and neutralized with the (as a rule) acid sugars, and contaminating it with their impurities. We can only do this on condition that the syrup is afterwards thoroughly eliminated, neutralized, and allowed to settle. The same must be said with regard to melting the seconds in the clarified juice; in this case also, an acid and impure body is dissolved in a clarified and neutralized juice, though this is preferable to melting the seconds in the syrup, because they dissolve much more readily in thin-juice than in the concentrated syrup. One drawback to this process is the incrustation of the tubes of the triple effect by the suspended impurities which settle on them during concentration.

From the point of view of obtaining a good clarification, the introduction of the seconds into the mill-juice is more to be recommended, as they will then

again be submitted to all the processes of clarification, against which may be set the drawbacks of the heavier juice subsiding slowly, and of the juice in the scum containing more sugar, so that more is lost in the scum-cakes.

The re-melting of seconds is usually a very wasteful operation. As there is not always an opportunity of disposing of them, they are frequently heaped up, and at the end of the grinding season, a large quantity of seconds have to be melted in an insufficient quantity of juice, causing the last massecuites of the season to consist almost exclusively of re-melted seconds. The waste due to re-melting is therefore a mechanical loss.

Returning Seconds into the Vacuum Pan.—This may be obviated by dissolving the second sugar in a little hot clarified juice to the concentration of syrup, skimming and neutralizing, and drawing it into the pan, in which a first massecuite is already half finished. The grain already formed in this is suspended in a liquid, the purity of which is in no way superior to that of the sugar solution drawn in. In this way there is no loss, the juices are not spoiled by being mixed with inferior sugar, and no objection can be brought against this plan, because the grain is already formed, and under ordinary conditions would increase at the expense of a syrup which is no purer than the solution drawn in. A good neutralization of this sugar solution is indispensable, as otherwise the acid molasses remaining between the crystals of the sugar might cause inversion and deterioration.

Drying the Sugar.—In many countries it is the custom to pack the raw sugars immediately after their being cured without drying them artificially. As, however, it has been proved that a dry raw sugar will keep better in storage and in transport than a moist one, drying the sugar is much more widely practised now than previously.

Formerly, the sugar was dried in the sun on a plastered floor, but, owing to the increased output of factories, this method has been generally abandoned, the more so because it was expensive, slow, and dependent on the weather. The impossibility of drying sugar on rainy days was not so serious then as now, because wet weather also interfered with the transport of canes and the drying of bagasse, so that all manufacturing operations stopped. Sugar dryers came into use as soon as railway transport and modern bagasse furnaces rendered the manufacturer quite independent of the weather and enabled him to continue crushing even during rainy weather.

Sugar Dryers.—At the present day, the sugar is dried in large iron cylinders, placed on an incline, and provided internally with scoops. The moist sugar is thrown in at the top, and the revolving motion of the cylinder causes it to fall from one scoop on to another until it reaches an outlet at the bottom, after remaining in the dryer for 7-10 minutes. During that time, a current of hot air is driven in, or drawn through the dryer, the air being heated by being first

drawn through a series of pipes built into a furnace under which bagasse is burnt. On entering the dryer, the hot air comes into contact with the falling moist sugar and deprives it of moisture.

Usually the hot air enters the dryer at a temperature of 120° C. (248° F.) and with a water content of 20 grms. per cubic metre, and leaves it at a temperature of 50° C. (122° F.) with a water content of 40 grms. per cubic metre. As air, at that temperature, is only saturated when containing 50 grms. of water per cubic metre, it is evident that this drying process is not as economical as it might be. The temperature of the dried sugar is also about 50° C., the refining crystals then contain 0·50 per cent. of moisture, and the white sugar not more than 0·1 per cent.

In some systems, the drying is effected with steam, or steam is conducted through heating tubes by means of a current of air, but those in which the sugar is dried by means of hot air are most generally used.

Dullness of Sugar dried in Revolving Dryers.—Sugar crystals retain their brilliancy when dried by means of dry steam in the centrifugals, or in the sun, though in the latter case they are somewhat bleached, but they become dull and grey after passing through a hot-air dryer, the commercial value of the sugar being thereby depreciated. This dullness is probably due to the rubbing of the sugar crystals against each other and against the metallic parts of the dryer; also to the finely powdered sugar arising from the crystals already dried and carried forward by the air current, and deposited upon the moist crystals entering at the top. In the case of white sugar, which ought to have a sparkling appearance, the drying should be effected in the centrifugal by means of dry steam.

Dried Sugar sometimes becomes Moist again.—Sometimes dried sugar absorbs moisture from the atmosphere after being passed through the dryer, and forms into a hard mass. This is observed when diseased or burnt canes have been worked up, and also when the sugar has been exposed to too high a temperature in the dryer. It may be remedied by passing the sugar through a second apparatus, similar to the dryer, through which a current of cold dry air is drawn. If sugar is bagged hot, it often sets into a hard brown mass, which, being very hygroscopic, readily attracts moisture from the atmosphere, so that a syrup liquid oozes out of the bags. When white sugar is bagged hot, it becomes dark-coloured in the centre of the bags after being stored for some time. These inconveniences are avoided by cooling in a second cylinder.

Sifting and Packing.—After being dried, the sugar is passed through copper strainers in order to remove lumps formed in the vacuum pans or centrifugals, or pieces of uncured massecuite which have dropped into the centrifugal after the sugar was cured. The finished sugar is then packed in bags, baskets, or casks.

II. Treatment of Massecuites Boiled " Smooth "

In many factories the last massecuites are not boiled to grain but are boiled " smooth " or to " string-proof."

Before reboiling the molasses a special clarification should not be omitted. The molasses themselves may contain suspended impurities, which ought to be removed before a new crystallization takes place, otherwise they may be occluded by the crystals and impart to them a dark colour. If, on the contrary, they remain suspended in the mother-liquor after crystallization has taken place, they are apt to render it viscous and difficult to separate from the crystals. Finally, molasses obtained from a badly boiled massecuite, or one which has been cured hot, may contain very minute crystals, due either to false grain which has passed through the centrifugal lining, or to after-crystallization of the hot molasses in the gutters or tanks. When the molasses are boiled smooth, the crystals settle on the coils and become charred, and also cause an irregular crystallization by their being mixed with the crystals which form during cooling. Strictly speaking, we should never omit to re-clarify all molasses before boiling them. To this end they are run into an eliminating pan, and heated by means of a steam jet ; a froth now appears on the surface which contains all the floating and suspended impurities of the molasses ; this is skimmed off. At the same time the steam, and the resulting dilution, dissolve the fine crystals which passed through the interstices of the centrifugal linings, and are suspended in the molasses.

The analyses of the froth, and of the molasses from which it is derived, are given below, both in their original state, and also calculated on dry substance so as to eliminate the influence of the dilution.

Constituents.	Molasses.		Froth.	
	On 100 Original Molasses.	On 100 Dry Substance.	On 100 Original Froth.	On 100 Dry Substance.
Sucrose	47·21	71·02	49·50	68·78
Reducing sugar	9·80	14·74	9·26	13·06
Water	33·53	—	28·03	—
Ash	1·98	2·90	3·25	4·57
Soluble part of the ash ..	1·23	1·85	1·18	1·67
Insoluble part of the ash ..	0·75	1·14	2·07	2·90
Silica	0·16	0·24	0·56	0·78
Calcium phosphate	0·08	0·12	0·38	0·53
Calcium carbonate	0·40	0·60	0·96	1·35
Magnesium carbonate ..	0·11	0·17	0·17	0·32
Wax	—	—	1·57	2·20

It follows that the froth chiefly consists of the same constituents as the molasses, but is rendered gummy by small admixtures of calcium phosphate and carbonate, wax, and organic calcium salts.

This admixture is similar in composition to the precipitate obtained during defecation, and to the various sediments encountered during the course of manufacture. It occurs in the defecation mud, on the copper gauze serving to filter the clarified juice, in the first vessels of the evaporating plant, in the sediment from the syrup, in the scale of the vacuum pan, and here again in the froth of the molasses. This shows how desirable it is to eliminate the scums after clarification, so that subsequent operations need not be hindered by having to repeat this operation at every stage of manufacture, thus causing much annoyance and delay.

It is true that such treatment does not raise the quotient of purity, and that the effect of clarification cannot be expressed in figures, but the elimination of the constituents which render the molasses viscous is a greater advantage than an increase of one or two points in purity, since such increase is generally more apparent than real.

During elimination the molasses can be neutralized with lime or with sodium carbonate, or may be bleached by means of sulphurous acid, just as in the case of syrup.

Boiling Smooth.—The diluted and eliminated molasses is drawn into the pan until all the coils are covered, concentrated, and further molasses added as the contents diminish through evaporation, being concentrated to "string-proof" as soon as the pan is full.

The concentration should be conducted to a point at which the cooled mass is sufficiently fluid to promote crystallization, yet has not become so solid as to retard it.

Great care must also be taken that the concentrated massecuite does not cool too rapidly, nor commence to crystallize before it enters the crystallizing tanks. The temperature should therefore be kept rather high during the finishing stage, not falling below 70° C. This high temperature, however, has the disadvantage that the unstable products of decomposition which have become accumulated in the seconds, attain such a degree of concentration that they may decompose spontaneously, and when once this has started it cannot be stopped, as will be shown under the heading "froth fermentation." But, although a high temperature is sometimes destructive, it is necessary in order to prevent a too rapid crystallization of the sugar.

Transport of Massecuite to Crystallizers.—When a molasses massecuite, boiled smooth, deposits crystals on its way to the crystallizing tanks, there is great risk of this sugar interfering with the regular crystallization of the massecuite and the formation of well-developed crystals. These very minute crystals

do not grow, but render the mother-liquid (in the cooled massecuite) viscous and turbid, and interfere with the curing, causing considerable loss. In some factories, the concentrated molasses flows into the crystallizing tanks through long gutters, or, still worse, through pipes—conditions which offer every opportunity for irregular crystallization and its attendant evils. For this reason, it is advisable to convey the low-grade massecuites to the crystallizing tanks with as little delay as possible, preferably in trolley-tanks which can be moved rapidly. The author has inspected many factories where the yield from massecuites was usually very low, and in such cases a rather rich molasses was sent away from the factory, because it would no longer yield any sugar. These wasteful practices disappeared as if by magic when these massecuites were no longer allowed to flow slowly through pipes to the crystallizing tanks, but were carried there rapidly in trolley-tanks.

Cooling in Motion.—At one time second massecuites, boiled smooth, were cooled in motion, sometimes after addition of sugar crystals as seed, but the results did not seem satisfactory and the practice was abandoned.

An example of a concentrated second massecuite, boiled smooth, and cooled for three days in motion is recorded here. The analyses are of the mother-liquor surrounding the crystals, and consequently the first one represents the composition of the original massecuite before crystallization had commenced.

Sample taken.	Temperature in °C.	Brix.	Polarization.	Reducing Sugar.	Quotient.	Sucrose crystallized out.
Before cooling ..	70	91·1	60·8	9·25	66·7	—
After 24 hours ..	56	86·8	46·8	11·02	53·9	25·3
After 48 hours ..	47	86·0	45·2	12·82	52·5	27·3
Separated molasses	—	85·9	42·8	13·92	49·8	30·6

Cooling at Rest.—When concentrating slowly, and at relatively low temperature, as is now done, molasses cannot be concentrated to the same degree as massecuites boiled to grain, because they would become too stiff to allow of proper crystallization.

In former times when all molasses were boiled smooth, they had to be left with sufficient water to allow crystallization to take place in the tanks and to again concentrate the mother-liquor after separating it from the sugar which crystallized out. Nowadays, only a small portion of the final massecuites is boiled smooth and all the rest is done to grain.

We give here an instance from older practice when all molasses were boiled smooth, and a total exhaustion could only be obtained after repeated concentrations and crystallizations. The figures refer to the analyses of a first, a second, and a third molasses, before and after boiling, in the same factory.

	Brix.	Sucrose.	Reducing Sugar.	Ash.	Water.	Purity.
First molasses before boiling ..	82·70	49·6	12·3	5·04	20·55	60·0
Second ,, ,, ,, ..	81·10	46·0	13·4	5·20	23·27	56·7
Third ,, ,, ,, ..	83·20	44·8	17·5	6·60	20·28	53·8
First ,, after ,, ..	89·70	53·6	12·7	5·45	16·8	59·8
Second ,, ,, ,, ..	88·40	49·6	16·0	5·30	18·3	56·1
Third ,, ,, ,, ..	90·70	48·5	17·9	6·87	14·10	53·4

In boiling molasses, not more than five or six per cent. of water is evaporated, hence the yield in sugar from molasses massecuite cannot be very considerable if, in curing the No. 1 sugar, all the crystals were separated from the molasses.

The molasses massecuites are discharged into crystallizing tanks, where they are allowed to cool down, and where the sugar that can now no longer be retained in solution finds opportunity to crystallize. The rapidity of this crystallization and the size of the crystals depend on the rapidity with which cooling goes on, and the nature of the liquid in which they are formed.

Pure sugar solutions crystallize much more quickly than impure and viscous ones, when the conditions of cooling are the same. The very impure and sticky molasses under consideration here take three weeks and more before crystallization has come to an end. Cooling and crystallization proceed more rapidly in small iron tanks than in large concrete cisterns built up from the ground.

As a rule, the tanks should be of sufficient capacity to hold the contents of one pan, and not so large as to be filled only after three or four consecutive operations, for in this case the crystallization is disturbed every time a fresh supply is run in, and the grain is irregular in form.

Danger of Excessive Concentration.—The hot massecuite is generally fluid, but becomes stiff and semi-solid during cooling, and as concentration is generally carried too far rather than not far enough, crystallization in the tanks is generally hindered. Crystals form, but grow so slowly or not at all, that

sometimes a massecuite, having a purity of 50°, consolidates into a hard mass which cannot be cured and is generally thrown away. It is, therefore, necessary to concentrate low-grade massecuites to about 94° Brix, this being the limit for a good crystallization at rest. Storing of the highly concentrated second massecuites in a hot room has been suggested for keeping the molasses fluid during crystallization, but the general construction of tropical factories would prove an obstacle in carrying out this suggestion, which has not gone beyond a few preliminary trials.

When the last massecuites are very stiff, crystallization can be promoted by covering them with water to a depth of one foot, and leaving them to stand for a month. A few planks are first laid on the top of the massecuite and the water poured upon them, in order to prevent the latter from directly mixing with the massecuite. The massecuite gradually absorbs water, becoming more fluid and allowing the sucrose to form into crystals. When crystallization is complete, the thin supernatant liquid is scooped off and the massecuite cured as usual.

Froth Fermentation.—During the cooling of low-grade massecuites, the surface sometimes becomes convex and eventually bursts, a brown froth oozing out of the crevices. This soon covers the whole surface, rising continuously and finally flowing over the top of the tank. Gases escape from this froth, emitting a very unpleasant smell, and having an acid reaction, the whole phenomenon being called "froth fermentation." With second and third molasses (in which no hard crystalline crust is formed on the surface) the whole mass starts frothing from the bottom, but in other respects the phenomena are the same as with first molasses. As this fermentation is not due to micro-organisms, all efforts to combat the phenomenon by means of disinfecting agents are fruitless. This froth fermentation does not decompose sugar, and as the massecuite is kept in motion by the disengaged gases, crystallization takes place very easily; hence such massecuites generally contain well-shaped crystals, and are very easily cured, for which reason it is customary to say that the froth changes into sugar, but this must not be understood literally. The most probable hypothesis is that the frothing is caused by the spontaneous decomposition of products formed by previous decompositions of sucrose or reducing sugar, forming carbonic, formic, and acetic acids, and caramel.

Perhaps both causes may promote froth fermentation. The author recollects a very striking instance of this phenomenon in a factory where the temperature in the carbonatation tanks had been allowed to rise too high. The first sugar and first molasses massecuites were quite normal, but suddenly two low-grade massecuites fermented violently. They entered the crystallizing tanks at a temperature of 70° C., but after a couple of days this rose to over 100° C., currents of acid gases escaping with such force that large lumps of the contents were sometimes flung up and fell back into the tank. Part of the massecuite

was charred by the intense heat; and, after fermentation, it appeared as a hard and porous mass of carbonaceous matter, almost insoluble in water, which extracted the following constituents:—

	Per cent.
Sucrose	7·69
Reducing sugar	38·4
Free acid (as acetic acid)	3·41

It has been proved that froth fermentation is also due to decomposition of products formed by overheating of sugars, and has been obviated by steaming and skimming the raw molasses, by which means the fine crystals suspended in the molasses are dissolved and can no longer become overheated by contact with the coils, and thus give rise to these troublesome products of decomposition.

A further proof that froth fermentation is due to decomposition of products of superheating is the fact that molasses are now no longer boiled repeatedly, but almost immediately removed as exhausted, and therefore there is less risk of superheating, with the result that froth fermentation has occurred much less frequently of late years.

The reason why such massecuites start frothing only while cooling down is that the unstable bodies decompose spontaneously when the concentration of the solutions reaches a certain point. As a massecuite cools and sugar crystallizes out, the dissolved impurities become more concentrated, and this promotes decomposition, which goes on steadily in the hot medium, when once started.

When this fermentation is accompanied by a considerable rise in temperature, sucrose, glucose, fructose, etc., are also decomposed and even charred, but the decomposition generally stops at the primary products of decomposition which are already present in the liquid. It is possible that such products are always present, but that some condition which induces decomposition is absent, and no fermentation is seen, but some local overheating, or excessive temperature, allows the fermentation to start. As it ceases when the massecuite cools down, it is never so violent in small iron tanks which cool rapidly, as in large ones where the heat is retained for a longer time. As already observed, no sugar is decomposed by this fermentation, but the mechanical losses may be considerable when the mass foams over the tanks on to the floor. If this is prevented by only half filling them, the available storage space is not utilized and the capacity of the plant diminished. These are sufficient reasons for trying to prevent the fermentation, by keeping defecated juices faintly alkaline to phenolphthalein paper before and during concentration. Further, all molasses should be diluted, steamed, and skimmed previous to being concentrated, and prolonged heating

at temperatures above 70° C. ought to be avoided. With these precautions, froth fermentation may be avoided.

Curing of Seconds.—When the grain is large and the crystals do not stick together, the curing is carried out in the usual centrifugals, but such massecuites are so fluid that they do not require to be prepared in a pug-mill. Formerly the centrifugals used for seconds were driven at a low velocity, but nowadays the seconds are cured in Weston centrifugals, which are started at a slow speed, and afterwards acquire their usual velocity.

Drying.—The second sugar can be washed in the centrifugals if necessary but, as a rule, this is omitted, and the moist sugar is sold, used as seed in the pan, or dissolved in the juice.

Sack Sugar, Tank Bottoms, or Black Stroop.—When a low-grade massecuite has a purity of 45-48°, the grain is generally too small to be recovered in centrifugals, so the massecuite is loaded into bags made of special palm-leaf mats. As soon as some molasses drains off, the bags are refilled with the massecuite, and this draining off and refilling is continued until the contents have assumed a semi-solid state. The bags are then sewn up and piled in heaps, by which means a further quantity of molasses oozes out of the bags. The piles are shifted a few times, and when the contents are considered sufficiently dry the sugar is sold as " sack sugar," " black stroop," or " tank bottoms."

As a rule, it contains 65 per cent. of sugar crystals, and 35 per cent. of exhausted molasses.

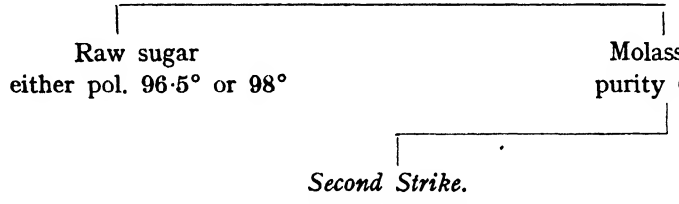
It is not advisable to return so low grade a product into the course of manufacture. It is better to sell it even at a low price than to run the risk of spoiling good juice through mixing it with so inferior a product.

III.—Schemes for Boiling and Curing

I.—SCHEME FOR TURNING OUT ONE GRADE OF RAW SUGAR, THE THIRD SUGAR BEING RETURNED INTO THE CRYSTALLIZERS CONTAINING THE FIRST AND SECOND STRIKES.

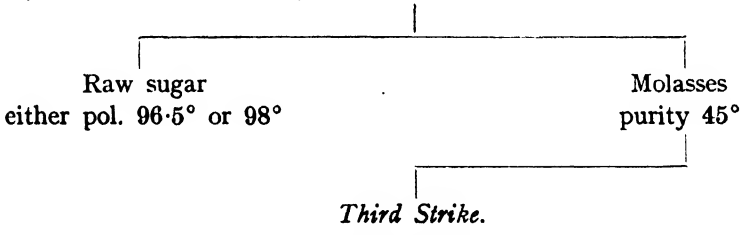
First Strike.

Using syrup, mixing if necessary the evaporator syrup with molasses of 60° purity to reach a final purity of 80–84°, giving a strike of the said purity which is boiled to 92° Brix.



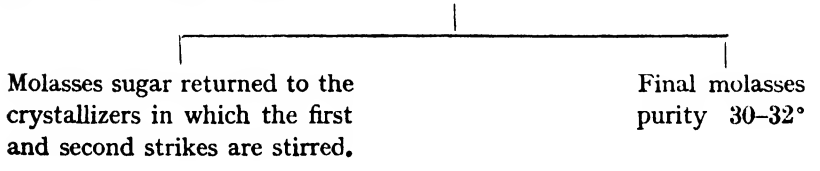
Second Strike.

Using the same kind of syrup as for the first strike and all the first molasses, giving a massecuite of 70° purity and 95° Brix.



Third Strike.

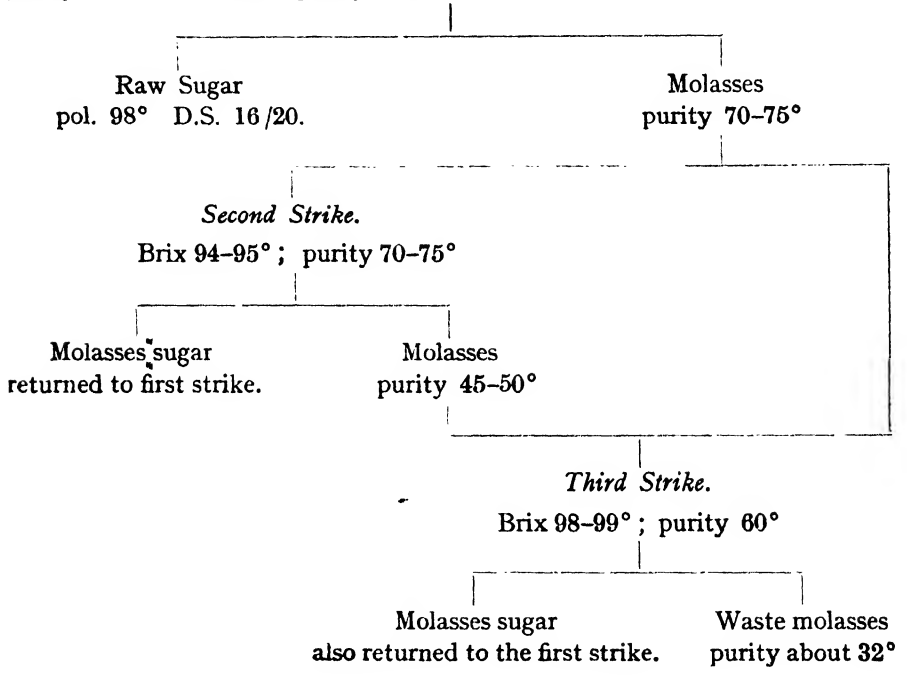
Using syrup as seed and all the second molasses, giving a massecuite of 60° purity and 98° Brix.



II.—Scheme for turning out one Grade of Raw Sugar, all Second and Third Sugars being returned to the Vacuum Pan as Seed.

First Strike.

Using evaporator syrup mixed with the second and third crop sugars, giving a strike of 85–90° purity which is boiled to 91–93° Brix.



III.—Scheme for turning out one Grade of Raw Sugar, with Partial Return of Third Sugar into the Vacuum Pan.

First Strike.

Using syrup mixed with part of the third sugar giving a strike of 85–90° purity and 91–93° Brix.

Raw sugar.

Molasses purity 70–75°, boiled to grain and mixed with so much of the second molasses as necessary to make up a strike of 76° purity.

Second Strike.

Purity 76° Brix 95°

Raw sugar.

Molasses purity 50–56°. Part of it is added to the first molasses forming a new second strike, the rest is boiled "string-proof" and cooled over a long period.

Third Strike.

Purity 50–56°

Molasses sugar to be returned to the vacuum pan as seed.

Final molasses purity 30–32°

IV.—Composition of Commercial Cane Sugars

Commercial Basis of Payment.—The bulk of cane sugars are "raws" destined for refining; they are either sold on the basis of polarization, or on the "net analysis." In the former case, the polariscope reading is stated without any correction for local temperature. The "net analysis" is found by subtracting the percentage of glucose (reducing sugar) and five times the percentage of ash from the polarization.

In many countries the duties on cane sugar are levied on a combined basis of polarization and colour.

Dutch Standard.—The Dutch Standard (D.S.) consists of a series of samples of cane sugar, ranging from a very dark No. 7 to an almost white product No. 25. They are prepared every year by two firms in Holland under the supervision of the Netherlands Trading Society (Nederlandsche Handel Maatschappij) in sealed sample bottles, which are sent to the various merchants and Customs depôts. As these grades serve as standards for the different classes of sugar on which taxes are levied, it is important that sugar merchants should know exactly to which class the sugar belongs that they want to import; and, therefore, the sets of samples comprising the Dutch Standard are daily used for comparison in many countries which export cane sugar.

Colour of Sugar an Indication of its Quality.—Although colour is not a criterion of the quality of a sugar, every number of the Dutch Standard (between 11 and 17) corresponds to a certain composition. The analyses of the high-grade sugars do not differ much from that of pure sucrose, the difference being mainly one of colour, rather than of composition. On the other hand, the differences in composition of the dark sugars may be so considerable that all relation between colour and analysis disappears. The average analyses of sugars between Nos. 11 and 17 D.S. are as follows:—

Analysis.	No. 11.	No. 12.	No. 13.	No. 14.	No. 15.	No. 16.	No. 17.
Polarization ..	96·10	96·50	97·10	97·70	98·30	98·70	99·0
Reducing Sugar ..	1·48	1·42	1·11	0·92	0·55	0·32	0·20
Ash	0·50	0·38	0·34	0·27	0·23	0·14	0·08
Moisture	0·82	0·80	0·82	0·50	0·33	0·32	0·25
Undetermined* ..	1·10	0·90	0·63	0·61	0·59	0·52	0·47
Total ..	100·00	100·00	100·00	100·00	100·00	100·00	100·00
Nett ..	92·12	93·18	93·69	94·43	96·60	97·68	98·4

* The temperature at which these analyses have been made is 28° C., or 8° C. over the temperature at which the polariscope is standardized. Therefore the polarization is about 8 × 0·003 = 0·25 lower than the real surcrose content, and consequently the figure for "undertermined" is too high by that amount.

Colour of the Crystal.—The colour of a sugar is only partly due to adhering molasses, being also due to impurities incorporated within the crystals, sucrose being one of those substances which possess this property of embodying colouring matters, etc. Von Lippmann* mentions that crystals of sucrose can incorporate logwood extract and congo red, and reports that the colouring matter in sugar candy is absorbed through the crystals. Further, crystallizations of metallic compounds with sugar are familiar, and then we know that mere traces of iron compounds and of caramel impart a dark tint to sucrose crystals formed in such solutions.

Coloration of Sugar by Iron Salts.—La Bastide† mentions a case of yellow-coloured sugar obtained from syrup which had been filtered over sand containing iron. Stolle studied the solubility of iron hydroxide in sugar solution, and stated that, notwithstanding the fact that ferric hydroxide, ferric oxide, ferrous oxide, and ferrous sulphide are very slightly soluble, as shown in the table underneath, yet these trifling quantities were more than sufficient to impart a yellow colour to the sucrose solution, with the exception of the solution containing iron oxide, which was coloured red.

The figures given here represent milligrams per litre.

Per cent. Sucrose.	Ferric hydroxide $Fe_2(OH)_6$			Ferric oxide Fe_2O_3			Ferroso-ferric oxide Fe_3O_4			Ferrous sulphide FeS		
	17·5°	45°	75°	17·5°	45°	75°	17·5°	45°	75°	17·5°	45°	75°
10	3·4	3·4	6·1	1·4	2·0	—	10·3	10·3	12·4	3·8	3·8	5·3
30	2·3	2·7	3·8	1·4	—	—	12·4	10·3	12·4	7·4	1·9	7·2
50	2·3	1·9	3·4	0·8	1·1	—	14·5	10·3	15·5	9·9	19·8	9·1

Herzfeld* repeatedly ascribed a greyish tint of the sugar to the presence of an iron compound, which remained dissolved in the juice owing to defective clarification. In bygone years, the author remembers seeing beetroot second products, that had assumed a greenish or dark brown tint, which examination proved to be due to an iron compound.

A number of first massecuites and first sugars were analysed at the commencement of a grinding season, and later on a number of similar products were again analysed after the rust in the pipes and tanks had been washed away, so that the juice dissolved less iron compounds than during the first few days.

* "Die Chemie der Zuckerarten," II., 1062.
† "Archief voor de Java Suikerindustrie," 1903, 954.
‡ "Zeitschr. Rübenzuckerind.," 1906, 338.

Class of Sugar.	No.	Mgrms. of Iron in 100 grms.		
		Sugar.	Massecuite.	Mother-liquor.
No. 25 D.S.	I.	4·6	5·5	
	II.	1·0	4·5	
No. 16 D.S.	I.	1·5	2·3	
	II.	2·3	2·3	
No. 17 D.S.	I.	7·0	2·3	
	II.	1·8	—	
Refining Crystals	I.	2·3	4·6	
	II.	1·6	4·6	
,, ,, ...	I.	1·8	4·6	
	II.	1·0	4·6	
,, ,, ...	I.	4·6	5·2	7·0
	II.	1·2	3·5	5·5
,, ,, ...	I.	2·3	4·6	
	II.	1·5	—	
,, ,, ...	I.	5·5	4·6	7·0
	II.	1·1	1·6	2·3
,, ,, ...	I.	2·0	4·6	4·0
	II.	1·5	4·6	
,, ,, ...	I.	35·0	25·5	
	II.	2·3	2·3	
,, ,, ...	I.	4·6	7·0	9·3
	II.	3·5	7·0	9·3
,, ,, ...	I.	3·5	5·5	
	II.	1·2	3·5	
		9·3	7·0	
		1·6	2·3	

The above figures represent milligrams of iron in 100 grms. of sugar, massecuites, and mother-liquor in the latter. The figures I. and II. refer to samples taken at the beginning and middle of the grinding season respectively.

Although the percentages of iron are very trifling, we see that the massecuites and sugars contain more iron at the commencement of each season than afterwards. The highest figure was 35 mgrms. per 100 grms. of sugar, and in this case the colour was deep brown and the taste very metallic. A sugar solution mixed on purpose with iron citrate or with caramel and iron saccharate, after evaporation and crystallization, yielded an almost black sugar, having a

very pronounced metallic taste, and containing not less than 100 mgrms. of iron per 100 grms.

Definition of First and Second Sugars.—Merchants often require a strict definition of first and seconds, and in former years this distinction was easily made. Firsts were obtained directly from syrup, and seconds were obtained by the concentration and curing of the molasses. At the present day, molasses boiled to grain yields a product which cannot be distinguished from a finely grained first product ; and, on the other hand, a mixture of syrup and molasses boiled smooth produces a product which closely resembles second sugar, so that a distinction is difficult, if not impossible. It has been suggested that the smell is a good characteristic of second sugar, but it is evident that this indication is much too uncertain to decide whether a given parcel of sugar is really firsts or seconds.

Polarization of Commercial Raws.—Whereas in former years a great number of assortments of raw cane sugar were encountered in trade, we find in these days chiefly two kinds of refining crystals, viz., those sold on a basis of 96° and 98° polarization respectively.

The enormous importation of raw cane sugars into the United States almost exclusively consists of the former assortment, as is clearly shown by the average polarization of the samples analysed in the New York Sugar Trade Laboratory throughout the different years of its existence.

Year.	Number of samples.	Average polarization.	Year.	Number of samples.	Average polarization.
1908	8,973	94·83	1916	22,334	95·30
1909	9,635	94·68	1917	21,562	95·71
1910	15,069	95·19	1918	16,283	95·41
1911	15,545	94·68	1919	19,211	95·55
1912	16,127	95·02	1920	19,666	95·09
1913	17,063	95·71	1921	15,147	95·56
1914	17,634	95·15	1922	22,165	95·48
1915	19,924	95·04	1923	16,921	95·98

The following table gives a distribution according to the grade of the samples.

Polarization.	Grade per cent.	Polarization.	Grade per cent.
99-100	—	91-92	0·254
98-99	0·053	90-91	0·112
97-98	5·567	89-90	0·041
96-97	52·923	88-89	0·024
95-96	31·931	87-88	0·024
94-95	6·660	86-87	0·047
93-94	1·637	85-86	0·041
92-93	0·656	84-85	0·030

The sugars polarizing between 96° and 97° comprised 52·92 per cent. of all samples, whereas during the ten years previous this percentage had varied from 33·63 to 40·31, and averaged only 38·09. The percentage of samples between 95° and 96° polarization was about normal, but that of samples between 90° and 95° showed the low record figure of 9·32. Sugars polarizing less than 90° amounted to only 0·21 per cent. of the total, while the number polarizing above 97° was about normal. It would appear from these results that, generally, the grade of raw sugars has improved, that they have been drier, and that little deteriorated sugar was received in 1923. One reason for this condition probably was the fact that about one-half of all the sugars was landed between March and June and that little sugar was stored for any length of time. Low-grade molasses sugars and mats seem to have practically disappeared.

The average polarization of the Java sugar of both chief assortments of raws has been as follows :—

Year.	Basis 98°	Basis 96·5°	Year.	Basis 98°	Basis 96·5°
1907	98·52	97·41	1916	98·47	97·39
1908	98·73	97·33	1917	98·48	97·27
1909	98·45	97·37	1918	98·59	97·57
1910	98·20	97·46	1919	98·42	97·33
1911	98·27	97·30	1920	98·41	97·29
1912	98·47	97·34	1921	98·63	97·42
1913	98·34	97·18	1922	98·48	97·26
1914	98·46	97·51	1923	98·53	97·23
1915	98·36	97·42			

Refining Quality.—Although polarization is the standard by which payment is effected, yet it fails to yield a full insight into the refining quality of the raws. It has been observed in the refineries that there exists a great difference between the filtration rates of solutions made from raws of different origin. While some sugars gave claírces (liquors) filtering with great rapidity, others were very sluggish in this respect and took a very long time to get through the filters. Generally, an uncommonly large amount of "gum" was believed to be the reason for this occurrence, while other authors believed a large amount of cush-cush or *bagacillo* accounted for the phenomenon. That indeed some raw sugars contain not inconsiderable amounts of fine bagasse is demonstrated by Hardin*, who showed that in about one hundred samples of Cuban sugars, picked at random, the average percentage of insoluble matter was 0·094 per cent., consisting of cane fibre, lime, rust, scale, and earth.

Direct analysis of the insoluble matter gave the following results :—

Insoluble.		Cane fibre.		Inorganic matter.
0·017	..	0·011	..	0·006
0·042	..	0·040	..	0·002
0·055	..	0·050	..	0·005
0·074	..	0·060	..	0·014
0·084	..	0·080	..	0·004
0·054	..	0·048	..	0·006
0·106	..	0·095	..	0·011
0·114	..	0·097	..	0·017
0·130	..	0·116	..	0·014
0·148	..	0·130	..	0·018
0·200	..	0·168	..	0·032
0·139	..	0·121	..	0·018
0·210	..	0·200	..	0·010
0·219	..	0·199	..	0·020
0·232	..	0·202	..	0·030
0·262	..	0·234	..	0·028
0·280	..	0·240	..	0·040
0·240	..	0·215	..	0·026
0·307	..	0·279	..	0·028
0·316	..	0·299	..	0·017
0·375	..	0·321	..	0·054
0·400	..	0·360	..	0·040
0·423	..	0·386	..	0·037
0·364	..	0·329	..	0·035
0·159	..	0·140	..	0·020

* "Int. Sugar Journal," 1923, 594.

Smith* showed that actually the "filtrability" depends on the content of suspended matter, and not on the presence of gums. The gums he found in a filtered solution were as plentiful as in the unfiltered liquid, and yet its filtration characteristics were decidedly changed. Neither was *bagacillo* found to be the disturbing factor, since, when in an experiment 25 times the normal amount of *bagacillo* was added and digested with lime at an alkalinity of 0·025 grams CaO per 100 c.c. for one and a half hours at a temperature of 35° C, the filtration rate of the refined sugar was actually increased more than 10 per cent. in three tests. It seemed probable that the *bagacillo* acted in a mechanical way as a filter-aid to increase the filtration rate of the refined sugar solution, so that it would not be responsible for the low filtrability of raw sugar containing fine cane fibre.

It appeared that the matter retarding filtration was nothing else than cane wax in suspension, which had come from the rind of the cane and had either not been eliminated by defecation, or been redissolved again from the mud by steaming and excessive washing and re-introduction of the sweet-waters.

Secondary Kinds of Commercial Sugar.—Besides the two assortments of raws referred to above, the cane sugar industry turns out all kinds of sugars fit for direct consumption, among which may be mentioned the superior first and soft sugars of Java, the plantation whites and yellows of Louisiana, the *vesou* sugars of Mauritius, the Demerara crystals of British Guiana, the grocery sugars of Barbados, and other islands, the *panela, dolce, gur, rab* and further denominations of that class termed "concrete sugars," etc., etc. Since, however, this work solely treats on the manufacture of raw sugar by modern methods, we shall abstain from going into details of the properties and characteristics of these other assortments, and confine ourselves to merely mentioning their names.

V.—Preservation of Sugar during Storage and Transport

As sugar must be of good quality, not only when shipped but also when it reaches the consignee, care must be taken to prevent deterioration before delivery.

Decrease in Polarization.—As regards decrease in polarization, this may be either very rapid and limited, or slow but continuous. In Java a difference in polarization has many a time been noticed between the date when the sugar left the factory and that of its arrival at port a couple of days later.

* Proceedings of the 43rd Meeting of the Hawaiian Planters' Association, 1924.

The difference sometimes amounted to about one per cent. and did not subsequently increase, but actually decreased in many cases.

The buyers insisted upon a very dry sugar, and stipulated that sugar polarizing under 96·5° was liable to heavy penalties. Each parcel of sugar was analysed before packing (as it left the dryer) in order to be quite sure that no single basket of sugar polarized below the limit of 96·5°. Notwithstanding these precautions, the samples taken from the baskets on their arrival at the shipping port repeatedly showed a lower polarization than those taken at the factory, and consequently the manufacturer sometimes had to incur the loss. These differences gave rise to complaints from the owners of the factories to their managers, and these latter on their part, complained against the sugar merchant and his clerks that they had not paid sufficient attention to the sampling, but had left this work to ignorant coolies, etc. As neither side could adduce proofs, the dispute frequently ended in suspicion, or even in dismissals.

In one instance, the polarization at the shipping port was 0·8 per cent. lower than when the same sample was tested at the factory three days previously, and the same difference occurred throughout the whole grinding season. But, on arrival in America, the sugar showed the same polarization as at the factory, proving that there had been no inversion or other decompositon of the sucrose. However, until this proof was received in Java, it was very naturally concluded that the sugar had deteriorated after leaving the factory, but whether due to inversion, absorption of moisture, or actual decomposition, it was impossible to say, because only the polarization had been determined at the two places. As all these sugars contained rather high percentages of reducing sugars it seemed not improbable that some glucose might have crystallized out along with the sucrose, giving a bi-rotation which would falsify the direct polarization. This explanation was disproved by the similarity in the ratio of ash to reducing sugars as determined in the finished sugar and in the molasses separated from it: also by obtaining the same polarization in hot and cold solutions, whereby any possible bi rotation was eliminated.

As a detailed analysis of these sugars was not made immediately after drying, their subsequent examination was not carried out, but the fact that the original polarization decreased after only three days, that such decrease did not continue after that period, but, on the contrary, disappeared entirely, coupled with the fact that these sugars contained much reducing sugar, led to the following hypothesis:—

The crystals under consideration are coated with a layer of artificially dried molasses containing much reducing sugar, and are, consequently, very hygroscopic. The sugar is packed on an estate situated in a dry district of the island, and its original polarization therefore represents well-dried sugar. This is then forwarded to the shipping port where the atmosphere is generally much more moist than at the inland factory. The sample of sugar taken from the bags at once absorbs moisture and shows a lower polarization than at the factory, while the great bulk of the sugar (protected by the bags) retains its original polariza-

tion. This sugar ultimately arrives in a country having a more temperate climate and a drier atmosphere, and when another sample is taken from the bags this does not absorb sufficient moisture to influence the polarization, which now strictly represents the bulk, and consequently agrees with the original polarization. The sugar in bulk does not change, but the sample taken for testing absorbs more moisture in a damp climate than in a dry one, and thus gives rise to the differences observed.

Effect of Inversion and Fermentation.—A much more serious deterioration during storage and transport is due to absorption of moisture and inversion or fermentation. It is evident that neither inversion nor fermentation are possible unless the sugar be moist, but whether the sugar was originally moist, or has become so by absorption of moisture from the atmosphere is a matter of indifference.

The fate of enormous quantities of raw sugar held up in the warehouses of Cuba and Java during the great war proved much more clearly than investigations made with small parcels that all deterioration started with absorption of moisture and proceeded at a steadily increasing rate.

Causes of Deterioration.—This deterioration initiated by absorption of moisture is due to inversion by fungi and yeasts. Water alone cannot invert at the temperatures which prevail in the sugar warehouses, otherwise sterilized sucrose solutions would be rapidly inverted, which they are not. Neither can fungi attack dry sugar, but when both causes co-operate, *i.e.*, when fungi or yeasts contaminate moist sugar, inversion proceeds very rapidly; the newly-formed invert sugar attracts more moisture with the result that a syrupy solution of inverted sugar oozes from the baskets and bags.

The analysis of such a syrupy liquid gave these figures:—

Polarization	9·80
Reducing Sugar	48·00
Ash	0·95
Brix	75·4

from which the following composition may be calculated:—

	Per cent.
Sucrose	25
Reducing sugar	48
Ash	1
Water	25
Undetermined	1

This shows much inversion; the sugar itself has also decreased in polarization.

Action of Micro-organisms.—The best proof that deterioration is brought about by the simultaneous presence of water and micro-organisms is furnished by an experiment in which portions of dry and of moist sugar (some of which were sterilized and others not), with pieces of the packing material, were preserved in tubes over a long period. Every month one tube out of each set was opened and the contents analysed. With a view to ascertaining whether an alkaline reaction of the sugar might prevent inversion, one sample of sterile and one of unsterile sugar were moistened with a two per cent. sodium carbonate solution.

	At the start.				After 1 month.				After 4 months.				After 6 months.				After 8 months.			
	Polarization.	Red. Sugar.	Water.	Reaction.	Polarization.	Red. Sugar.	Water.	Reaction.	Polarization.	Red. Sugar.	Water.	Reaction.	Polarization.	Red. Sugar.	Water.	Reaction.	Polarization.	Red. Sugar.	Water.	Reaction.
ilized.																				
. . . .	99·6	trace	0·09	neutr.	99·6	trace	0·05	neutr.	99·6	trace	0·16	neutr.	99·6	trace	0·06	neutr.	99·5	trace	0·10	neutr
1% water . .	98·8	,,	0·95	,,	98·7	0·10	0·90	,,	99·2	0·32	0·44	,,	98·6	0·48	0·57	,,	98·7	0·70	0·48	acid
2% ,, . .	97·9	,,	1·90	,,	97·6	0·10	1·80	,,	97·1	0·96	0·94	,,	96·5	1·28	0·90	,,	96·0	1·44	0·95	,,
2% soda . .	98·1	,,	1·75	alk.	98·0	0·15	1·63	alk.	97·1	0·53	1·90	alk.	Glass broken.							
sterilized.																				
. . . .	99·6	,,	0·09	neutr.	99·6	trace	0·18	neutr.	99·2	0·28	0·16	neutr.	98·5	0·71	0·30	acid.	97·7	1·40	0·60	,,
% water . .	98·7	,,	1·01	,,	97·5	0·92	0·58	,,	96·8	1·07	1·15	,,	96·1	1·92	0·82	,,	95·8	1·90	0·91	,,
2% ,, . .	98·0	,,	1·83	,,	95·5	1·72	2·10	,,	93·7	2·32	2·20	,,	93·8	3·19	2·25	,,	90·5	3·70	1·85	,,
2% soda . .	97·7	,,	2·11	alk.	97·1	0·53	2·24	alk.	94·0	2·13	2·03	,,	92·3	3·33	2·02	neutr.	90·7	3·88	2·11	neutr.

This experiment proves that dry sugar, even when contaminated with germs, remains unchanged, but unsterilized sugar in a moist condition is at once attacked and partly inverted by the germs, though the original reaction be neutral. Sugar in which the micro-organisms have been killed as far as possible (sugar cannot be completely sterilized at high temperature without becoming decomposed), also deteriorates, but by no means so quickly as when unsterilized. Alkalinity of the sugar is not a sufficient protection against inversion. Even when the reaction is still strongly alkaline (0·2 per cent. of sodium carbonate) inversion proceeds slowly, but as soon as this is neutralized (either by acids formed through the action of the fungi or from the glucose), there is no difference between neutral and alkaline sugars in this respect. Kamerling* showed that sugar coming out of the dryer is practically sterile, and that the source of infection is to be sought in the manipulations during packing and in the packing material itself. Indeed, numerous germs cling to the bags in which the sugar is packed, including those organisms which feed on sugar and invert it.

This latter fact has been observed as far back as 1874 by Serrurier, who ascribed the formation of a moist layer of inverted sugar against the sides of the

* "Archief voor de Java Suikerindustrie," 1903, 122.

baskets arriving in Amsterdam, to the influence of the mats in which the sugar was packed. This portion of the sugar contained 7 to 10 per cent. of reducing sugar, while that in the centre contained not more than one per cent. The fact that the mixed sugar contained only 1½ per cent. of reducing sugar proves that the amount of damaged sugar was inconsiderable and that the inversion had, therefore, confined itself to the outer layers.

Micro-organisms which cause Deterioration.—Among the micro-organisms which cause deterioration, Maxwell* mentions the organisms of the butyric and lactic acid fermentations, while Storey ascribes it to fungi, especially the *Penicillium glaucum*. Kamerling† found in dried sugar chiefly a fungus flora related to *Penicillium* (over 20 varieties were observed, and not less than 19 belonged to the group *Sterigmatocytes*, *Aspergillus*, *Penicillium* and *Citromyces*). After the sugar has become very moist, the yeasts predominate, and among these are *Saccharomyces*, *Torula*, *Monilia*, &c., which invert sugar strongly, so that during the course of deterioration of raw sugar, the first attack is made by fungi, and after inversion and absorption of moisture have commenced, they are superseded by yeasts which play the principal part in the deterioration.

Greig-Smith and Steel‡ ascribed it to a bacillus which they called *Bacillus levaniformans*, and Noël Deerr and Norris‖ report having found five different species of bacteria, none of which could positively be identified with the *Bacillus levaniformans*.

Schöne§ emphasized the importance of moulds and torulæ in inversion and noted the presence of *Penicillia*, especially *P. glaucum* and *Mucor*. He found that considerable acidity was developed in sugar solutions inoculated with pure cultures of some of these organisms. In another connection he notes the isolation of *Penicillium* and *Rhizopus*.

Scott¶ observed the fungi on the surface of sugar solutions, noting *Penicillia* and *Aspergilli*. He found that sugars were attacked by these organisms in the following descending order: Low Brazilian, Peruvian, Jamaican and Javanese. The inverting power was noted and precautions advised against infection.

Amons†† isolated by plating on synthetic agar four *Aspergilli*, two *Penicillia* and a *Rhizopus*. Mycelial threads observed in moist sugars were identified as indicating the presence of *P. glaucum* in an active form. The deterioration of sugar by this fungus was demonstrated by inoculating sugar (sterilized with absolute alcohol) with spores and comparing the absorption of moisture (from a saturated sterile sugar solution under a bell jar) with a sterile check. It was found that the sterile sugar absorbed no moisture after thirty-three days, while the inoculated sugars absorbed a considerable amount of water. This

* "Louisiana Planter," 1896, 154.
† "Archief voor de Java Suikerindustrie," 1899, 629.
‡ "Int. Sugar Journal," 1903, 448.
‖ "Int. Sugar Journal," 1908, 333.
§ "Deutsche Zuckerindustrie," 1906, 1338; 1908, 638; 1911, 247.
¶ "Int. Sugar Journal," 1912, 582.
†† "Archief voor de Java Suikerindustrie," 1916, 1911; 1917, 1225.

is interpreted as signifying an appreciable fungus growth which inverted the sucrose, the hygroscopic reducing sugars being responsible for this absorption of moisture.

In his mycological observations Browne* isolated and studied the inverting power of *Torulæ* and two *Monilia* (named " nigra " and " fusca " respectively). The presence of fungi such as *Penicillia* was noted and their possible importance emphasized. Some interesting points are made with regard to sources of infection and consequent prevention of deterioration of raw cane sugars.

Owen† after an exhaustive survey of the activities of bacteria and torulæ investigated the rôle played by moulds in sugar deterioration. From a large variety of Louisiana sugars he isolated a number of *Aspergilli* and studied their activity in pure culture in sugar solutions of high concentration.

In an interesting group of experiments concerned with the conditions limiting the respective activities of the three groups of micro-organisms in sugars, Owen found that a fungus inoculation caused marked deterioration in a 69° Brix solution, which proved to be of too great a density for the activity of either bacteria or torulæ, the latter two groups inverting sucrose at 52° and 64° Brix respectively as the upper limits. He regards the fungi as constituting the most dangerous group of micro-organisms in sugar because of their strong inverting power, their ability to exercise this power in highly concentrated solutions of sucrose of varying reaction, and also on account of their ability to develop on media which are very deficient in nutrients.

Nicholas and Lillian Kopeloff‡ isolated in a variety of media fungi from a wide range of cane sugars, belonging chiefly to the *Aspergilli* and *Penicillia*. *Aspergillus niger* and a *Blue Aspergillus* occurred in practically all samples. A microscopic examination by these authors revealed fungus mycelium in some sugars. Sterilized sugars inoculated with pure cultures of fungi deteriorated rapidly when the moisture content was appreciable. Little, if any, deterioration occurred when the moisture content was reduced to a minimum. The fungus which appeared with greatest frequency in all sugars, the *Blue Aspergillus*, also had the greatest deteriorative power.

Although different investigators ascribe deterioration to different albeit kindred organisms, they all agree that some organism or other is the direct cause, but that the sugar must be moist to allow of their growth and activity. A dry sugar can be kept unaltered for a long time, while a moist one soon becomes inverted and falls off in quality, owing to the activity of micro-organisms.

Preventing Deterioration.—There are therefore two methods of preventing deterioration during storage or transport. First, by eliminating the micro-organisms by sterilizing the packing material, and, second, by drying the sugar

* " Louisiana Planter," 1915, 281; " Int. Sugar Journal," 1915, 331.
† " Louisiana Bulletin," Nos. 125, 146, 153 and 162. ‡ *Ibid.*, No. 166.

and taking care that it does not re-absorb moisture, so that even if the spores or germs are present they cannot develop, and thus remain inoffensive.

Disinfected Packing Material.—Kamerling* found that disinfection of the packing material by means of a hot one per cent. solution of carbolic acid is sufficient to kill the germs of the destructive organisms, and suggested treating all the packing material with this disinfectant before bringing it into contact with the sugar.

Wishing to test this point, the author requested the Director of the Netherlands Indies Agricultural Society (Nederlandsch Indische Landbouw Maatschappij) to induce the managers of all the factories belonging to that Company to pack the same kind of sugar in both disinfected and non-disinfected material, to allow one package of every kind to stand over for one year and

BASKETS.

Disinfected.								Not Disinfected.							
No.	Original Polarization.	Original Polarization on 100 Dry Substance.	Polarization at the end on 100 Dry Substance.	Difference.	Reducing Sugar original on 100 Dry Substance.	Reducing Sugar at the end on 100 Dry Substance.	Difference.	No.	Original Polarization.	Original Polarization on 100 Dry Substance.	Polarization at the end on 100 Dry Substance.	Difference.	Reducing Sugar original on 100 Dry Substance.	Reducing Sugar at the end on 100 Dry Substance.	Difference.
I.	97·45	97·89	98·14	-0·25	1·20	1·21	0·01	I.	97·10	97·87	98·01	0·14	1·33	1·38	0·05
II.	96·50	97·38	97·34	0·04	1·54	1·15	-0·39	II.	96·50	97·41	96·83	0·58	1·54	1·61	0·06
III.	97·60	98·07	97·60	0·47	1·10	1·54	0·53	III.	97·60	98·12	96·96	1·16	1·10	2·04	0·94
IV.	96·70	97·80	96·94	0·86	1·44	2·07	0·63	IV.	96·80	97·86	94·43	3·43	1·39	3·47	2·08
V.	97·50	97·95	97·19	0·76	1·24	1·76	0·52	V.	97·10	97·76	94·71	3·05	1·29	3·73	2·44
VI.	97·00	97·45	97·31	0·14	1·49	1·62	0·13	VI.	97·10	97·53	97·55	0·02	1·44	1·55	0·11
VII.	96·30	97·34	95·13	2·21	1·54	2·99	1·45	VII.	97·40	98·11	96·54	1·57	1·20	2·21	1·01
VIII.	97·40	98·20	97·53	0·67	1·00	1·43	0·43	VIII.	97·40	98·20	96·82	1·38	0·97	2·04	1·07
IX.	97·40	97·97	94·88	3·09	0·91	3·24	2·33	IX.	97·40	97·97	93·95	4·02	0·91	3·89	2·98
X.	96·70	97·00	96·41	0·59	1·40	2·02	0·62	X.	96·80	97·15	96·74	0·41	1·28	1·85	0·57
XI.	97·50	98·17	96·91	1·26	0·99	2·04	1·05	XI.	97·50	98·19	96·43	1·76	0·99	2·41	1·52
XII.	96·50	97·32	96·33	0·99	1·29	2·27	0·98	XII.	96·80	97·61	94·63	2·98	1·21	3·22	2·01
XIII.	97·70	98·11	97·54	0·57	1·24	1·47	0·23	XIII.	97·60	98·02	97·03	0·99	1·24	1·94	0·70
XIV.	—	—	—	—	—	-	—	XIV.	97·50	98·19	97·25	0·93	1·04	1·68	0·64
XV.	97·00	97·74	96·25	2·49	1·24	1·69	0·45	XV.	97·20	97·85	94·21	3·64	1·24	3·40	1·16
XVI.	97·20	97·69	97·72	-0·03	1·18	1·47	0·25	XVI.	97·30	97·75	97·08	0·67	1·13	1·85	0·72
XVII.	97·50	97·89	95·74	2·15	1·13	2·72	1·59	XVII.	97·40	97·87	97·18	0·69	1·12	1·63	0·51
XVIII.	97·10	97·69	97·35	0·34	1·33	1·54	0·21	XVIII.	97·20	97·58	95·93	1·55	1·38	2·41	1·03
XIX.	97·10	97·71	97·45	0·26	1·21	1·43	0·21	XIX.	97·10	97·91	95·99	1·72	1·21	2·22	1·01
Average	97·14	97·80	96·88	0·92	1·25	1·87	0·62	Average	97·20	97·82	96·22	1·60	1·26	2·34	1·08

BAGS.

XVI.	97·30	97·94	96·88	1·06	1·10	1·94	1·84	XVI.	97·20	97·78	96·27	1·51	1·12	2·04	0·92
XVI.	98·70	98·90	97·54	1·36	0·62	1·54	1·92	XVI.	98·70	98·90	96·44	2·46	0·58	2·27	1·69
XVII.	—	—	—	—	—	—	—	XVII.	99·30	99·50	95·07	4·43	0·30	3·35	3·05
XIX.	—	—	—	—	—	—	—	XIX.	97·20	97·79	95·98	1·81	1·17	2·60	1·43
Average	98·00	98·42	97·21	1·21	0·86	1·74	0·88	Average	98·10	98·50	95·94	2·55	0·79	2·56	1·77

* "Handelingen voor het 4de Congres te Semarang," 1900, 104.

to send a sample of the mixed contents of every package to the Experimental Station. The mats, baskets, and bags were dipped in a hot one per cent. carbolic acid solution and dried, after which they were used for packing the sugar.

During the period of storing, the decrease in polarization was caused by absorption of moisture quite as much as by inversion, consequently the first-mentioned factor has been eliminated in the above tables by calculating the polarization and reducing sugar on 100 parts of dry substance, leaving the difference due solely to the action of the inversion. We notice that the sugar in the disinfected packing material was also partially inverted, but to a less extent than in the unsterilized bags, and that this was the case with bags as well as with baskets.

Impermeable Packing Material.—As it appeared that disinfection was not sufficient to protect the sugar against micro-organisms, and since the bags and baskets usually employed did not prevent absorption of moisture, the following experiments were made by packing the sugar in an impermeable material.

Sugar, which had already attracted some moisture from the atmosphere, was packed in bags lined with waxed paper, and placed in a warehouse together with the ordinary bags filled with the same sugar, which latter served as a check. The analyses at the commencement, and after some time, were as shown on page 282.

A second experiment was carried out with bags lined with paper rendered impermeable by immersion in a rubber solution. The bags were filled on August 12th, and analysed on the 12th of October, December, and February, with these results:—

	August 12th.	October 12th.	December 12th.	February 12th.
Polarization ..	97·80	97·10	96·10	95·0
Reducing Sugar ..	0·82	0·98	1·65	2·05
Ash	0·31	0·30	0·31	0·30
Water	0·52	0·78	1·18	1·34
Undetermined ..	0·55	0·84	0·76	1·31
Nett	95·43	94·62	92·90	91·45

The lined bags retained the syrupy liquid which gradually collected between the sugar crystals, and the bags were externally clean, whereas the ordinary bags became damp and sticky from the sweating of the contents, but

Analyses.	Single Bag.	Bags Lined with Thin Waxed Paper.	Bags Lined with Thick Waxed Paper.
At the beginning.			
Polarization	96·70	96·70	96·70
Reducing sugar	1·48	1·48	1·48
Ash	0·45	0·45	0·45
Water	0·73	0·73	0·73
Undetermined	0·64	0·64	0·64
Total	100·00	100·00	100·00
Nett	92·97	92·97	92·97
After 2½ months.			
Polarization	95·00	95·00	95·10
Reducing sugar	1·48	1·48	1·45
Ash	0·45	0·45	0·45
Water	2·12	2·14	2·12
Undetermined	0·95	0·93	0·88
Total	100·00	100·00	100·00
Nett	91·27	91·27	91·30
After 4½ months.			
Polarization	88·60	86·70	90·10
Reducing Sugar	2·84	2·84	2·32
Ash	0·48	0·47	0·48
Water	3·91	5·17	3·14
Undetermined	4·17	4·82	3·96
Total	100·00	100·00	100·00
Nett	83·36	81·51	85·68

did not hinder the free passage of gases and vapours. The sugar attracted moisture from the atmosphere both in the lined and unlined bags and afterwards became inverted in the same manner, to the same degree, and in the same period of time.

Keeping Sugar Dry.—It being impossible to modify the packing material so that the absorption of moisture and development of micro-organisms may be prevented, the only alternative is to deliver the sugar dry and to store it in dry, well-constructed and ventilated stores. If this is feasible, the keeping power of the sugar is guaranteed, but if not, nothing will prevent it from absorbing moisture and deteriorating in the end.

Sugar crystals are not hygroscopic, but the surrounding film of molasses is so, especially if dried artificially. In order to determine the hygroscopic power at various temperatures and moisture conditions of the atmosphere, quantities of 100 grms. of different qualities of sugar were spread out in porcelain dishes and placed in a room the temperature and humidity of which could be regulated. The dishes were weighed several times each day and the temperature and humidity of the air recorded.

ORIGIN AND ANALYSIS OF THE SUGARS.

No.	Description of the Sugars.	Polarization.	Reducing Sugars.	Ash.	Water.
1	Refined sugar from Europe	99·7	—	—	0·05
2	Sugar over No. 25 D.S. from a carbonatation factory	99·6	—	—	0·05
3	,, ,, defecation	99·65	—	—	0·10
4	Refining Crystals, No. 14 D.S.	98·1	0·37	0·17	0·62
5	,, No. 11 ,, ...	98·2	0·48	0·18	0·40
6	Refining crystals from a massecuite of 80 quotient	97·8	0·82	0·31	0·52
7	,, ,, ,, 72 ,,	97·5	0·87	0·20	0·50
8	,, ,, mixed massecuite ..	97·5	1·34	0·20	0·56
9	Molasses sugar from a massecuite of 62 quotient ..	85·3	4·46	1·27	2·66
10	The same but very finely grained	84·8	1·95	1·67	3·70
11	First sugar (carbonatation), No. 18 D.S.	99·1	0·37	0·13	0·14
12	,, ,, No. 16 D.S.	98·8	0·62	0·14	0·32
13	,, (defecation) No. 20 D.S.	98·9	0·55	0·14	0·26
14	,, ,, No. 18 D.S.	98·4	0·92	0·16	0·38
15	,, ,, No. 16 D.S.	98·1	0·71	0·14	0·56
16	Second sugar (carbonatation) No. 20 D.S.	98·2	0·54	0·16	0·68
17	,, (defecation) No. 18 D.S.	95·2	1·20	0·16	2·88
18	,, ,, No. 10 D.S.	91·5	3·50	0·91	2·44

The following tables contain the readings of dry and wet bulb thermometers with the corresponding values for absolute and relative humidity of the atmosphere, and the loss or increase in weight of the sugars, taking their original weights as 100. A decrease is shown by a minus sign.

Date.	Hour.	Thermometer.		Humidity.		1.	2.	3.	4.	5.	6.	7.	8.	9.	10.
		Dry Bulb.	Wet Bulb.	Absolute.	Relative.										
22nd July	10·5	26·2	20·4	18·0	72	0	0	0	0	0	0	0	0	0	0
,, ,,	3	26·0	23·0	19·0	76	0	0	0	0	0·4	0·7	0·7	0·5	0·3	0·7
,, ,,	5	25·8	23·0	19·2	78	0	0·1	0	0	0·4	0·7	0·7	0·2	0·3	0·7
23rd ,,	7·5	24·0	21·8	18·0	81	0·3	0·3	0·2	0·3	1·2	2·2	2·0	0·8	1·7	2·7
,, ,,	12	26·6	24·4	21·4	82	0·2	0·2	0·2	0·3	0·6	1·7	1·8	0·5	1·5	2·2
,, ,,	3	25·8	28·4	19·9	81	0·3	0·2	0·1	0·3	0·6	1·7	1·7	1·5	1·5	2·2
24th ,,	7·5	24·8	28·0	19·8	85	0·3	0·2	0·2	0·5	1·4	2·7	3·0	2·2	3·4	4·2
,, ,,	12	27·2	28·8	19·8	73	0	0·1	0·2	0·2	1·2	2·5	3·0	1·5	3·4	4·2
,, ,,	3	26·6	24·2	21·0	83	0·2	0·5	0·3	0·2	1·2	2·7	3·0	1·7	3·8	4·2
,, ,,	5	28·0	24·6	20·9	74	0·2	0·2	−0·1	0·2	0·9	2·7	2·8	0·7	3·5	4·2
25th ,,	7·5	26·0	23·4	19·8	79	0·2	0·2	0	0·3	1·0	2·2	2·7	0·5	3·5	4·4
,, ,,	10·5	27·7	23·6	19·1	69	0·1	0	0	0·2	0·8	1·5	2·5	0	3·3	4·1
,, ,,	12·5	31·1	25·4	20·6	61	0	0	−0·1	0	0·6	1·2	2·5	0	2·8	3·7
,, ,,	3	32·0	26·0	21·3	60	0	0	−0·1	0	0·4	0·7	2·0	0	2·3	3·0
,, ,,	5	30·6	24·8	19·7	61	0	0	−0·1	0	0	0·7	1·2	0	1·3	2·2
26th ,,	7·5	28·0	24·4	20·5	72	0	0	−0·1	0	0	0·5	1·3	0	1·1	1·8
,, ,,	12	30·1	25·0	20·4	64	0	0	−0·1	−0·1	0	0·3	1·0	0	1·0	1·6
,, ,,	3	31·2	25·4	20·7	61	0	0	−0·1	−0·2	−0·1	0·2	0·7	−0·1	0·7	1·2

Date	Hour.	Thermometer.		Humidity.		11	12	13	14	15	16	17	18
		Dry Bulb.	Wet Bulb.	Absolute.	Relative.								
26th August	8	28·4	24·8	21·0	73	0	0	0	0	0	0	0	0
,, ,,	12	30·2	25·2	20·7	65	0	0	0	−0·2	−0·2	−0·3	−0·3	−0·2
,, ,,	3	30·7	25·4	20·8	63	−0·2	−0·2	−0·2	−0·3	−0·3	−0·7	−0·5	−0·3
,, ,,	5	30·6	25·4	20·7	63	−0·2	−0·2	−0·1	−0·4	−0·4	−1·0	−1·2	−0·5
27th ,,	7·5	27·2	25·0	20·2	83	0	0·3	0·3	0·2	0·2	−0·5	−1·2	1·3
,, ,,	3	30·1	25·6	21·6	68	−0·1	0·3	0·3	−0·1	−0·2	−0·7	−2·5	1·5
,, ,,	5	29·0	24·6	20·3	68	−0·2	0·2	0·2	−0·2	−0·2	−1·0	−2·8	1·1
28th ,,	7·5	26·6	23·8	20·2	78	0	0·7	0·3	0	0	−0·5	−2·0	3·1
,, ,,	3	30·6	25·4	20·9	64	−0·2	0·3	0·1	−0·3	−0·3	−1·0	−2·3	2·1
,, ,,	7·5	27·2	24·4	21·0	78	0·5	0·3	0·3	0	0	−0·8	−2·5	2·1
29th ,,	12	27·2	24·8	21·8	81	0·3	0·4	0·3	0	0	−1·0	−2·4	3·8
,, ,,	5	27·4	25·0	20·1	81	0·3	0·5	0·3	0	0	−1·0	−2·4	3·8
30th ,,	7·5	25·8	24·0	21·0	85	0·4	0·8	1·0	0·5	0·5	−0·2	−1·2	6·3
,, ,,	12	27·0	23·4	19·6	74	0·3	0·6	0·7	0·2	0·2	−0·6	−1·6	6·0
31st ,,	7·5	26·2	24·6	22·0	87	0·5	0·8	1·0	0·3	0·2	−0·5	−1·3	7·0

When the experiment commenced, the sugars had already absorbed the full amount of moisture corresponding with the humidity and temperature of the atmosphere at that time.

The experiments indicate that pure sugar is not hygroscopic and contains very little moisture, whereas sugar crystals coated with molasses can absorb a considerable amount of moisture and thereby favour the development of

micro-organisms. The figures for decrease or increase in moisture content indicate that it is the relative rather than the absolute humidity of the air which influences the absorption of moisture.

It should be remembered that the absolute humidity is the tension of the water vapour in the air expressed in mm. of mercury, while the relative humidity is the relation between that figure and the tension of the water vapour at that same temperature when the air is fully saturated with water. This latter is taken as a base, with the figure 100.

It is, therefore, not the tension of the aqueous vapour which determines the absorption or evaporation of water but the degree of saturation of the atmosphere with water vapour, whereby a given sugar absorbs water and then gives it off again at different temperatures but with the same tension.

The tables show that at temperatures between 24° and 33° C., refined sugar and very high-grade raw sugars absorb moisture at a relative humidity of 80° and over, and give it off under that figure. The limit may be put down at 75° for white sugars, at 70° for refining crystals and molasses sugar, and between 70° and 75° for seconds, according to their quality. It made no difference whether the refining crystals were dark coloured or light, finely or coarsely grained, made from pure syrup or from mixed syrup massecuite and returned molasses. The only active condition is the percentage of adhering molasses.

Owen* investigated the degree of moisture absorption of white plantation sugar under conditions prevailing in the climate of Louisiana.

		Over Water.					Over 25° Brix Sulphuric Acid.				
Sample.	Sucrose.	24 hr.	48 hr.	72 hr.	96 hr.	Total.	24 hr.	48 hr.	72 hr.	96 hr.	Total.
1	99·55	0·74	1·90	—	1·50	1·38	1·11	1·50	1·09	1·44	1·24
2	99·17	0·98	1·47	—	1·08	1·14	1·003	1·23	0·80	0·97	0·95
3	99·2	1·37	1·49	—	0·82	1·11	0·97	1·06	1·14	1·10	1·07
4	99·5	1·57	1·36	1·56	—	1·51	1·03	0·58	0·54	1·30	0·88
5	99·42	1·41	1·04	—	—	1·36	4·20	2·28	0·57	1·40	1·72
6	98·62	2·77	1·81	—	1·48	2·00	3·60	0·86	0·69	3·10	1·50
7	99·35	2·78	0·73	—	0·78	1·13	2·50	1·76	1·10	1·50	1·50
8	99·53	2·99	1·03	—	1·07	1·50	2·08	1·44	1·85	1·38	1·75
9	99·21	1·73	0·66	—	1·03	0·99	0·94	0·85	0·82	0·85	0·87
10	83·9	2·63	1·55	1·23	2·01	1·82	2·95	2·80	2·10	1·89	2·45
11	99·55	1·60	1·80	0·57	1·38	1·30	0·92	0·88	0·80	0·58	0·80
12	98·67	1·34	1·27	1·21	1·10	1·24	2·72	1·53	1·44	1·46	1·69
13	99·55	1·06	0·90	1·10	0·85	0·97	1·62	1·50	1·41	1·23	1·43
14	99·82	0·82	0·84	0·80	0·74	0·80	—	—	—	—	—
15	99·9	0·55	0·51	0·56	0·84	0·61	0·62	0·76	1·27	1·40	1·01
Average	..	1·62	1·22	1·004	1·13	1·257	1·87	1·21	1·115	1·4	1·34

* "Louisiana Planter," 1923, LXX., 469.

Weighed portions of a great number of representative samples were exposed on watch-glasses to water-vapour in a desiccator, placed in an incubator having a temperature of 30° C. In one series the desiccator contained water, and in a second sulphuric acid of 25° Brix or 15 per cent.

In the first case the relative humidity was 100 and in the second 93°

The results are shown in the table on page 285, expressed in terms of the ratio of per cent. moisture increase of the sample examined, to that of a sample of refiners' granulated, the latter being taken as unity.

In the following table the results of the experiments are given in actual percentages of moisture absorbed and the results averaged.

Sample.	Over Water.					Over 25° Brix Sulphuric Acid.				
	24 hr.	48 hr.	72 hr.	96 hr.	Total.	24 hr.	48 hr.	72 hr.	96 hr.	Total.
Refined ..	2·78	2·28	—	5·64	10·71	2·84	1·23	3·25	2·50	9·82
1 ..	2·08	4·33	—	8·47	14·78	3·18	1·89	3·54	3·62	12·23
2 ..	2·75	3·37	—	6·14	12·26	2·85	1·52	2·61	2·43	9·41
3 ..	3·83	3·40	—	4·66	11·09	2·95	0·72	1·78	3·27	8·72
Refined ..	3·29	2·89	5·00	—	11·18	1·70	3·45	4·86	1·82	11·83
4 ..	5·22	3·95	7·8	—	16·97	—	—	—	—	—
5 ..	4·65	3·02	—	7·60	15·27	7·15	7·89	2·77	2·56	20·37
6 ..	9·13	5·48	—	8·37	22·98	6·17	3·00	3·37	5·76	18·30
Refined ..	2·09	5·09	—	4·06	11·24	4·46	6·09	5·38	2·85	18·78
7 ..	5·81	3·74	—	3·20	12·75	1·51	1·18	1·35	0·75	4·79
8 ..	6·25	6·29	—	4·37	16·91	3·15	1·70	2·50	1·04	8·39
9 ..	3·63	3·39	—	4·20	11·22	1·42	1·01	1·12	0·64	4·19
Refined ..	4·29	4·70	5·11	4·12	18·22	2·28	1·39	1·76	1·83	7·26
10 ..	11·32	7·32	6·32	8·32	33·28	6·73	3·94	3·70	3·46	17·83
11 ..	6·86	8·49	2·92	5·69	23·96	2·10	1·24	1·42	1·07	5·83
12 ..	5·78	6·00	6·22	4·62	22·62	1·42	1·07	2·25	2·61	7·35
Refined ..	6·24	7·08	4·01	5·86	23·19	2·00	2·10	4·46	2·95	11·51
13 ..	6·67	6·40	4·55	5·00	22·62	3·25	3·28	6·33	3·65	16·51
14 ..	5·13	5·97	3·21	4·39	18·70	1·47	1·26	1·53	0·88	5·14
15 ..	3·46	3·67	2·28	4·96	14·37	5·44	3·23	6·46	4·34	19·47
Average ..	4·8	4·98	4·75	5·60		3·5	2·5	3·19	2·72	
Refined ..	3·64	4·40	4·70	4·99		2·01	1·87	3·13	1·97	

In studying the tables we find that the average ratio of moisture absorption of all the samples of plantation sugars to that of the refined was 1 : 1·62 over water and 1 : 1·87 over the sulphuric acid solution.

In both the series, the highest ratio was during the first, and the least during the third period. We find that in both series approximately one-third of the samples proved less hygroscopic than the standard granulated sample.

It is evident that the moisture absorption of plantation granulated sugars is determined less by the minute quantities of non-sucrose solids present than by the size of grain and more particularly by the uniformity of grain and

presence of broken grain and sugar dust. We thus find that both raw and white sugar will absorb moisture from the atmosphere as soon as they find an opportunity to do so and that the packing material is unable to prevent this. The opportunity arises as soon as the sugar comes in contact with air containing over 70-75 relative humidity at temperatures prevailing in the tropics.

Factor of Safety.—The Colonial Sugar Refining Company of Australia has established a factor, which they called the "factor of safety," which shows when there is danger of the deterioration of raw sugar during storage. The factor is based on the hypothesis that there is no such danger so long as the moisture content remains under half the amount of the non-sucrose. This may be expressed by stating that when $\frac{\text{moisture}}{100 - \text{pol.}} < 0{\cdot}333$, the sugar will not deteriorate.

For the climate of Australia the critical moisture content for sugar of various polarizations would be :—

Polarization.	Critical moisture content.	Polarization.	Critical moisture content.
99·9	0·032	97·0	0·98
99·8	0·065	96·5	1·04
99·5	0·155	96·0	1·22
99·3	0·221	95·5	1·38
99·0	0·322	95·0	1·55
98·5	0·488	94·5	1·62
98·0	0·655	94·0	1·88
97·5	0·821	88·0	3·88

Browne* showed that for Cuban and Porto Rican sugars the value 0·333 is too high and puts it at 0·3. He found that sugars with a factor lying between 0·313 and 0·346 deteriorate while others with a factor between 0·253 and 0·289 do not. In other climates the critical point is perhaps otherwise, and our observations made during a series of years in the hot and damp climate of Java showed us that temperature and humidity of the air in the warehouses in that island are such that sugar may absorb moisture even in well-ventilated rooms.

If such a period does not last too long and is followed by a dry spell in which the surplus may evaporate again, no great harm will be done, but if for some reason or other sugar is being stored over a long period in a moist atmosphere, the sugar in the bags at the outside of the piles becomes so moist

* "Journal Soc. Chem. Ind.," 1918, 103.

that the factor of safety is exceeded, after which the ubiquitous fungi and yeasts start their work of destruction. The sugar becomes partly inverted, and is then much more hygroscopic than before, while a thick liquid containing in solution sucrose and inverted sugar is soon observed trickling down the sides of the piles.

In order to guarantee as far as possible the keeping quality of stored sugars, we should fulfil the following requirements:

Clarification of the juice should be conducted in such a manner that only very little cane fibre or bagacillo can be detected in the raw sugar.

During clarification, the cane juice should never be exposed for more than a very short while to alkaline reaction at elevated temperature.

The sugar crystals should be uniform and of a reasonably large size in order to prevent adhering molasses remaining in the product.

The sugar should be bagged in a dry and cold state.

Langguth Steuerwald* enumerates the points to be observed in the construction of storehouses in which sugar has to be kept during a lengthy period, as the preservation of the product greatly depends on the more or less adequate design of the rooms in which it is being kept. If these are built in such a way that moisture cannot penetrate, either by the floor, the roof, or the walls, the excessive moisture from the atmosphere is very rapidly absorbed by the sugar, which establishes a kind of equilibrium. The composition of the sugar and the humidity of the air in the " go-down " remain practically unchanged, save for slight oscillations as a consequence of changes in temperature.

Any amount of water-vapour, however, penetrating into the warehouse immediately disturbs the equilibrium, so that practically a similar quantity of water is absorbed by the sugar, owing to which fact the conditions for the destructive work of the micro-organisms are improved.

Hence to preserve the stored sugar as far as possible it will be necessary to keep the degree of humidity low in the warehouses, and in order to be able to do so, it is indispensable to measure it. The feeling of dryness or moistness in the warehouse is a very subjective factor and greatly dependent on the prevailing temperature; it is therefore advisable to place hygrometers inside and outside the storehouses, so that one can know when to ventilate the sugar, and when to keep the doors closed and exclude the free admission of moist air.

Finally, the points of contact between the sugar and the eventual sources of moisture should be reduced as far as possible by heaping up the bags in large and close piles at a convenient distance from the exterior walls of the buildings.

* " Int. Sugar Journal," 1918, 543.

CHAPTER V

EXHAUSTED MOLASSES

I.—Definition and Formation of Molasses

Definition.—On curing the final massecuites, a syrupy liquid is obtained, which, on further concentration and cooling, does not give any more crystals, and bears the name of " exhausted molasses." Although from it sugar can no longer be crystallized out, it always contains (in conjunction with the accumulated impurities originating from the clarified juice) a considerable amount of sucrose, which is therefore practically lost. This is due to the fact that the non-sugars present in the molasses prevent a certain percentage of sucrose from crystallizing out even if the concentration be pushed still farther.

Impossibility of avoiding Molasses.—Unless we succeed in removing from the juice everything but sucrose and water during clarification, which is an impossibility, we are sure to find molasses at the end of every process of sugar extraction ; and systems which claim to make sugar without any molasses can, without any further investigation, be deemed unreliable.* The nature and conditions of molasses formation have for a long time been a point of dispute between different investigators, and even now the question is not yet definitely settled, although unanimity exists as regards the principal points.

The Mechanical Theory.—Formerly it was the general opinion that sucrose could not crystallize from the final molasses because the non-saccharine constituents made the liquid so viscous at high concentrations that the sucrose molecules were prevented from combining into crystals, and were thus forced to remain in solution. At first sight, this explanation appears very plausible,

* The so-called " concrete sugar " is only an apparent exception to this rule, as it contains the whole of the dry substance of the juice, crystals, and molasses in one agglomerated mass, and is therefore not a pure crystallised sugar.

but its probability decreases if we remember that sucrose crystallizes slowly yet perfectly from very stiff jellies, and a sucrose crystal can be made to grow regularly when suspended in a solution of sugar in isinglass.

Further, this theory is at variance with the fact that sugar crystallizes from an osmosed molasses (where the non-diffusing viscous colloids are accumulated), whereas, according to the theory, crystallization should have totally ceased.

Chemical Molasses Theory.—The so-called " mechanical theory " was soon abandoned, and the opinion was formed that the prevention of crystallization was due to chemical influences. A general idea prevailed that the sucrose combined with other constituents of the molasses to form very soluble bodies, so that the solubility of the sucrose and of the accompanying constituent was much greater than when both were present separately.

Mutual Increase or Decrease of Solubility between Sucrose and Salts.—According to Dubrunfaut, a solution of common salt dissolves more sucrose than the water contained in the solution would do, if it did not contain the salt. On the other hand, a saturated sucrose solution dissolves twice as much salt as the water contained in the solution would alone have done. Assuming that sucrose and sodium chloride enter into a very soluble combination, we can easily explain the formation of molasses; and if this simple conclusion from Dubrunfaut's experiment had been made sooner, it would have saved much misunderstanding.

So-called Positive, Indifferent, and Negative Molasses-formers.—In beetroot molasses, the solubility of sucrose in the contained water is greater than in pure water at the same temperature, and as in former days beetroot molasses was the sole object of investigation, the idea arose that the solubility of sucrose is always higher than the normal in every molasses, and all theories and observations were based on that principle. Whilst it was known that some salts increased (as was the saying) the solubility of sucrose, and therefore possessed a melassigenic power, a great number of substances were tested as to their property of increasing or decreasing the solubility of sucrose in water, and thus determining their melassigenic power. Under the impression that the molasses-forming power was always identical with the increase in the solubility of sucrose, the different bodies were divided into positive, negative and indifferent molasses-formers, according to the increase, decrease, or stability of the solubility of sucrose in aqueous solution in the presence of those bodies. This division is still in great favour in most treatises and publications, and the terms *positive* and *negative molasses-formers* are still widely used. It was, however, observed that some bodies proved to be negative molasses-formers in dilute concentrations, but positive in high concentrations, and finally the subject led to a mass of misunderstanding and controversy.

Simple Combination of Sucrose and Salts.—The simplest explanation assumes that all salts, both organic and inorganic, combine with sucrose to form hydrated bodies, each having its own solubility. If we first consider the case where the solution contains as much sucrose and salt as possible, we observe two alternatives. Either the combination is very soluble in water, much salt and sucrose enters into solution, and we may call the salt a positive molasses-former; or the combination is only slightly soluble, the saturated solution therefore only contains relatively little salt and sucrose, and the salt thus belongs to the negative molasses-formers.

Köhler* showed that, in general, the solubility of sucrose in a solution of non-sugar, and also the solubility of the non-sugar in a sucrose solution, are mutually in such a relation that from either body as much is dissolved as it can dissolve of the other body, and that solutions containing sucrose and large quantities of non-sugar contain (with the same proportion of water), sometimes more and sometimes less sucrose than corresponds with the solubility of pure sucrose in pure water. The most evident explanation is that, when present in an excess of both sucrose and salt, water can dissolve so much of both as corresponds with the solubility of the new combination formed between the sucrose and the salt. According to that solubility, the water can dissolve more or less combined sucrose than if the salt had not been present and the sucrose had remained in the uncombined state.

Köhler investigated how much salt was dissolved at the temperature of 31·25° C. in 100 c.c. of pure water, and how much in 100 c.c. of a sucrose solution saturated at that same temperature, and finally how many grams of sucrose were contained in 100 c.c. of this salt solution.

Name of the Salts.	Grms. of Salt in 100 c.c. of pure water.	Grms. of Salt in 100 c.c. of Saturated Sucrose Solution.	Grams. of Sucrose in 100 c.c. of the Salt Solution.
Potassium Chloride ..	38·2	44·8	246·5
Potassium Carbonate ..	95·9	105·4	265·4
Potassium Acetate ..	286·3	293·5	324·8
Potassium Citrate ..	159·7	219·0	303·9
Sodium Chloride ..	35·9	42·3	236·3
Sodium Carbonate ..	22·0	24·4	229·2
Sodium Acetate ..	46·9	57·3	237·6
Potassium Sulphate ..	12·4	10·4	219 0
Potassium Nitrate ..	47·4	41·9	224 7
Sodium Sulphate ..	45·4	30·5	183 7
Calcium Acetate ..	35·4	26·3	190·3
Calcium Chloride ..	88·5	79·9	135·1
Magnesium Sulphate ..	47·5	36·0	119·6

* "Zeitschr. Rübenzuckerind.," 47, 441.

Solubility of the Combinations.—As, in every case, a decreased solubility of the salt coincides with a lower solubility of the sucrose, it is evident that in every instance, where both the components occur in such large quantities that they can combine together to their full extent, the solubility of the constituents is regulated by that of their combination, and no longer by that of the respective bodies in an uncombined state. In connection with this point Degener* found that the lowest solubilities of salt and sucrose in solutions of these two in water correspond with the formulæ of $NaCl + 4\ C_{12}H_{22}O_{11}$ for the sodium chloride combination, and with $CaCl_2 + 3\ C_{12}H_{22}O_{11}$ for the corresponding calcium chloride combination.

Solubility of Sucrose.—In the case of one of the constituents being deficient, it is clear that no more of the combination is formed than corresponds with the amount of that constituent, so that we can no longer find the solubility of the combination only, but have now to take into account the amount of the still uncombined portion of the other constituent. If it is the salt which is deficient, then there exists in the solution, besides the sucrose in combination, a certain amount of free sucrose, the solubility of which may not be neglected although it may be influenced by the presence of the combination. As these combinations are hydrated bodies, we can suppose the solution to consist of free water, free sucrose, combined water and a sucrose-salt combination. If we wished to ascertain whether the presence of a salt influences the solubility of sucrose in water and in which direction, and with that object only took into account the total amount of sucrose and the total amount of water, we can easily imagine what highly varying results might accrue, according to the different proportions in which the four constituents mutually existed.

When the amount of salt is small and that of sucrose considerable, only a little of the hydrated sucrose-salt combination will be formed, and very little free sucrose and free water will be withdrawn from the solution but sufficient to prevent the remaining free water from keeping all of the remaining free sucrose in solution, part of the latter crystallizing. We have, therefore, in the remaining portion of free water its full content of sucrose, and in the hydrate a little combined sucrose, and if the sum of these two quantities happens to be smaller than corresponds with the figure for the solubility of sucrose in water, the total amount of sugar in the total amount of water is smaller than the normal solubility, and we should call it a negative molasses-former. When the amount of salt increases, then of course the quantity of salt-sucrose combination increases equally, until at the end all the sucrose has combined with salt, and we no longer have to deal with a solution of sucrose in a decreasing amount of free water, but only the solubility of a sucrose-salt combination, which is, as we saw before, always higher than that of the sucrose existing in it. This

* "Deutsche Zuckerindustrie," 20, 2149.

explains why in Herzfeld's experiments* additions of small portions of non-sugar were apt to lower the solubility of the total sucrose in the total water, whereas larger portions increased it.

Cases from Practice.—In pure juices and syrups we see instances of the former case; in these products the amount of the combination is far less than the free sucrose, and accordingly the solubility of the total sucrose in the total water should be lower rather than higher than the normal figure. In the exhausted molasses, on the contrary, from which the superfluous sucrose has been removed by successive crystallizations, and only the saturated sucrose-salt combination left, it depends solely on the degree of concentration of this strongly evaporated product how much sucrose is found on 100 parts of water. In beetroot molasses, crystallized in large crystallizing tanks, this amount of water is generally such that the ratio of sucrose to water is greater than in a pure saturated sucrose solution, but, as we shall see later on, this is only a surmise; it might also have been the reverse. But because beetroot molasses were studied almost exclusively, and in this substance the higher proportion was met with, the idea of supersaturated solutions has unconsciously taken root in the minds of all investigators who studied molasses and their formation, and they have always endeavoured to explain that supersaturation by the hypothesis of the presence of substances which prevented crystallization.

Definition of an Ideal Molasses.—A short definition of molasses (*i.e.*, of an ideal molasses) according to the author's theory is: *A hydrated syrupy-liquid combination of sucrose and salts.*

After his experience of beetroot molasses, he was struck by the much smaller ratio of sucrose to water in cane molasses than corresponds with the solubility of sucrose in water, instead of the higher one he had always been led to suppose.

Ratio of Sucrose to Water in Cane Molasses.—At the average Java temperature of about 28°C. one part of water can keep in solution 2·162 parts of sucrose, so that if that solubility was not changed by the presence of other bodies in the molasses, the polarization of a molasses with 25 per cent. of water should be 53·9. According to the following table of analyses of mother-liquors crystallized in tanks, the ratio of sucrose to water is generally less than that of the two pure ingredients, and generally those containing the most glucose show the lowest ratio:—

* "Zeitschr. Rübenzuckerind.," 42, 182, 240.

No.	Dry Substance.	Sucrose.	Reducing Sugar.	Ash.	Water.	Quotient of Purity.	Sucrose on 100 parts of water.*	Ratio of Red'cing Sugar to Ash.
0	—	—	—	—	—	100·00	216·2	—
1	74·88	59·20	7·29	6·07	25·12	79·06	235·7	1·20
2	74·29	58·70	9·97	3·82	25·71	79·01	228·3	2·61
3	82·88	47·80	14·53	6·66	17·12	57·67	279·2	2·18
4	74·69	50·20	15·14	4·35	25·31	67·21	198·3	3·48
5	82·90	43·70	15·80	6·50	17·10	52·17	255·6	2·43
6	72·23	49·00	16·52	2·52	27·77	67·84	176·4	6·56
7	83·55	47·90	17·44	7·00	16·45	57·33	291·2	2·49
8	71·07	42·90	19·93	3·00	28·93	60·36	148·3	6·64
9	71·55	43·10	20·87	3·39	28·45	60·23	151·5	6·16
10	77·10	34·10	23·00	7·80	22·90	44·23	148·9	2·95
11	71·40	32·40	23·00	5·20	28·60	45·38	113·3	4·42
12	76·80	36·90	26·30	4·97	23·20	48·05	159·1	5·29
13	75·86	30·90	27·47	6·37	24·14	40·73	128·0	4·31
14	73·88	32·90	27·53	3·72	26·12	44·53	126·0	7·40
15	80·90	35·30	27·60	8·08	19·10	43·63	184·8	3·42
16	74·62	34·05	27·78	4·16	25·38	45·63	134·2	6·68
17	75·50	33·70	28·12	3·65	24·50	44·64	137·6	7·70
18	77·02	33·80	28·20	6·88	22·98	43·88	147·1	4·10
19	73·78	32·19	30·30	3·77	26·22	43·64	122·8	8·04
20	74·23	30·69	31·73	3·65	25·77	41·34	119·1	8·69

Molasses-forming Property of Reducing Sugar.—This appears very strange, since, in existing literature, glucose is always referred to as a molasses-former, and one would expect high solubilities, instead of the low values found by actual analysis. Gunning** has said that glucose renders a certain amount of sucrose uncrystallizable. Pellet† put down the melassigenic coefficient of glucose as 0·60. Flourens‡ allows various values for that figure, fluctuating between 0·30 and 1·0, whilst Degener§ mentions having found glucose to possess no melassigenic power of its own, but able to transform sucrose into invert sugar by the action of its acid products of decomposition. In that connection Pellet† also mentions two distinct sources of molasses-forming by glucose, namely, one as a consequence of the inverting action of glucose, which he calculates as 0·16, and further the pure melassigenic power, at a value of 0·60. The alleged inverting action, which has, however, never been proved to exist, may be ruled out of consideration here, where only the melassigenic power itself is considered.

* At 28° C. ** Saccharimetrie en Accijns. † "Zeitschr. Rübenzuckerind," 1879, 806.
‡ "Journal des Fabricants de Sucre," 1880, 40. § "Zeitschr. Rübenzuckerind," 1881, 514.

While many authors thus ascribe different values to the melassigenic power of glucose, the author has made some experiments to ascertain how this sugar influences the solubility of sucrose in water.

His laboratory experiments led him to the conviction that neither glucose nor invert sugar affects the solubility of sucrose, and also that this sugar does not possess an inverting action under the conditions prevailing on simple dissolution and crystallization at low temperatures.

Melassigenic Power of other Non-Sugars.—Neither can the other constituents of the molasses be made responsible for the observed lowering of the solubility of sucrose in cane sugar molasses, because they—organic and inorganic salts, pectin, gums, nitrogenous bodies, caramel, etc.—are also found in beetroot molasses, and are there looked upon as causing the higher solubility of the sucrose.

Joint Action of Reducing Sugar and Salts.—As the joint action of reducing sugars and salts might cause the phenomenon alluded to here, the author* prepared at the boiling point solutions containing sucrose, reducing sugar, salts of organic and inorganic acids and water. The amounts of sucrose and of water, which were the same in every one of the solutions, were thus chosen so that in all probability sucrose would crystallize out after the cooled solution had been allowed to stand during a long time. The amount of salts was such that the amount of ash was the same in every experiment, while the proportion of the reducing sugar showed great variations.

When dissolving the salts it was observed that they passed into solution much more slowly and more difficultly in the thick liquid containing sugars than they would have done in the same amount of pure water as was present in the syrups.

Inversion by Reducing Sugar and Inorganic Salt.—The mixtures containing reducing sugar and inorganic salts revealed a striking fact; their presence gave rise to so much free acid of a highly invertive power from the inorganic salt, that at the high temperature at which the solutions had been made some sucrose was inverted. For this reason the action of inorganic salts on the solubility of sucrose in presence of reducing sugar could not be studied, while their influence in the absence of that body is sufficiently known from Herzfeld's experiments. When organic salts were also present in solution, the inversion occasioned by the far less invertive acids was so small that in every case enough sucrose was left intact to give a supersaturated solution, as is shown by the large quantities of sucrose which crystallized. Owing to this circumstance it was only possible to study the action of inorganic salts by comparing them with mixtures containing organic ones.

* "Archief voor de Java Suikerindustrie," 1895, 318.

We observe that in every case where reducing sugar and salts were present in the solutions, the amount of sucrose crystallizing out increased in proportion as the liquid contained more of the reducing sugar for the same amount of salts.

Identical Action of Organic and Inorganic Salts.—In the mixtures containing both organic and inorganic salts, this crystallization is analogous to that found in those which contain the same amount of organic salts only, which shows that in this respect the action of organic and inorganic salts does not differ.

A completely identical result cannot be expected, as every salt has an action of its own, but we see that substitution of a part of the organic salts by the equivalent weight of an inorganic one has a slight tendency to promote crystallization of sucrose. Calcium salts exert the strongest influence, next come potash, and finally soda salts.

Reducing Sugar can take the Place of Sucrose.—Having seen that the simultaneous presence of any kind of salt and reducing sugar promotes crystallization of sucrose and that crystallization is more rapid when, for a given amount of salt, the proportion of reducing sugar is greatest, we extended our hypothesis already mentioned still further. We had already remarked that, in absence of reducing sugar, molasses is a hydrated combination between salts and sucrose, which is stable in a concentrated state and is only dissociated in a very dilute state. If we add reducing sugar to that combination, the salts will combine with the glucose and fructose just as they did with sucrose, giving rise to a sucrose-glucose-fructose-salt combination, or more shortly, a sugar-salt combination. The proportion in which the five components (three sugars, water and salts) occur depends on the nature of the salt; but in presence of glucose or invert sugar, less sucrose enters into combination for the same amount of salt, than when invert sugar is not present in the mixture. This amount of sucrose then becomes free, and can crystallize out.

If this hypothesis be true, then, when mixing dry glucose with a quantity of the sucrose-salt combination, we must expect to see a part of the sucrose displaced by the invert sugar, and so be caused to crystallize.

For the same reason, when bringing together a saturated solution of sucrose with glucose and a dry salt, the salt will combine with the sugars and with water, and if the conditions of the experiment are well chosen, there will not remain sufficient free water left to keep the whole of the still free sucrose in solution; that surplus will therefore crystallize.

Definition of an Ideal Cane Molasses.—Experiments have corroborated this hypothesis, and have led us to the following definition of an ideal cane molasses:—

Molasses is a hydrated combination between sugar and salts, which cannot be broken up by dissociation in a concentrated state, and therefore cannot yield sucrose in a crystallized form.

Its Composition.—The total amount of sugars (sucrose+glucose+fructose) in an exhausted cane molasses is about 55 per cent., and their relative proportions depend solely on the ratio of reducing sugars (glucose + fructose) to ash. The water content is about 20 per cent., and the ash about 9 per cent. In the case of juice containing no reducing sugar, the salts combine with sucrose only, and sucrose is the only sugar represented in the 55 per cent., so that the proportion between sucrose and water is in this case as 55 : 20. If, on the contrary, the juice contains reducing sugar as well, then this will also combine with a part of the salts, causing the total salts to hold less sucrose in combination. If, for instance, the amount of reducing sugar be 10 per cent., and of sucrose 45 per cent., then on 20 parts of water, we find only 45 parts of sucrose ; and in a case (which often occurs in cane molasses) of 30 sucrose and 25 reducing sugar, we only find a proportion of 30 sucrose to 20 water.

Sucrose and Water in Presence of Invert Sugar.—The supposed increase or decrease of the solubility of sucrose in the water of the molasses had long dominated the study of molasses-formation. If we accept that idea we notice the following solubilities for the corresponding relations between sucrose and invert sugar :—

Invert Sugar	0	..	275	parts of sucrose	on	100 water.
,, ,,	5	..	250	,,	,,	,,
,, ,,	10	..	225	,,	,,	,,
,, ,,	15	..	200	,,	,,	,,
,, ,,	20	..	175	,,	,,	,,
,, ,,	25	..	150	,,	,,	,,

It will be seen that the so-called solubility of sucrose falls as the amount of glucose (or, in general, invert or reducing sugar) rises. Generally speaking, we can say that with a high ratio between glucose and ash, the solubility of sucrose decreases.

But this statement is far from being precise, and is apt to give rise to much confusion. There is no question of solubility of sucrose at all, but only of the composition of a combination, which contains on an average 55 per cent. sugars, 25 per cent. salts, and 20 per cent. water. If there is a large proportion of reducing sugar in that 55 per cent., then there is little sucrose ; if, on the contrary, the percentage of glucose is small, the syrupy combination contains much sucrose. The ratio between the sucrose and water in such combinations has nothing whatever to do with a positive or negative melassigenic power of the constituents of the molasses.

It is evident that in the author's theory there is no place for negative molasses-formers ; the non-sugars, especially salts, combine with sugars, and it

depends entirely on the mutual relation of sucrose and glucose whether much or little sucrose enters into combination.

Van der Linden's Observations.—Van der Linden* considered the formation of molasses from a view-point of the phase rule and distinguished the cases, in which the constituents of which the molasses is composed, *i.e.*, sucrose, non-sucrose and water, enter into mutual compounds, and in which they do not.

Further the question may be asked if molasses has or has not to be looked upon as an undercooled eutectic mixture of the various components, or as an exactly saturated, either stable or meta-stable sucrose solution.

It is not impossible that in practice both cases may occur, and that therefore some molasses must be regarded as eutectic mixtures, while others are exactly saturated sucrose solutions and very probably stable ones.

In the first case molasses and final massecuites possess the properties enumerated below, which are identical whether the components mutually form compounds or not.

1. A fixed value for degrees Brix goes with every final massecuite of a given quotient of purity, below which the massecuite cannot be concentrated if it is desired to obtain an exhausted molasses after cooling and centrifugalling.

2. Every final massecuite concentrated beyond the degrees Brix referred to above may be diluted with water to that degree Brix, without the quotient of purity of the molasses undergoing any noticeable increase.

In this respect two cases have to be distinguished :—

(*a*) the purity of the exhausted molasses sinks as the water content rises.

(*b*) the purity rises with the water content.

The former possibility is the most probable one.

3. If more water is added to the final massecuite so that the Brix comes down under the limit, the purity will rise in both cases.

4. The lower the quotient of purity of a final massecuite the larger the amount of water, *i.e.*, the lower the degree Brix to which it may be kept.

5. A final massecuite having a low quotient of purity will stand a greater dilution with water than a high purity one, concentrated to the same degree Brix.

6. The higher the degree Brix is to which the final massecuite has been concentrated, the higher the temperature may be at which it is being cured, while yet a low purity molasses is obtained.

Here again the two cases mentioned under (2) may be distinguished ; that

* "Archief voor de Java Suikerindustrie," 1915, 1033, 1389.

is to say, if the purity of the exhausted molasses rises with the concentration, such a practice will be favourable, but unfavourable in the other case.

In both cases the temperature at which curing is carried out should never be allowed to rise over the temperature of equilibrium of the corresponding molasses. This point should especially be considered in cases where a high content of reducing sugars is accompanied by a low ash content.

7. Behaviour of exhausted molasses on addition and evaporation of water.

(*a*) The quotient of purity of the molasses sinks on adding a small amount of water to an exhausted molasses and cooling the mixture; and sucrose separates out, thus giving a decrease in the quotient of purity. If, on the contrary, water is evaporated, the mass will remain homogeneous, provided that no non-sucrose separates out.

(*b*) The quotient of purity of the molasses rises. Addition of water and consecutive cooling may cause non-sucrose to separate ; if this is not the case, we only see dilution.

Evaporation of water may lead to crystallization of sucrose, *i.e.*, decrease of the quotient of purity of the molasses. The limit is only attained when all of the water is evaporated.

8. A molasses, of which the non-sucrose chiefly consists of salts, will have a high quotient of purity (beet molasses).

9. A molasses, of which the non-sucrose chiefly consists of reducing sugar, will have a low quotient of purity (cane molasses).

10. The quotient of purity of the molasses depends on the ratio between reducing sugar and ash ; if the ratio is high, the purity will be low, and if the ratio is low, the purity will be high.

11. Addition of reducing sugar to syrup cannot decrease the purity of the exhausted molasses to be expected therefrom below a fixed limit.

12. Any body having a low melting point can have the same effect as reducing sugar in this respect, provided that its molecular weight does not differ too much and that it does not separate out.

As regards the second case, viz., that the molasses has to be considered as an exactly saturated stable sucrose solution, all the properties known by experience to belong to molasses and final massecuites might be traced back to that possibility.

It appears that the diagram representing the system sucrose-glucose-salt-water shows a somewhat peculiar shape, from which the conditions of solubility of sucrose in presence of salt and glucose can be derived. An explanation of that peculiar condition of the diagram is not to be expected from the phase rule, but will have to be evolved from the so-called theories of formation of molasses.

Since in the literature dealing with sugar no record of investigations could

be found giving an absolute answer to the question, as to which of the two categories referred to here the molasses belongs, it was found necessary to continue the researches.

The procedure was already indicated by the definition of the two cases. If molasses is an undercooled eutectic mixture, no sucrose will be dissolved on heating molasses with sucrose crystals till the temperature is attained at which a eutectic mixture of the given composition is just in equilibrium. If, therefore, the purity of the mother-liquor is continually ascertained at various temperatures during a range of sufficient length, the quotient of purity will be found to remain the same at the beginning and to start rising from a certain point onward.

If, on the contrary, molasses is an exactly saturated sucrose solution, the quotient of purity will rise at once if the temperature is raised, since solubility of sucrose in its mother-liquors is increased as temperature rises.

The results of the experiments undertaken by van der Linden were rendered uncertain by the development of very minute sucrose crystals, which separated from the thick masses and could not be filtered off.

Helderman's Investigations.—Helderman* directs attention to the determination of the solubility of sucrose in water in presence of reducing sugar by Van der Linden, whose tables have been mentioned on page 11 of this work. These investigations show that, contrary to the views expressed by many other authors enumerated on pages 294 and 295, reducing sugar diminishes the solubility in water.

Further, Helderman†, equally contrary to the opinion of a great many other investigators, maintains that neither sucrose nor glucose enters into compounds with salts, and corroborates his view-point by references to the phase rule, which have, however, been contested by others.

He comes to the conclusion that molasses may not be considered as undercooled eutectic mixtures, but as exactly saturated sucrose solutions.‡

Finally he is convinced that the conclusion derived from the so-called chemical molasses theory, viz., that an exhausted molasses is unable to give off sucrose crystals on further evaporation, but only loses water of hydration during that operation, does not agree with the facts.

On the contrary, he is of opinion that every molasses can crystallize out still further on continued evaporation; only the velocity of crystallization and the size of the crystals become very much reduced, so that the last crystals may be only visible under the microscope.

Helderman gives the following definition for molasses: Molasses is a

* "Archief voor de Java Suikerindustrie," 1921, 1253.
† *Ibid.*, 1920, 1701; 1920, 2305; 1921, 1167.
‡ "Int. Sugar Journal," 1921, 687.

solution saturated in sucrose, the solubility of which is influenced by the presence of various substances, such as reducing sugar, salts and other substances, colloidal and crystalloid.

Conclusions.—We shall refrain from further discussing the theories and confine ourselves to expressing them one after another. We only want to observe here that in such concentrated mixtures as final molasses we should no longer use as a basis of comparison the terms of solubility of sucrose in water.

We have seen in the foregoing chapter that final massecuites may be evaporated to a density of about 100° Brix, and that therefore the amount of water still left in the exhausted molasses is a very insignificant one. It is therefore not adequate to apply the conditions of dissolution of sucrose in and crystallization from aqueous solutions unchanged to those in and from liquids from which the water as far as possible has been driven out.

If one wants to consider molasses as a saturated sucrose solution, one should make sure not to think of a solution in water but in a melted mass of non-sucrose.

Further it is clear that the conditions of the formation of molasses must be studied under similar circumstances as occur in practical working. We must, therefore, start from an excess of sugar (chiefly sucrose) over salts, because in practical sugar manufacture the sucrose always predominates in the juices and syrups; it constantly grows less through the consecutive crystallizations until no more is left than can possibly be left in solution in the stiff, almost anhydrous mass, which constitutes the final molasses.

Hence, in this work, the solubility of sucrose in water is not studied beyond the stage where the juice is concentrated to syrup. Sucrose still preponderates in the concentrated syrups, as compared with the relatively small amount of non-sucrose, but in the first molasses (from which a great deal of sucrose has been withdrawn) this proportion is already so disturbed that it may be regarded as a solution of sugar in a mixture of reducing sugars, salts and water, which latter constituent gradually decreases till it reaches its minimum in the exhausted molasses.

This same observation holds as well for the case in which the sugars and salts enter into compounds, as for that in which uncombined sucrose is simply dissolved. In the former case the sucrose is immobilized by its being combined with the salts; in the latter it is immobilized by its being dissolved in the non-sucrose.

II.—Composition of Exhausted Molasses.

When speaking hereafter of "exhausted molasses," we only mean that the molasses removed from the course of manufacture is *practically* exhausted, even though it might be possible to extract yet more sucrose from it by some device or other.

Composition of Java Molasses.— In the first edition of this work we gave the full analyses of a great number of Java molasses, dating from 1905.*

We here only reproduce ten of them, divided into two groups, viz., those obtained from final mixed massecuites boiled to grain and those obtained from massecuites boiled to string-proof and cooled in tanks.

As the molasses were obtained in the usual course of manufacture, they were probably somewhat diluted, or might have contained minute sugar crystals in suspension, and were therefore not identical with the mother-liquors from the final crystallization.

I.—MOLASSES FROM MASSECUITES, BOILED TO GRAIN.

Number.	Purity, Original Juice.	Degrees Brix.	Actual Dry Substance.	Direct Polarization.	Polarization after Clerget.	Apparent Purity.	True Purity.	Reducing Sugar.	Ash.	Gummy Matter.	Organic Non-Sugar.	Rotatory Power of the Reducing Sugar.	Factor for the difference between Brix and Dry Substance.	Sucrose dissolved in 100 parts of Water.	Ratio between Reducing Sugar and Ash.
1	82·0	87·02	81·30	32·6	38·43	36·32	47·27	21·73	8·21	1·71	12·83	— 26·8	0·68	206	2·65
2	85·1	91·92	85·41	31·1	35·8	33·83	41·92	24·6	10·45	1·22	14·56	— 19·1	0·62	244	2·35
3	84·06	88·32	81·76	31·8	36·05	36·00	45·35	23·81	9·23	1·84	12·67	— 19·6	0·71	197	2·58
4	85·3	92·56	87·70	33·2	39·0	35·86	44·47	29·4	8·00	2·01	11·23	— 19·7	0·61	317	3·67
5	86·9	86·42	81·15	32·2	37·80	37·26	46·58	22·73	8·39	2·00	12·23	— 24·7	0·63	205	2·71

Number.	Difference in Purity (apparent) between Juice and Molasses.	Difference in Purity (true) between Juice and Molasses.	Total Ash.	Ash insoluble in Water.	Ash soluble in Water.	Iron and Calcium Phosphate.	Lime.	Magnesia.	Potash.	Soda.	Silica.	Sulphuric Acid.	Carbonic Acid in the Ash.	Chlorine.	Lime in 100 parts of Ash.
1	45·68	34·73	8·21	1·66	6·55	0·42	0·96	0·16	3·93	0·20	0·16	0·82	1·69	0·34	11·69
2	51·27	43·18	10·45	1·75	8·70	0·44	0·37	0·15	5·48	0·12	0·22	0·69	2·27	0·99	3·54
3	48·06	38·71	9·23	2·18	7·05	0·41	0·85	0·17	4·01	0·04	0·15	1·44	1·60	0·64	9·21
4	49·44	40·83	8·00	2·75	5·25	0·52	0·67	0·10	3·34	0·00	0·51	0·86	1·46	0·17	8·37
5	49·64	40·32	8·39	2·10	6·29	0·76	0·47	0·49	3·76	0·13	0·34	1·21	1·15	0·68	5·61

* See "Int. Sugar Journal," 1906, 29.

II.—MOLASSES FROM MASSECUITES BOILED TO STRING-PROOF AND COOLED IN TANKS.

Number.	Purity, Original Juice.	Degrees Brix.	Actual Dry Substance.	Direct Polarization.	Polarization after Clerget.	Apparent Purity.	True Purity.	Reducing Sugar.	Ash.	Gummy Matter.	Organic Non-Sugar.	Rotatory Power of the Reducing Sugar.	Factor for the Difference between Brix and Dry Substance.	Sucrose dissolved in 100 parts of Water.	Ratio between Reducing Sugar and Ash.
6	85·24	81·94	76·72	30·2	33·6	36·86	43·80	21·2	8·60	1·11	13·32	— 16·0	0·61	145	2·47
7	86·3	84·66	77·36	27·6	33·78	32·60	43·67	24 39	9·24	2·47	9·95	— 27·8	0·79	149	2·64
8	83·44	80·92	77·52	23·8	27·7	29·41	35·86	27·8	7·14	1·69	14·88	— 14·0	0·48	123	3·89
9	88·9	72·96	69·96	27·0	27·78	37·01	39·69	16·95	7·22	1·60	18·01	— 4·5	0·42	92	2·35
10	89·94	87·00	82·76	29·8	32·1	34·25	38·79	26·3	8·09	1·91	16·27	— 8·7	0·52	186	3·25

Number.	Difference in Purity (apparent) between Juice and Molasses.	Difference in Purity (true) between Juice and Molasses.	Total Ash.	Ash insoluble in Water.	Ash soluble in Water.	Iron and Calcium Phosphate.	Lime.	Magnesia.	Potash.	Soda.	Silica.	Sulphuric Acid.	Carbonic Acid in the Ash.	Chlorine.	Lime in 100 parts of Ash.
6	48·38	41·44	8·60	2·40	6·20	0·37	0·77	0·14	3·76	0·44	0·10	1·01	1·32	0·45	8·95
7	53·7	42·63	9·24	1·53	7·71	0·91	0·20	0·07	4·68	0·15	0·18	0·85	1·49	0·72	2·16
8	54·03	47·58	7·14	1·93	5·21	0·65	0·48	0·09	3·17	0·09	0·22	0·65	1·43	0·20	6·72
9	51·89	49·21	7·22	1·84	5·38	0·55	0·54	0·15	3·24	0·06	0·16	0·74	1·50	0·24	7·48
10	55 69	51·15	8·09	2·43	5·66	1·07	0·53	0·10	3·54	0·00	0·30	0·30	1·95	0·27	6·55

Since that time conditions in Java have changed in so far that the string-proof boiled massecuites have dropped entirely into the background, while almost all final massecuites are now boiled to grain. Further, concentration has been pushed much farther nowadays than was formerly the case, causing the figure for degrees Brix to be now much higher than in the instances given above.

We give here the averages for the final molasses in Java during a series of years derived from the final tables of the Mutual Control of Java Sugar Factories.

DEFECATION.

Constituents.	1914.	1915.	1917.	1918.	1919.	1921.	1922.	1923.
Brix	90·87	91·44	90·93	91·14	91·93	92·35	92·15	92·68
Polarization	29·56	29·44	29·70	30·16	29·48	29·72	29·35	28·97
Q. Purity $\frac{\text{Brix}}{\text{Pol.}}$	32·5	32·19	32·70	33·1	32·1	32·2	31·9	31·3
Sucrose	33·25	33·26	33·58	33·91	33·62	33·40	32·93	32·47
Q. Purity $\frac{\text{Brix}}{\text{Sucrose}}$	36·59	36·4	37·0	37·2	36·6	36·2	35·7	35·0
Reducing Sugar	29·13	29·68	27·31	26·56	27·65	27·78	27·66	26·71
Ash	—	8·77	9·11	9·50	9·96	9·85	9·82	10·44

DEFECATION—SULPHITATION.

Constituents.	1914.	1915.	1917.	1918.	1919.	1921.	1922.	1923.
Brix	91·38	92·06	91·60	92·16	93·20	92·82	92·98	93·39
Polarization	29·90	29·77	29·60	30·24	29·82	30·06	29·41	29·05
Q. Purity $\frac{\text{Brix}}{\text{Pol.}}$	32·7	32·24	32·3	32·8	32·1	32·4	31·6	31·1
Sucrose	33·36	33·3	33·15	33·90	33·32	33·52	33·07	32·47
Q. Purity $\frac{\text{Brix}}{\text{Sucrose}}$	35·41	36·2	36·1	36·8	35·8	36·1	35·6	34·7
Reducing Sugar	29·66	29·38	28·10	27·05	28·39	26·91	27·46	26·46
Ash	—	9·23	8·65	9·19	9·59	9·72	9·70	10·10

CARBONATATION.

Constituents.	1914.	1915.	1917.	1918.	1919.	1921.	1922.	1923.
Brix	88·77	89·44	90·77	91·18	91·67	91·51	90·86	91·55
Polarization	29·72	29·94	30·17	30·73	30·11	30·73	30·31	29·27
Q. Purity $\frac{\text{Brix}}{\text{Pol.}}$	33·5	33·5	33·2	33·7	32·9	33·6	33·4	32·0
Sucrose	32·09	32·31	32·77	33·19	32·74	33·34	32·56	32·16
Q. Purity $\frac{\text{Brix}}{\text{Sucrose}}$	36·15	36·1	36·1	36·4	35·7	36·4	35·8	35·5
Reducing Sugar	27·38	25·81	26·68	24·66	25·76	26·70	26·05	27·05
Ash	—	—	8·29	9·38	9·22	9·07	8·97	9·25

Composition of Hawaiian Molasses.—Van Heemskerk Duker* publishes the following data concerning exhausted molasses from Hawaii subdivided according to the methods of boiling and cooling.

* "Louisiana Planter," 1924, 71, 112.

BOILED TO GRAIN.

No.	Dry Substance.	Apparent Purity.	Per Cent. Dry Substance.		
			Sucrose.	Red. Sugar.	Total Sugars
1	80·19	36·18	39·82	19·60	59·42
2	81·07	37·23	41·55	18·03	59·38
3	79·42	39·95	43·67	16·61	60·28
4	79·69	34·47	39·24	20·27	59·51

BOILED TO STRING-PROOF.

No.	Dry Substance.	Apparent Purity.	Per Cent. Dry Substance.		
			Sucrose.	Red. Sugar.	Total Sugars
1	74·23	36·43	43·60	18·47	62·07
2	78·53	40·36	46·16	14·70	60·86
3	77·76	41·33	47·07	15·93	63·00
4	78·47	42·90	49·02	13·50	62·52

We also abstract from the periodical statistics of the Sugar Club of Havana the following instances, picked at random, from the data of 1924, on exhausted molasses in Cuba.

No.	Brix.	Pol.	Sucrose.	Q. Pur. $\frac{\text{Brix}}{\text{Pol.}}$	Q. Pur. $\frac{\text{Brix}}{\text{Sucrose}}$	Red. Sugars
1	85·20	31·21	38·94	36·63	45·34	—
2	88·02	34·03	37·70	38·66	42·54	14·39
3	85·36	27·06	36·09	31·70	46·50	21·70
4	83·03	29·90	32·44	36·01	39·07	14·99
5	86·92	33·92	37·27	33·92	42·87	16·44
6	88·47	27·85	36·92	31·48	45·06	23·02
7	82·27	29·71	32·44	36·11	39·43	15·84
8	89·08	27·46	35·92	30·83	43·55	24·82
9	88·87	25·81	28·74	29·04	32·34	17·84
10	88·54	28·64	35·79	32·35	43·33	20·36

Composition of Cuban Molasses.—The yearly statistics of the Secretary of Agriculture, Commerce and Industry in Cuba, mention the following averages for the quotient of purity of exhausted molasses in the year 1920-21, in the different provinces.

Pinar del Rio	38·70
Habana	32·88
Matanzas	34·87
Santa Clara	34·11
Camaguey	34·35
Oriente	34·39

Composition of Argentine Molasses.—Cross* cites the analyses of exhausted molasses of a number of factories in Tucuman in 1915, to be as below :—

	1	2	3	4	5	6	7	8
Brix	76·2	80·2	82·8	83·9	86·5	80·4	83·1	77·6
Polarization ..	25·0	38·0	34·4	31·2	28·4	27·7	26·8	30·3
Apparent Purity ..	32·8	47·38	41·55	36·71	32·83	34·46	32·25	39·04
Solids	73·03	76·60	76·50	78·78	80·40	76·80	78·20	72·70
Sucrose	31·90	43·37	39·50	37·40	33·00	36·40	34·20	34·60
True purity ..	43·68	56·62	51·63	47·47	41·04	47·39	43·73	47·58
Reducing sugar ..	20·1	15·1	12·5	16·1	21·9	22·45	16·9	11·3
Nitrogen	0·54	0·59	0·76	0·85	0·60	0·55	0·74	0·70
Ash	7·44	6·82	10·89	9·68	8·85	6·94	9·54	10·06
Silica	0·25	0·21	0·46	0·40	0·35	0·26	0·31	0·30
Iron oxide and alumina ..	0·12	0·14	0·22	0·21	0·18	0·11	0·16	0·13
Lime	0·71	0·46	0·72	0·82	0·97	0·38	0·95	0·64
Magnesia	0·19	0·18	0·22	0·19	0·21	0·19	0·24	0·23
Potash	3·34	2·96	5·18	3·87	4·11	3·26	4·05	4·93
Soda	0·14	0·17	0·26	0·40	0·16	0·18	0·17	0·26
Phosphoric acid ..	0·13	0·17	0·14	0·10	0·15	0·13	0·15	0·21
Chlorine	0·26	0·69	1·48	0·78	0·26	0·17	0·30	1·01
Sulphuric acid ..	0·64	0·80	1·22	1·04	0·81	0·66	1·26	1·21
Carbonic acid in ash	1·32	0·70	0·90	1·09	1·40	1·15	1·32	0·97

Composition of Philippine Molasses.—Westley† gives the following data regarding Philippine molasses :—

* "Revista Industrial y Agricola de Tucuman," 1922, 99.
† "Int. Sugar Journal," 1924, 100.

	1	2	3	4	5	6	7	Average of 13 factories.
Brix ..	85·00	87·29	91·70	85·33	88·31	87·54	86·00	88·29
Apparent Purity ..	32·10	32·09	31·61	33·29	—	31·86	38·80	32·40

These analyses which agree fairly mutually for the various countries give rise to the following observations.

Degrees Brix.—As was said in the case of massecuites and first molasses, the degree Brix of the exhausted molasses is always higher than the per cent. of actual dry substance, due to the fact that many of the impurities which accompany the sucrose possess a higher specific gravity than the latter in solutions of equal concentrations. The specific gravity tables corresponding to degrees Brix are calculated for pure sucrose, so that the presence of such impurities causes the degrees Brix (calculated from the specific gravity) to differ from the actual dry substance. The importance of these deviations is shown in the following table, in which the actual concentration, specific gravity, and the corresponding degrees Brix of various substances are brought together :—

Substances.	Concentration.	Specific Grav. $\frac{17·5}{17·5}$	Degrees Brix.
Sucrose	10	1·04014	10·0
Glucose	10	1·04013	10·0
Fructose	10	1·03990	10·0
Sodium chloride	10	1·07312	17·7
Potassium chloride	10	1·06518	15·9
Calcium chloride	10	1·08647	20·7
Potassium sulphate	10	1·08235	19·8
Potassium acetate	10	1·0496	12·3
Sodium acetate	10	1·0538	13·25
Calcium acetate	10	1·0492	12·2
Potassium tartrate	10	1·0650	15·9
Albumin	10	1·0261	6·6
Dunder from cane sugar molasses	9·36	1·04356	10·8
Sodium glucinate*	10·13	1·05494	13·5
Potassium ,, ...	9·74	1·05358	13·2
Calcium ,, ...	9·92	1·04769	11·8

* The glucinates were prepared by boiling a solution of invert sugar with lime till the complete decomposition of the reducing sugar had occurred and their conversion into the potash and soda salt by means of potassium and sodium carbonate.

With a few exceptions, all the substances mentioned here, when in solution, show a higher specific gravity than sucrose and the two other sugars.

Leaving out of question the possible interaction or combination between the different constituents, and assuming that each of these retains its own specific gravity, a solution of the following composition would possess an actual percentage of 70 per cent. dry substance and yet show a density corresponding to 77·7 degrees Brix :—

		Brix.
Sucrose	40 =	40
Glucose	10 =	10
Fructose	10 =	10
Sodium chloride	10 =	17·7
	70	77·7

All cane molasses contain salts, and since the degree Brix of solutions of both organic and inorganic salts is in excess of their actual content of dry substance, it is very probable that the degree Brix of the molasses is too high.

The analyses of a number of artificial mixtures proved that when a solution only contains sucrose and reducing sugars, the sum of the degrees Brix and the water does not deviate much from 100, but falls below 100 as the proportion of reducing sugar increases.

When salts are also present, the sum of the degrees Brix and the water rises above 100.

No.	Brix. 17·5 C°.	Sucrose.	Reducing Sugar.	Ash.	Water.	Sum of Brix + Water
1	68·1	68·12	0·0	0·00	31·9	100·02
2	69·2	66·8	2·4	0·02	30·7	99·9
3	71·7	57·5	11·0	0·03	27·81	99·51
4	74·7	51·6	21·7	0·03	24·85	99·55
5	74·8	43·8	29·4	0·03	24·7	99·50
6	77·9	43·6	33·3	0·03	21·27	99·17
7	84·3	37·4	39·2	0·03	15·0	99·30
8	82·4	1·8	75·0	0·06	16·6	99·0
9	68·6	—	—	15·13	36·5	105·1
10	81·8	62·6	1·92	12·42	26·93	108·73
11	74·9	58·8	2·10	8·41	29·60	104·5
12	80·5	54·5	11·90	8·46	25·07	105·57
13	78·5	47·9	15·2	6·75	26·00	104·5
14	79·6	46·8	21·3	5·80	24·23	103·83
15	84·2	41·4	31·5	6·45	20·40	104·60
16	83·5	34·4	38·4	4·46	17·83	101·33

Although the salts of *inorganic* acids are fully represented in the ash, those of *organic* acids leave carbonates after incineration, the weight of which is only a part of that of the original salt. In the case of salts containing much organic acid, the amount of ash is smaller than if the majority of the salts had been of inorganic acids. For this reason, and also because every salt in solution has its own relation between specific gravity and dry substance, no fixed factor can be found for converting the degree Brix to the actual dry substance in the molasses. Former researches gave an average factor of 0·6 to 0·7, so that for an indirect determination of solid matter in an exhausted molasses, the figure for ash was multiplied by 0·6 to 0·7 and the product subtracted from the degree Brix at 17·5° C. in order to get the actual dry substance. In the analyses recorded in the afore-mentioned tables, the author found an average factor of 0·63, with a maximum of 0·89 and a minimum of 0·47, and only about 20 per cent. of the molasses showed larger deviations from the average than 0·10.

Polarization.—It has already been observed that the direct polarization does not yield the true percentage of sugar, because clarification by means of basic lead acetate precipitates part of the fructose and therefore causes the polariscope reading to be somewhat high. But, as has already been remarked, the lack of a suitable clarifying agent, not possessing this or similar inconveniences, compels us to accept this slight error. From the difference between direct polarization and polarization by Clerget's method, we can compute the quantity of other optically active substances in the molasses. Clarification with the lead reagent named eliminates all the active acids, leaving only the active sugars, and as the sucrose is already accounted for by polarization by Clerget's method, the difference between the total polarization and that of the sucrose is due to reducing sugars.

The rotatory power of the reducing sugar, expressed in degrees Ventzke, calculated from the data mentioned on page 304, is for factories using the carbonatation process between —8 and —10·2; for those using the common defecation process between —13 and —15; and for those using sulphitation, of the limed juice between —12 and —13.

One sees from these figures that the processes of manufacture in use now, where the juice is only very sparingly exposed to alkaline reactions, the transformation of the reducing sugar, as referred to on page 35, is no longer a considerable one, while, at the same time, the figures show that inversion of sucrose does not play any important rôle owing to the rapid course of manufacture.

Quotient of Purity.—The Brix being higher than the actual per cent. of dry substance, and the polarization being lower than the actual per cent. of sucrose, we see that two errors co-operate to make the true purity much higher than the apparent purity. The former is obtained by dividing 100 × the per cent. sucrose by the dry substance; and the latter by dividing 100 × the polarization by the degrees Brix.

Ash.—The amount of ash in cane molasses is fairly constant so far as the ash proper is concerned, apart from the sand, clay and other impurities contaminating the molasses and not belonging to the real molasses. The percentage of ash ranges between 7 and 10 per cent., with only slight deviations above and below these limits.

Gummy Matter.—Under the heading of "gummy matter" are included all organic substances precipitated by alcohol from an acid solution of the molasses. In many English and American textbooks the word "gums" is used for organic substance precipitated by basic lead acetate, which is quite another thing.

A sample of the precipitate obtained from an acidified molasses solution contained 41·8 per cent. of ash. The amount of pentosans, determined by Tollens' method, was 9·13 parts on 100 parts of the organic matter in the precipitate, while the estimation of galactan by the mucic acid method gave a content of 7·06 per cent. of galactan on 100 parts of organic matter. A second sample was found to contain 7·1 per cent. of pentosan on 100 parts of organic matter.

Hazewinkel and Langguth Steuerwald showed that all pentosan present in the syrup could be found again in the molasses, and therefore did not undergo any change or transformation during the course of manufacture. This, however, was not the case with the percentage of pentosan on 100 parts of total gummy matter. Whereas gum from syrup contained only 9·7 per cent. of pentosan, the gum from molasses showed a percentage of 23·6. One or other of the gum constituents must have been broken up or eliminated from the solution, while careful analysis of a number of specimens of gums extracted from a variety of molasses indicated a widely varying constitution.

The gummy matter in the molasses therefore consists neither exclusively of pectin, nor of gums dissolved from the fibre, nor yet of slime, but of a mixture of the three in varying proportions.

Organic Non-Sugar.—The difference between total dry substance and the sum of sucrose, reducing sugar, and ash is entered in analyses as "organic non-sugar." This includes the gum, and the remainder is divided between organic acids, nitrogenous bodies, caramel, and other products of decomposition of sucrose, glucose, and fructose.

The figure for "organic non-sugar" is rather considerable, and as yet no further subdivision has been made. The carbonic acid in the ash is an approximate measure of the amount of organic acids, but does not yield an absolute value because we neither know what these acids are, nor their combining weights. The acids mentioned in Chapter I possess the following combining weights: malic acid = 67, succinic acid = 59, and acetic acid = 60, or on an average = 60. On decomposing glucose by lime, calcium salts of organic acids are formed containing 21·1 and 18 per cent. of calcium oxide

respectively, or, on an average, 20 per cent. of calcium oxide, equal to 14·3 per cent. of calcium. The combining weight of those acids is therefore 121, for

$$\frac{100 \times 20}{(121 - 1) \times 20} = 14{\cdot}3.$$

These acids were formed by decomposition with large amounts of lime, and perhaps those formed with smaller quantities possess a still higher combining weight; but, as not much of these acids is formed on defecation, we obtain a high figure when we assume the average combining weight of all the organic acids to be 100. On incineration only 22 parts of carbonic acid are left, so we may roughly estimate the total amount of organic acid in the molasses to be about 4½ times that of the carbonic acid in the ash. As the figure for carbonic acid is already accounted for, the figure for caramel, nitrogenous bodies and products of decomposition may be found by subtracting 3½ times the carbonic acid from the figure for organic non-sugar, minus the gum.

Nitrogen.—The nitrogen content of cane molasses does not exceed 0·2 per cent., so that the nitrogenous bodies form not more than 1 per cent. of the weight of molasses.

Undetermined.—Calculated in this way, we find a balance of about 5 per cent. left for caramel and the other products of decomposition, though we must not forget that all errors of analysis are included in that figure, the accuracy of which is not above suspicion.

We quote from a great many analyses the following two in order to show how the amount of undetermined matter may be calculated:—

I—Organic non-sugar		12·71
Gummy matter	2·39	
Nitrogenous bodies	1·00	
Organic acids (= 3½ CO_2), ..	3·81	
	——	7·20
Balance		5·51
II—Organic non-sugar		11·16
Gummy matter	1·51	
Nitrogenous bodies	1·00	
Organic acids (= 3½ CO_2) ..	4·16	
	——	6·67
Balance		4·49

Although it is as yet not feasible to determine either the absolute amount or the real chemical character of the undetermined matter, yet it does undoubtedly exist.

Hazewinkel* showed in an extensive examination that actually the balance remaining unaccounted for on analysing cane molasses is not caused by analytical errors of some sort or other, but is really due to some organic body.

From an investigation, made by the author in collaboration with Langguth Steuerwald,† it was found that when a neutral solution of invert sugar was heated for a considerable time, it assumed a dark coloration and an empyreumatic odour, while at the same time the reducing power diminished. Addition of salts of organic or inorganic acids accelerates and intensifies the reaction. The colour becomes dark-brown, the amount of solids decreases by the escape of gaseous decomposition products, which are characterized by the empyreumatic odour that they diffuse, and the reducing power is greatly impaired. Polarization however, in so far as this can be ascertained in the dark-coloured liquor, does not show great changes. When the salts have been chosen in such a way that they are not precipitated by lead acetate, the solution remains clear after addition of both neutral and basic lead acetate, before it has been heated; but after that operation we observe precipitation of lead compounds,

Duration of heating, Hours.	Invert Sugar.	Salts.	Total of Matter determined.	Degree Brix.	Gravity Solids.	Undetermined Organic Matter.
I.						
0	7·51	8·58	16·10	19·0	16·10	0
20	7·45	—	—	18·7	15·90	—
40	7·13	—	—	18·7	15·90	—
64	6·65	8·57	15·22	18·6	15·81	0·59
70	5·96	8·55	14·51	17·9	15·21	1·14
80	5·59	8·57	14·16	18·0	15·30	1·94
II.						
0	8·21	4·33 4·08	16·62	24·2	16·62	0
20	8·30	4·32 4·17	16·79	24·2	16·62	—0·17
35	7·89	4·40 4·08	16·37	24·2	16·62	0·25
55	7·68	4·34 4·18	16·10	24·15	16·60	0·50
65	7·25	4·30 4·16	15·71	24·1	16·58	1·07

* "Archief voor de Java Suikerindustrie," 1908, 492.
† *Ibid.*, 1910, 147.

which proves that the invert sugar has been broken up with the formation of bodies giving precipitates with the lead reagents.

When analysing, by methods used for the molasses analysis, a solution of invert sugar and salts which had been heated over a long period, a balance was found between the total solids and the total amount of determined constituents. This is shown in the tables on page 312.

Table I. has reference to a solution of 7·51 per cent. of invert sugar, and 8·58 per cent. potassium acetate; and Table II. to a solution of 8·21 per cent. of invert sugar, 4·33 per cent. potassium acetate, and 4·08 per cent. potassium sulphate. The mixtures were heated at 100° C. over a long period and analysed.

At the outset we observe no undetermined matter at all, but gradually a deficit between total and determined solids creeps in, and we see the same phenomenon as appears in the analysis of a cane molasses.

It is therefore very probable that during the course of manufacture part of the reducing sugar has been broken up into other bodies which escape detection and determination by the usual analytical methods. These products of decomposition are combustible solids, and are therefore not determined by the estimation of mixture or ash. They have a very feeble rotative power, which by direct polarization exerts an insignificant influence on the estimation, and none at all on the Clerget sucrose determination. Their reducing power is much smaller than that of the reducing sugar, and since the percentage of the latter is only derived from the weight of cuprous oxide, separated on analysis, it is clear that their presence merely serves to raise the amount of reducing sugar in the analysis sheet slightly above the real percentage, while the great bulk of the products of decomposition remains unnoticed. They are soluble in acidified alcohol; and are, accordingly, not found among the gummy matter, so that in no item of the analysis sheet is either their presence or their quantity recorded. Under such circumstances we consider ourselves justified in concluding that among the undetermined matter still unaccounted for in the analysis of molasses, the decomposition products of reducing sugar are strongly represented.

This conclusion is further corroborated from the fact that after the data of the Mutual Control of Java Sugar Factories* the glucose coefficient of juices constantly sinks during manufacture, thus yielding a proof of the disappearance of reducing sugar.

Further, we know that every effort to establish a kind of reducing sugar account in sugar factories has failed to give a satisfactory balance, owing to the circumstance that in every instance the final amount of this constituent was found to be considerably under the initial one.

* "Archief voor de Java Suikerindustrie," 1909, 488,

III.—Loss of Sucrose in Molasses

Sucrose Content of Exhausted Molasses.—We have shown in the foregoing pages that the sucrose content of the exhausted cane molasses is a fairly constant one, fluctuating around the figure of 30 per cent. within rather restricted limits. The only exception to this general rule is supplied by molasses from juices possessing a high ash content together with a low amount of reducing sugar, in which case the sucrose content will be high. On the other hand, a high percentage of reducing sugar and a low one of ash will produce molasses having a low sucrose content.

Quotient of Purity.—A few examples of exhausted molasses of high purity follow, and in every case the ash content is high and that of reducing sugars low, causing a high sucrose content.

Analysis.	1	2	3	4
Brix	90·1	85·7	83·5	89·7
Polarization	37·4	38·0	35·2	35·8
Reducing sugar	11·9	15·4	14·5	18·87
Apparent purity	41·5	44·3	42·6	40·0
Ash	13·26	9·33	9·3	10·86
Potash in the ash	7·64	3·5	3·84	5·76
Reducing sugar : ash ..	0·90	1·6	1·6	1·7

On some estates the content of reducing sugar is always low, so that a rather rich molasses may yet be considered exhausted.

Juice from an Egyptian estate contained a great deal of sodium sulphate, and gave the following analytical results :—

Brix	18·2
Polarization	14·6
Reducing sugar	0·51
Ash	0·98
Reducing sugar : Ash	0·52
Apparent purity	80·25

When such juice is worked up without being mixed with other juice, the purity of the exhausted molasses will not fall below 50°, and is similar in character to beet molasses.

Ratio of Reducing Sugars to Salts.—This, however, is an extreme case, but when the ratio of reducing sugar to ash amounts to 1·5, it will generally be impossible to reduce the purity of the molasses to 30°. The limit of exhaustion lies well above that figure, so that further concentration only renders the molasses more viscous and stiff without causing any further yield of sugar. On this account, it is impossible to reduce the purity of the molasses down to 30° on some estates; in many cases the juice of one cane variety can be reduced to a molasses of 30° purity, while that of another variety yields molasses of higher purity even on the same estate. The figures on pages 101 and 102, show that one variety assimilates more salts from the soil than another, so it is evident that the nature of some cane varieties may prevent the reduction of purity of the exhausted molasses.

An unfavourable ratio of reducing sugar to ash is the only cause which lies beyond the control of the manufacturer; the other causes (to be enumerated below) may be got rid of, so that every juice possessing a favourable ratio of reducing sugar to ash should yield exhausted molasses of 30° apparent purity or under.

Excessive Evaporation.—One reason is excessive evaporation, owing to which the cooling mass becomes semi-solid with the formation of a very fine grain, which cannot be recovered but passes through the centrifugal linings along with the molasses. When such a massecuite is cooled in rotating crystallizers, it can be improved by diluting it to a density of 96° Brix with diluted molasses before curing.* When cooled in fixed tanks, an over-concentrated massecuite may be improved by covering it with water to a depth of about a foot, allowing it to stand for a month, after which time the supernatant layer of water is removed, and the massecuite cured.†

As prevention is better than cure, massecuites boiled to grain should be diluted at once, and those boiled smooth should not be concentrated too far.

Too Rapid Cooling when Smooth-Boiled.—Another reason why massecuites boiled to string-proof are difficult to cure is that they are cooled too fast, crystals being formed during their transport to the crystallization tanks,‡ so that an excessive quantity of grain is formed which does not grow, and cannot be separated from the molasses. This may be prevented by keeping the temperature of the massecuite high when it leaves the pan, and by transferring it quickly to the crystallizing tanks.

These tanks should be neither too small (in order to prevent too rapid cooling) nor too large, but should have a capacity equal to one or two strikes of the pan. If several strikes of the pan are required to fill the tank, the crystallization of the first strike will be disturbed by each subsequent addition of hot massecuite, which is detrimental to crystallization.

* See page 243. † Compare page 262.
‡ See page 259.

High Gum Content.—A high percentage of gums also renders the molasses thick, so that they cannot be properly concentrated. In some cases, the molasses contain so much gummy matter that unless a good deal of water is left in the massecuite the latter sets hard on cooling. If evaporation be carried too far, crystallization is interfered with, and the crystals formed cannot be separated from the mother-liquor and are consequently lost. If, on the contrary, sufficient water is left in the molasses to keep them fluid, too much sucrose remains dissolved, so that in this case, also, the loss of sucrose is excessive.

Hazewinkel and Langguth Steuerwald* mention an instance of so high a gum content of a syrup that this liquid coagulated to a solid block after being acidified with acetic acid and heated in the laboratory. Evaporation was greatly interfered with owing to the presence of that body.

Canes grown in some districts contain much gummy matter, while some varieties of cane, *e.g.*, the Uba, are exceptionally rich in this substance. When this is known, a much larger quantity of lime may be used in clarification and the excess neutralized with sulphurous acid. The heavier precipitate drags the gummy matter down into the mud, the juice is rendered limpid, and the molasses can be exhausted to the usual low purity.

Repeated Boiling of the same Molasses.—When the same molasses is repeatedly returned into the massecuites it becomes sticky and difficult to separate. The following analyses of a fresh molasses and of one that has been a long time in circulation, both from the same factory, may explain the reason :

	Fresh Molasses.	Molasses a long Time in Circulation.
Brix	87·62	79·55
Dry substance	83·51	76·47
Polarization	36·00	39·80
Apparent purity	41·09	50·03
True purity	43·11	52·05
Reducing sugar	27·00	20·80
Ash	5·22	4·74
Soluble ash	2·25	2·01
Insoluble ash	2·97	2·73
Silica	0·16	0·19
Iron oxide and alumina	0·68	0·48
Lime	0·84	0·80
Carbonic acid in the ash	0·75	0·59
Gummy matter	1·94	1·90

The molasses which has been in circulation a long time contains more

* "Archief voor de Java Suikerindustrie," 1911, 313.

water and sucrose than fresh molasses, due to the addition of "purging water" and solution of the sugar, which in itself is an indication that the molasses were gummy, otherwise purging would not have been necessary. No increase of gums could be detected by analysis, but a much larger amount of dirt settled from the old molasses when solutions of the two samples were kept for some time.

100 grms. of each molasses were dissolved in 100 grms. of water and the solutions allowed to subside in conical glasses. When the deposits ceased to increase in volume they were measured; that yielded by the fresh molasses was 2·4 c.c., while 11·5 c.c. or five times as much was obtained in the other case. The deposit consisted of a slimy substance, containing fragments of incrustations and of decomposition products. It is true that a portion of molasses is withdrawn daily from the course of manufacture and replaced by fresh molasses, but the dirt seems to accumulate in the circulating molasses and renders them so much more sticky than fresh molasses. The remedy is, of course, to reduce the quantity of molasses in circulation* and thus avoid the repeated re-boilings.

Deficient Clarification.—If insufficient attention is paid to the subsiding of the syrup, or to the steaming and skimming of the molasses, a considerable quantity of a gelatinous substance remains suspended in the molasses, rendering the same very sticky and difficult to separate from the crystals.

The composition of such molasses follows here:—

Brix	85·1
Polarization	30·8
Purity	36·2
Reducing sugar	20·4
Ash	9·1
Soluble ash	6·09
Insoluble ash	3·01
Gummy matter	2·08

The figures are normal, and do not afford any clue as to the inferior quality of the molasses. It was, however, very dull in appearance and viscous, while a considerable quantity of dirt deposited from a solution in water. A large quantity of the molasses was diluted with water, and after a few days' rest the supernatant clear solution was decanted off and analysed. The residual dirty liquid was brought to the same density and also analysed.

In the subsided dirty portion we find an increase in silica and organic lime salts, the other constituents being present in much the same proportion in the two liquids. The viscosity, which is here the principal factor, is much greater in the dirty liquid, the rate of flow being four times that of the clear liquid, and it is therefore not astonishing that notwithstanding a normal chemical composition such molasses are very difficult to cure.

* See page 221.

	Clear Portion.	Dirty Portion.
Brix	65·7	66·8
Dry substance	60·9	60·8
Ash	6·9	8·6
Soluble ash	4·45	4·5
Insoluble ash	2·45	4·12
Alkalinity of the ash as CaO	4·18	5·51
Silica	0·23	0·5
Lime	0·84	1·5
Magnesia	0·15	0·16
Sulphuric acid	0·15	0·15
Iron oxide and alumina	0·42	0·72
Gummy matter	2·40	2·21
Viscosity (rate of flow)	11 min. 15 sec.	47 min.

It follows that good clarification and subsiding of the juice, combined with careful clarification of the syrup and first molasses, can eliminate these gummy and sticky substances, so that such inferior final molasses do not occur.

Viscosity.—In discussions regarding the yield obtained from low-grade massecuites, especially when deficient, too much importance is attached to the viscosity of the molasses, and in many a case this property is made responsible for every inferior yield of sugar from massecuite. A high quotient of purity of the exhausted molasses is explained away on the assumption that the molasses is so viscous that a high sucrose content cannot be avoided. The author does not believe that the viscosity of juices and syrups is sufficient to hinder crystallization, but rather that careless treatment can cause any molasses to become viscous.

It is a well-known fact that the viscosity of pure sucrose solutions increases with the concentration and decreases as the temperature rises, and that this latter decrease is stronger than the former increase.

Influence of Temperature on Viscosity.—The same thing is also true of molasses, and the table given below shows how even a slight difference in density of a solution of a given molasses causes a considerable difference in the rate of flow of 200 c.c. from an Engler's viscometer.

Cane molasses: Brix, 83·2; polarization, 25·0; reducing sugar, 30·8; ash, 6·13; diluted to the Brix degrees mentioned.

Brix.		Mins.	Secs.
72·62	Rate of flow	22	46
71·15	,,	20	44
71·07	,,	20	2
70·82	,,	19	0
70·72	,,	18	35
65·30	,,	6	55

Cane molasses: Brix, 80·2; polarization, 31·2; reducing sugar, 26·9; ash, 8·34; diluted to the Brix degrees mentioned.

Brix.				Mins.	Secs.
70·12	..	Rate of flow	..	26	44
69·95	..	,,	..	26	11
69·63	..	,,	..	25	45

The undiluted molasses was far too stiff to flow from the viscometer, and the experiments were for that reason continued with concentrations of 65-70 per cent. dry substance.

A number of molasses were diluted to about 65 per cent. dry substance, and the rate of flow of those solutions and of sucrose solutions was determined at the temperature of 28° C.

VISCOSITY OF VARIOUS LIQUIDS HAVING THE SAME DENSITY.

	Brix.	Dry Substance	Polarization.	Reducing Sugar.	Ash.	Gummy Matter.	Rate of Flow.	
							Mins.	Secs.
Molasses 1 ..	70·3	65·0	23·4	26·9	7·60	1·87	16	30
,, 2 ..	69·9	65·06	26·0	30·9	6·77	1·90	20	20
,, 3 ..	69·9	64·96	25·6	24·4	7·38	0·48	22	25
,, 4 ..	69·0	65·24	27·3	23·5	5·37	1·80	24	11
,, 5 ..	70·7	65·08	20·52	25·5	7·18	1·85	18	35
Sucrose ..	65·0	65·9	65·0	—	—	—	8	57
,, ..	70·3	70·3	70·3	—	—	—	24	10

Viscosity of Constituents of Molasses.—These results show that for the same dry substance content, the viscosity of different molasses does not vary much, but is higher than that of sucrose solutions having the same dry substance content. But the non-saccharine impurities of the molasses evidently possess a higher viscosity than sucrose; so in order to ascertain to which constituent this property was chiefly due, the author determined the rate of flow of a number of substances in solution of 65 per cent. at 28° C.

	Mins.	Secs.
Sucrose	8	15
Glucose	6	33
Fructose	4	40
Sucrose and Glucose (equal weights)	7	39
Concentrated " dunder " from a cane sugar molasses distillery	43	10

The substance which accounts for the increased viscosity is therefore one or other of the complex non-saccharine substances, and not the reducing sugar.

The analysis of the concentrated " dunder," or residue left behind in the still after the distillation of fermented cane molasses, was as follows :—

	Per cent.
Dry substance	61·8
Reducing matter	3·9
Ash	15·13
Soluble ash	3·40
Insoluble ash	11·73
Carbonic acid in the ash	2·88

It therefore contained some unfermented glucose as well as caramel, decomposition products of sucrose and glucose, and salts. Half of these salts were combined with organic and the other half with inorganic acids.

Viscosity increased by Impurities.—10 per cent. of the different substances were then added to a 65 per cent. solution of equal parts of sucrose and glucose, and the viscosity of the mixtures determined at 28° C.

Substances of which 10 per cent. is added to the 65 per cent. Sucrose and Glucose Solution.	Degrees Brix of the Solution.	Rate of Flow. Mins.	Secs.
Sodium carbonate	76·25	30	23
Sodium chloride	73·9	11	40
Ammonium chloride	65·3	5	0
Calcium chloride	76·8	27	5
Magnesium chloride	67·7	8	27
Barium chloride	77·0	9	26
Potassium oxalate	76·3	11	50
Sodium glucinate*	70·61	16	15
Potassium glucinate*	69·85	10	6
Calcium glucinate*	69·45	22	0
Dry substance from " dunder "	68·45	15	40
Gum	66·9	73	0
Sucrose and glucose	67·8	46	0

As stated by Claassen,† sodium salts increase the viscosity more than potassium salts ; and again, calcium salts more than sodium salts, as is very clearly shown by the organic salts used here.

* The glucinates were prepared by boiling a solution of invert sugar with lime till the complete decomposition of the reducing sugar had occurred and their conversion into the potash and soda salt by means of potassium and sodium carbonate.

† " Zeitschr. Rübenzuckerind.," 1898, 535.

With the exception of gum (which, properly speaking, does not belong to the class of constituents examined), sugar is the most viscous. Of the different substances added to a 65 per cent. solution of sucrose and glucose, the viscosity was greatest when the addition consisted of sucrose and glucose.

Density the Greatest Factor.—This seems strange in view of the fact that pure syrups (in which sucrose is chiefly represented) are much less viscous than impure ones, in which the other constituents are accumulated. But this apparent contradiction is explained when one bears in mind that a pure sucrose solution cannot contain more than 68 per cent. dry substance, while impure molasses can contain even 84 per cent. and over. The greater viscosity of impure molasses is, therefore, due not so much to certain substances having a high viscosity, but to a higher density than a saturated sucrose solution can attain.

As the content of dissolved substances greatly influences the viscosity, it is necessary to examine the viscosity of molasses in an undiluted state, since a comparison of the viscosity of molasses which are diluted to the same dry substance content affords no clue to the viscosity of the undiluted molasses in the practice of sugar manufacture.

As the minute crystals present in factory molasses interfere with the viscosity test, the author had recourse to the series of artificial molasses, which had rendered good service in his studies on the formation of molasses.*

A number of mixtures were prepared from sucrose, invert sugar, water, potassium acetate, and calcium chloride (in which the amounts of potash and lime were identical), and allowed to stand until all the sucrose which was liable to crystallize had done so. The saturated mother-liquors were poured off from the crystals, analysed, and tested in an Engler viscometer at 28° C. The figures express the rate of flow of 200 c. c. of the liquids.

The experiments showed that viscosity increases with the amount of dry substance, but not in direct proportion, as is shown by the table underneath.

Dry Substance.	Rate of Flow.			Dry Substance.	Rate of Flow.	
	Hours	Mins.	Secs.		Hours.	Mins.
68·1	0	14	23	75·3	1	21
69·3	0	16	30	75·77	7	1
70·4	0	33	0	78·73	4	2
72·19	0	36	0	79·6	33	15
73·07	3	11	0	80·87	10	6
74·0	2	13	0	82·5	22	11
74·93	2	45	0	83·17	66	4
75·15	1	19	0	85·0	118	0

* "Int.-Sugar Journal," 1908, 285.

It is a peculiar fact that for a given dry substance content the saturated solutions containing salts have a greater viscosity than those which only contain sugars, while in the table for unsaturated solutions given a few pages back* the solution free from salt was more viscous than one in which the amount of salt was replaced by sugars. It appears that, at high concentrations, the viscosity of the complex of sugars and salts differs much from that at low concentrations. At a concentration of 65 per cent. dry substance, the non-sugar in the molasses is much more viscous than that of a sucrose solution of the same concentration, whereas when 10 per cent. of dry non-sugar is added to a 65 per cent. sucrose-glucose solution, a much less viscous mixture results than when 10 per cent. of the same mixture of sucrose and glucose is dissolved. This is another warning against judging the viscosity of the molasses from their behaviour in a diluted state, instead of determining their viscosity in their original, saturated, but crystal-free condition.

In the case of artificial molasses, some regularity was detected in the increase of viscosity with an increase of dry substance, but such regularity is not so obvious in practical working. There, we sometimes encounter very high viscosities combined with comparatively small dry-substance contents, and this is due to the presence of other bodies in the molasses than were made use of in the artificial liquids.

Influence of Gums and Suspended Matter.—The most prominent among these are gummy substances, decomposition products of reducing sugar, and also perhaps silica. It is a well-known fact that cane gum and pectin can sometimes occur in exhausted molasses to an extent of 5 or 6 per cent., and it is very evident that such bodies might increase the natural viscosity of molasses to a considerable extent.

The decomposition products of reducing sugar also affect the viscosity of syrups and molasses. The quantity of silica found in molasses (not exceeding 0·20-0·30 per cent.) is too small to be capable of increasing its viscosity.

To ascertain the influence of gum, so much cane gum was mixed with a sucrose solution of 66·5 per cent. that its content amounted to 3·86 per cent. on 100 parts of the mixture. Dry glucose was added to a second portion of that solution so that the dry substance content was equal to that of the gum-containing one. The two liquids then contained the same amount of dry substance, sucrose, ash, etc., but as in one case 3·86 per cent. of the glucose was replaced by a similar weight of gum, any difference in viscosity was exclusively due to the gum. The rate of flow of the gum-containing molasses was 31 minutes and that of the other 10 minutes 40 seconds, showing the great influence of gum on the viscosity. An addition of 0·50 per cent. of dissolved silica had no influence, while the addition of a heated calcium glucinate solution could not be ascertained because that substance separated black, greasy flocks, which choked up the apparatus and thus prevented the determination.

* See page 320.

A further cause of viscosity of molasses is due to the presence of finely divided suspended matter. This was proved by the following experiments, in which three portions of similar sucrose solutions were triturated with 5 per cent. of finely pulverized sugar, 2 per cent. of fine river silt, and 2 per cent. of washed and dried filter-press mud respectively. The rate of flow of the four liquids was as follows :—

	Mins.	Secs.
Sucrose solution without addition	13	10
,, ,, with 5 per cent. pulverized sugar ..	21	0
,, ,, ,, 2 per cent. river silt	18	35
,, ,, ,, 2 per cent. dried mud	21	0

This shows how finely divided insoluble substances, such as often occur in molasses, greatly influence the viscosity and can cause large differences in that property when the dissolved constituents are normal.

Apart from these differences, due to suspended matter, we are justified in declaring that the viscosity increases with the dry substance content, and that a molasses, which has been over-concentrated, has so increased in viscosity that much labour and trouble are required to separate it from the crystals during curing operations.

Influence of Temperature.—One factor has so far remained unconsidered, viz., temperature. All the determinations of viscosity recorded here are made at 28° C. and relate exclusively to that temperature. As Claassen has proved, temperature has a great influence on viscosity, especially between 15° and 40° C. In order to investigate this influence three molasses (free from minute sugar crystals) were tested at different temperatures with these results :—

	I.	II.	III.
Dry substance ..	69·73	72·73	80·50
Polarization ..	35·4	28·0	22·4
Gum	5·94	1·72	1·35

	Hrs.	Mins.	Secs.	Hrs.	Mins.	Secs.	Hrs.	Mins.
Rate of flow 28° C.	3	50	0	3	16	0	27	0
,, 35° C.	1	42	0	1	47	0	19	40
,, 40° C.	0	44	10	0	53	10	6	43
,, 45° C.	0	29	10	0	36	45	4	13
,, 50° C.	0	20	0	0	25	10	2	36
,, 60° C.	0	12	40	0	11	40	1	30

We see then the great influence of temperature, especially degrees under 45° C. ; above this point, a decrease in temperature is not so evidently accompanied by an increase in viscosity.

These researches have taught us to regard concentration and temperature

as the principal factors of viscosity; and as secondary ones, the content of gum and the decomposition products of reducing sugar, also finely divided impurities, and fine grains of sugar. It is therefore desirable from a manufacturer's point of view that the molasses be as little viscous as possible in order to enable it to be easily separated from the crystals without much washing in the centrifugals. This may be accomplished by taking care not to push the concentration of the last massecuites too far; or, in case they have become too much concentrated, to dilute them with dilute molasses in a judicious manner.

A water content of 10 per cent. (equivalent to 96° Brix) for massecuites (boiled to grain) after dilution, and a water content of 18 per cent. (equivalent to 88° Brix) in the resulting molasses are the most favourable.

The cooling must not be pushed further than 45° C., and should remain preferably a couple of degrees over that figure, as at that temperature crystallization is finished, and below that the viscosity increases so considerably that cooling down to that point only causes trouble without any compensating advantage.

The increase of viscosity occasioned by secondary circumstances, such as gummy matter, insoluble substances or false grain, can, of course, be avoided by well-conducted clarification, subsiding, filtering, boiling, and cooling processes. Thus we see that a high viscosity is by no means a property of some particular juice or syrup; but, on the contrary, it is within the power of any sugar-boiler to keep his molasses fluid and capable of easy separation from the sugar crystals.

Dextro-rotating Impurities in Molasses.—A high quotient of purity of exhausted molasses has been attributed to the presence of a dextro-rotating substance (other than sucrose) in the molasses. In all such cases, a careful examination has failed to detect such substances; on the contrary, the direct polarization was always lower than the true sucrose content, owing to the levo-rotating invert sugar present in all cane products.

The absence of dextro-rotating impurities in molasses may therefore be considered as settled, and from the foregoing pages it will be seen that there is no reason why the apparent purity of a fully exhausted cane molasses should be much above 30°, except in the case of an unfavourable ratio between reducing sugars and ash. Leaving out of consideration cases where this ratio is 1·5 and thereabouts, we shall only here consider the normal case of an exhausted molasses of 30° purity, or 27° direct polarization.

Quantity of Molasses.—The quantity of molasses and the loss of sugar therein depend, therefore, on the amount of non-sugar in the juice, since an impure juice will yield more molasses than a pure one, and we can easily calculate how much molasses should be obtained on 100 parts of sugar for

each degree of purity of the clarified juice. We assume the polarization of the sugar to be 96·5 and calculate the quantity of molasses of 90° Brix and 30° purity obtained on every 100 parts of such sugar for different purities of the juice. No allowance is made for the loss of sucrose by inversion and spilling. The figures between the limits of 77° and 92·9° purity are as follows :—

	0	1	2	3	4	5	6	7	8	9
77	46·5	46·2	45·9	45·6	45·3	45·0	44·7	44·4	44·1	43·8
78	43·5	43·2	42·9	42·6	42·3	42·05	41·8	41·5	41·2	40·9
79	40·6	40·3	40·0	39·8	39·5	39·2	38·9	38·6	38·4	38·1
80	37·8	37·5	37·3	37·0	36·7	36·45	36·2	35·9	35·7	35·4
81	35·1	34·9	34·6	34·3	34·1	33·8	33·5	33·3	33·0	32·7
82	32·45	32·3	32·0	31·8	31·5	31·3	31·0	30·8	30·5	30·3
83	29·9	29·6	29·4	29·15	28·9	28·65	28·4	28·15	27·9	27·65
84	27·4	27·2	26·9	26·7	26·4	26·2	26·0	25·7	25·5	25·2
85	25·0	24·8	24·5	24·3	24·1	23·85	23·6	23·4	23·2	22·9
86	22·7	22·5	22·3	22·0	21·8	21·6	21·4	21·2	20·9	20·7
87	20·5	20·3	20·1	19·9	19·7	19·45	19·2	19·0	18·8	18·6
88	18·4	18·2	18·0	17·8	17·6	17·4	17·2	17·0	16·8	16·6
89	16·35	16·2	16·0	15·8	15·6	15·4	15·2	15·0	14·8	14·6
90	14·45	14·3	14·1	13·9	13·7	13·55	13·4	13·2	13·0	12·9
91	12·6	12·4	12·2	12·1	11·9	11·7	11·5	11·3	11·2	11·0
92	10·8	10·6	10·45	10·3	10·1	9·95	9·8	9·6	9·4	9·3

Loss of Sugar in Molasses.—At a juice purity of 77°, not less than 46·5 parts of exhausted molasses are obtained per 100 parts of sugar, but this is reduced to 9·3 at a juice purity of 92·9°.

To find the probable loss of sugar in molasses these figures are multiplied by $\frac{90\times30}{100\times100} = 0{\cdot}27$, and the probable loss on 100 of cane is found by multiplying that product by the yield of sugar per 100 cane, and dividing by 100.

A factory recovered 10·95 parts of sugar per 100 cane and the purity of the clarified juice was 85·3°. The probable quantity of molasses on 100 cane was therefore $\frac{10{\cdot}95\times24{\cdot}3}{100} = 2{\cdot}66$ parts, and the loss of sucrose $= 0{\cdot}72$; assuming that the purity of the final molasses was 30°, the polarization of the sugar equalled 96·5°, and that no sucrose was lost by spilling or by inversion.

The statistics of the Mutual Control of Cane Sugar Factories in Java show the following losses of sucrose in molasses for the last few years :—

Year.	Purity of the clarified juice.	Sugar made on 100 cane.*	Loss of Sucrose in Molasses.		
			On 100 cane.	On 100 Sucrose in cane.	On 100 Sugar made.
1908	85·7	10·30	0·97	7·89	9·42
1909	85·7	10·26	0·92	7·57	8·97
1910	86·6	10·76	0·98	7·81	9·11
1911	86·3	10·80	0·96	7·55	8·89
1912	83·2	10·21	1·16	9·39	11·36
1913	82·7	10·00	1·22	9·73	12·20
1914	81·1	9·76	1·01	8·48	10·35
1915	83·5	9·65	1·09	9·38	11·30
1916	85·7	10·42	0·85	6·84	8·16
1917	87·3	11·00	0·91	7·11	8·27
1918	87·9	11·68	0·94	6·95	8·05
1919	88·3	10·44	0·90	7·27	8·62
1920	86·9	11·13	0·93	7·11	8·36
1921	86·9	11·51	1·01	7·16	8·77
1922	86·2	11·08	0·98	7·62	8·84
1923	86·3	11·43	0·80	6·09	7·00

* Polarization = 96·5°.

IV.—Utilization of the Molasses

1.—RECOVERY OF SUGAR FROM MOLASSES

Extraction of Sucrose from Molasses.—Since about 8 per cent. of the sugar present in the cane is lost in molasses, it is not surprising that attempts have been made to extract this portion also. Experiments made on a small scale with a view to separate salts and sucrose by osmosis have not led to favourable results.

Steffen's Extraction Process.—Experiments with Steffen's extraction process and similar methods, which have proved successful with beet molasses, have been unsuccessful in the case of cane molasses, because of the high content of reducing sugar and the requirement of low temperatures.

Deguide's Process.—A new process invented by Deguide* aims at the precipitation of sucrose from molasses by means of baryta and decomposition of the separated barium saccharate by carbonic acid. As, however, the reducing sugar is a serious impediment to the proper separation of the saccharate, the former has first to be got rid of. To this end the cane molasses is diluted with four times its volume of water and boiled for one to two minutes with a solution containing twice as much crystallized baryta as the quantity of reducing sugar to be eliminated. The liquid is filtered and the filtrate, which now is practically free from reducing sugar is evaporated to a density of 40° Baumé. At a temperature of 63° C., so much of a hot and saturated solution of baryta is added that the amount of the latter substance is 1·2 times that of the sucrose in the molasses. The mixture solidifies to a porous cake of barium saccharate, which gives off freely the mother-liquor from which it is originated. It is washed with a hot 2 per cent. solution of baryta till all of the mother-liquor is removed. The yellow-coloured sandy precipitate is mixed with its own weight of water and decomposed by means of a current of carbonic acid gas, till the reaction of the liquid is no longer alkaline. After filtration from the precipitate of barium carbonate, the sucrose solution is evaporated and boiled in the usual way.

The mother liquor and washing waters are also carbonated, and filtered from the barium carbonate thrown down during that operation.

The liquid is evaporated, calcined and worked up to potash salts, while the total barium carbonate obtained is dried, mixed with silica and burnt at a high temperature to form tribarium silicate:

$$3\ BaCO_3 + SiO_2 = (BaO)_3\ SiO_2 + 3\ CO_2$$

When this is boiled with water, it is split up into monobarium silicate and baryta, which latter may be used again in manufacture.

$$(BaO)_3\ SiO_2 + 2\ H_2O = (BaO)\ SiO_2 + 2\ Ba\ (OH)_2$$

Beet molasses treated in the way described here gave up 95 per cent. of its sucrose content, but on treating cane molasses the reducing sugar was found to give trouble.

An experiment with Java molasses containing 32·50 per cent of sucrose and 28·50 of reducing sugar showed that per kilogram of molasses 600 grams of baryta were required for the destruction of reducing sugar and 500 for the precipitation of the sucrose. 92 per cent. of the sucrose or 300 grams was recovered in a solution of 99° purity, which has therefore necessitated the employment of almost four times its weight of baryta.

When considering these relations between sugar extracted and chemical required, one should bear in mind that in many countries the profit obtained by extracting sugar from molasses is not exclusively due to the value of the sugar, but chiefly to the difference in duty levied on sugar from the roots or from the molasses or from sugar imported and domestic sugar. A change in the tariff may at once wipe out the profit by extraction of molasses and turn it into a loss.

* Albert Meurice, "l'Ingénieur chimiste," March, 1924.

2.—USE OF MOLASSES AS FODDER

"Molascuit."—Exhausted cane molasses is readily consumed by horses, mules, oxen, and sheep, but the great difficulty is to store and transport the viscous fluid and to divide it into proper rations. An important step was achieved by Hughes, who found that a mixture of fine bagasse and molasses formed a dry meal, having a high sugar content, and suitable for storage and transport in bags without sweating or staining the packing material.

This product, named "Molascuit," consists of a mixture of 75 parts of molasses and 25 parts of dry, fine parenchyma fibre (pith) of sugar cane. It should not contain less than 45 per cent of total sugars (sucrose and reducing sugars) and not more than 15 per cent. of moisture. The mixture should be homogeneous and dry, so that the bags in which it is transported do not sweat nor become stained, while the molascuit itself should not become sticky, nor cake.

Preparation.—The principal condition is that the fibre be exclusively from the pith cells and not from the fibrovascular bundles, because, as we showed on page 99, the absorptive power of the latter is so much inferior to that of the former. Further, the fibre from the parenchyma or pith cells is much more digestible than that of the bundles, which moreover exert an irritating action on the intestines. The fine bagasse (chiefly from the fibre of the pith) can be obtained by sifting the green bagasse as it comes from the mills. Large sieves of coarse gauze are placed under the slide of the mills, the bagasse falls on them, and the finer parts pass through. This fine bagasse is dried to 90 per cent. of dry substance either by hot air, or by steam, or on the chimney flue, after which it is sifted again, if necessary, and mixed with hot concentrated molasses of 88° Brix (85 per cent. of dry substance).

3.—USE OF MOLASSES AS FUEL.

Calorific Values.—As molasses contains about 70 per cent. of organic substances, many attempts have been made to use it as fuel. The calorific value of molasses containing 9·28 per cent. ash, and 19·4 per cent. moisture, was found to be 5515 B.T.U.; or, calculated to 100 parts of dry organic matter, 7366·4. It is, therefore, superior to that of sucrose (7119), and equal to 80 per cent. of that of cane fibre (8550).

Mixing with Bagasse.—The use of molasses as fuel presents certain difficulties; it burns very badly without addition of some other fuel, and, when mixed with bagasse, forms a voluminous mass of coke, which chokes up the grate and hinders the free access of air. At a high temperature the lime and alkali of the molasses combine with the silica from the bagasse, forming a kind of glass, which covers the grate bars and again prevents the admission of air.

In some factories, where the bagasse was sprinkled with molasses on its passage from the mills to the furnaces, much labour was necessary to clear the furnace and remove the slag from the grate, so that in every instance the addition of molasses to the bagasse was discontinued.

Special Molasses Furnaces.—In very large factories it will pay to build special molasses furnaces, such as are used in several cane-growing countries, and were described in the *Hawaiian Planters' Monthly* of December, 1903. These furnaces have no grate, and the air enters under the fuel through holes made in the walls. The molasses are either blown into the furnace by means of a steam jet through a kind of injector, or are poured upon an iron plate in front of the furnace, allowed to dry, and thence shovelled into the fire. This is started with a little firewood, but, once being lit, it is kept burning with the dried molasses. The combustion is perfect, the ash fine and almost free from carbon, while the heating power of two parts of molasses is approximately equal to one part of dry firewood. In most cases, the amount of available molasses will be insufficient to furnish fuel for one boiler, so that only very large factories can profitably adopt this practice.

The ash is very rich in potash, containing as much as 35 per cent. of this constituent. It may therefore be used by glassmakers, and (after being refined) by soapmakers, or as a fertilizer.

4.—MOLASSES AS RAW MATERIAL FOR RUM PRODUCTION

The principal application of waste molasses is in the manufacture of rum or arrack in countries where the excise regulations permit of this, and where there is a market for the distilled product. As this industry is quite distinct from sugar making, we shall not enter into a discussion of fermenting and distilling here, although these form a very important item in many sugar factories.

5.—USE OF MOLASSES FOR MOTOR SPIRIT PRODUCTION

A new use for alcohol has sprung up in recent times as liquid fuel for combustion motors. Common denaturated alcohol could not be used as a reliable combustible in such motors since its heat of combustion is not sufficient to guarantee the steady supply of gas. This may, however, be remedied by mixing the alcohol with a proper quantity of a more volatile body such as naphtha, benzol, ether and the like.

A mixture patented in many countries is " Natalite," consisting of :—

Alcohol (95 per cent.) ..	54·50
Ether	45·00
Ammonia	0·50
	100·00

While in past times the manufacture of ether in a tropical sugar factory was a rather awkward operation, owing to its volatility, the introduction of automatic plants, in which the ether vapours are condensed by alcohol and which turn out a single product, viz., finished alcohol-ether, have greatly reduced the danger of conflagration. Besides mixtures of alcohol and ether, a great many other mixtures of alcohol and volatile bodies have been successfully put on the market and are doing good service.

Instead of lowering the boiling point of alcohol by means of ether, raising the heat of combustion by addition of heavy hydrocarbons, e.g., petrol, may also serve the purpose. In order to obtain mixtures of alcohol and petrol, which are stable and do not separate into two layers on exposure to low temperature, it is indispensable that the alcohol should be anhydrous. Up to a short time ago, the preparation of absolute alcohol was a wasteful and expensive operation, but now it has been found possible to separate the 95 per cent. alcohol into its two constituents, viz., anhydrous alcohol and water, in a practical manner by a continuous method.

When distilling a mixture of 95 per cent. alcohol and benzene, or any other suitable liquid, in a rectifying column, the distillate may be divided into three fractions. The first consists of a ternary mixture of water, benzene and alcohol, of which the boiling point is lower by at least 10° C. than that of alcohol. The second fraction is a mixture of very little anhydrous alcohol and much benzene, while the third portion consists of pure anhydrous alcohol.

The first fraction passes into a decanting vessel in which it separates into two layers. The upper one, containing a large proportion of benzene and a small quantity of alcohol is returned into the column in order to extract a further amount of water from it. The undermost layer, consisting of water, alcohol and very little benzene, is rectified in a small column and separated into water and alcohol of 95 per cent. contaminated with some benzene, which is returned to the first column again. The second fraction goes back into the column too. Mixtures of this anhydrous alcohol and heavy hydrocarbons may be used as motor spirit and will not separate out again.

6.—USE OF MOLASSES AS A FERTILIZER

Some estates employ molasses as a fertilizer, or as a means to break up rocky subsoil, thus facilitating tillage. When used as manure, molasses is simply poured over the earth on the banks.

7.—OTHER USES

All attempts to manufacture gas from molasses have failed ; nor can molasses be used for the manufacture of cyanides and other nitrogenous substances as is the case with beetroot molasses, because it contains only trifling amounts of nitrogen.

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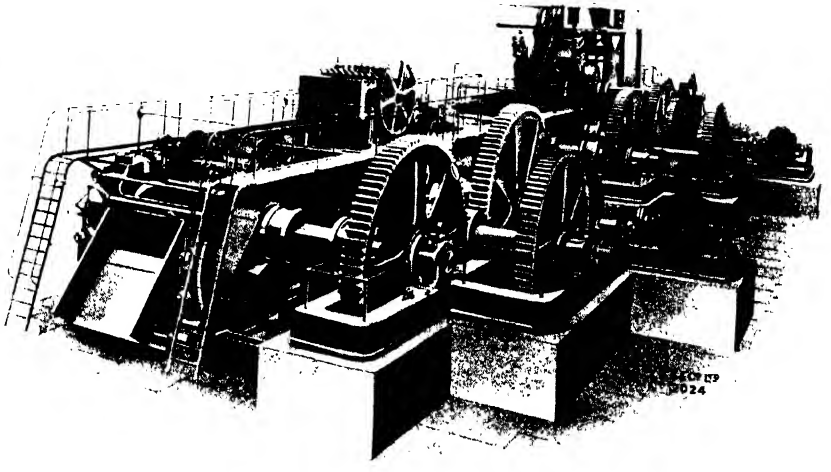

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The NORIT PROCESS applying Norit, a vegetable highly activated and fibrous carbon and revivifying same for re-use is IN USE SINCE 1911, and **IS STILL THE ONLY PROCESS** IN PRACTICAL USE IN THE CANE AND BEET SUGAR INDUSTRY whereby decolorizing carbon, other than bonechar, is used and re-used after revivification.

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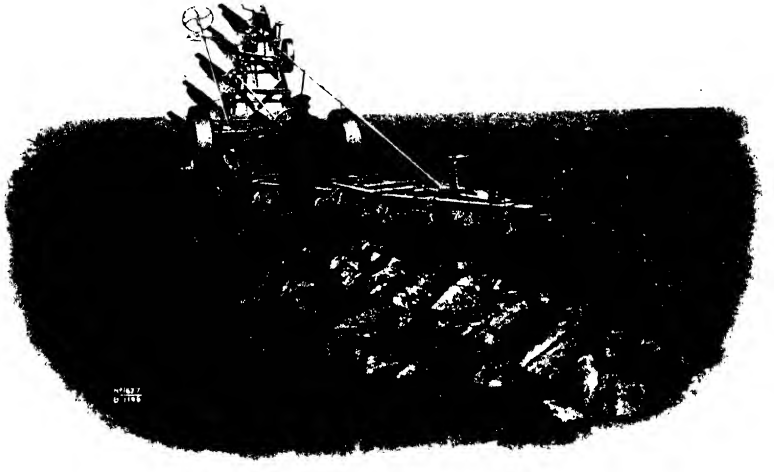

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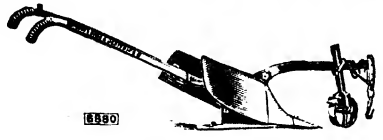

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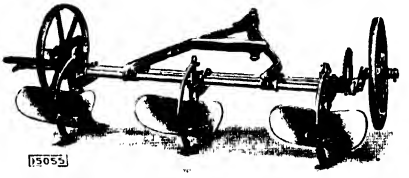

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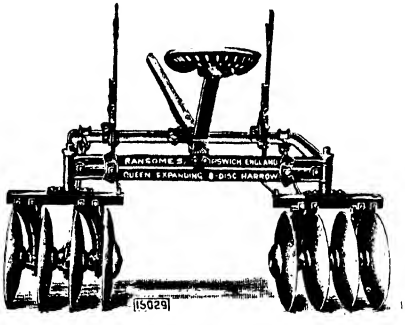

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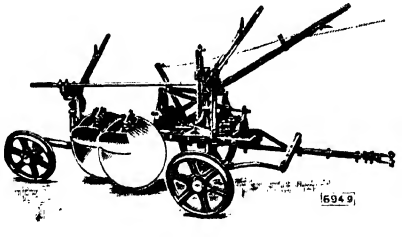

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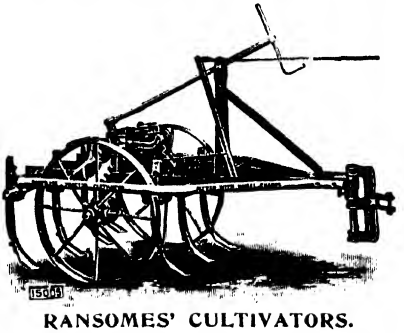

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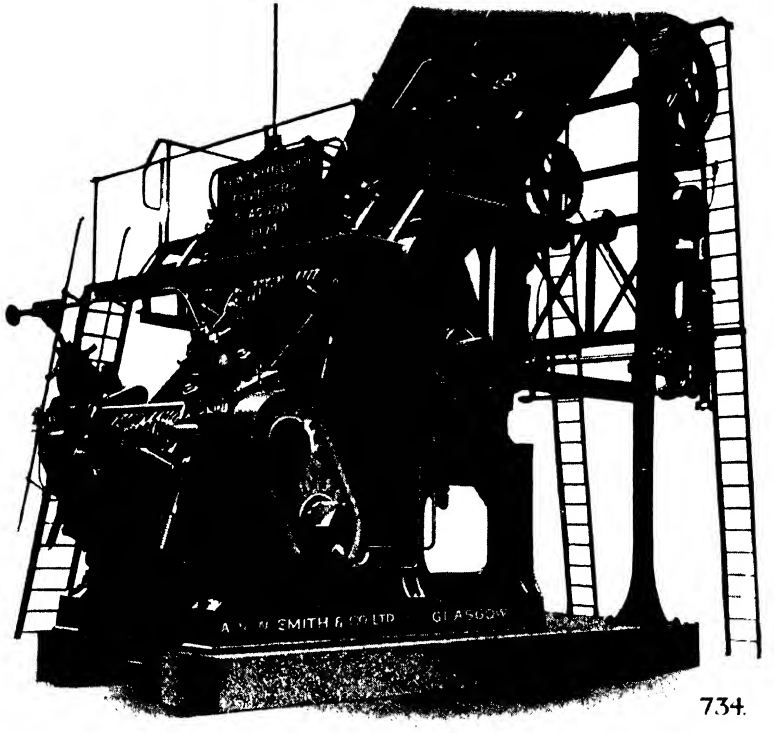

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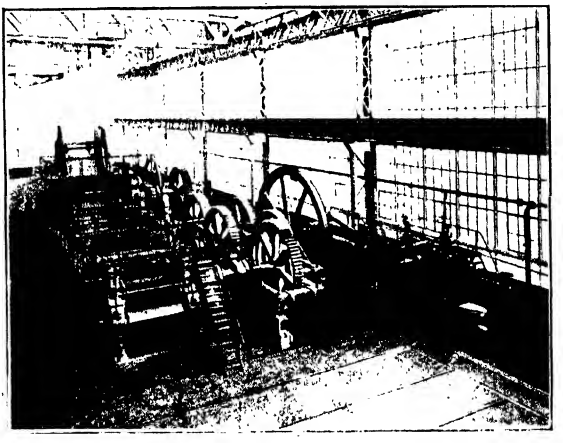

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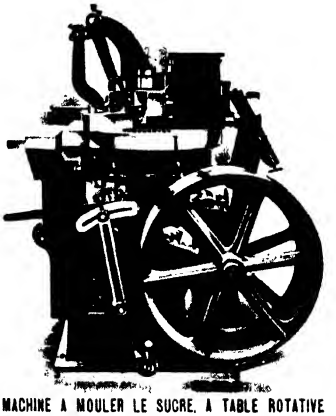

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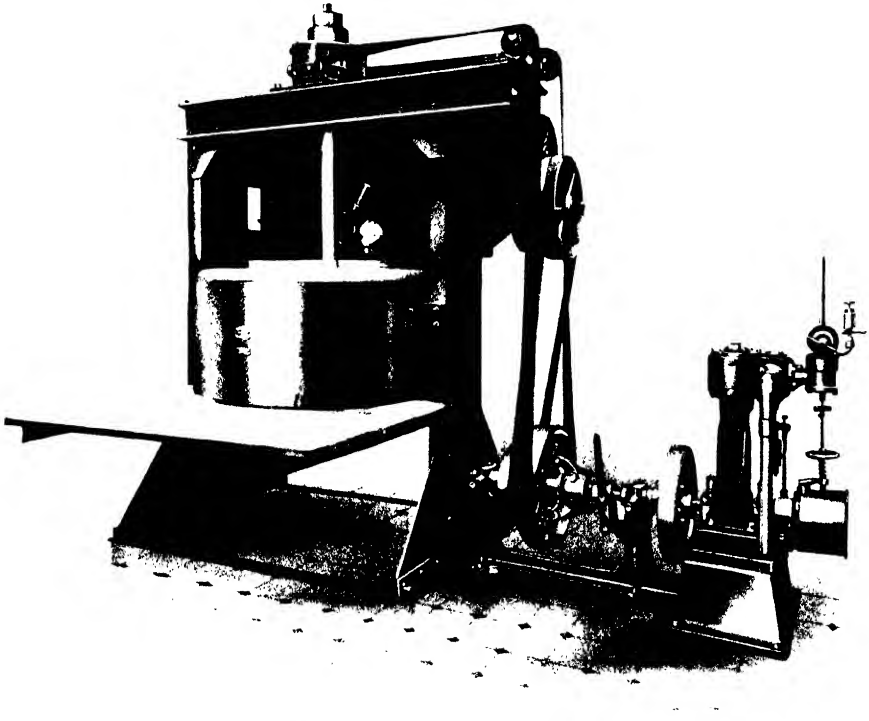

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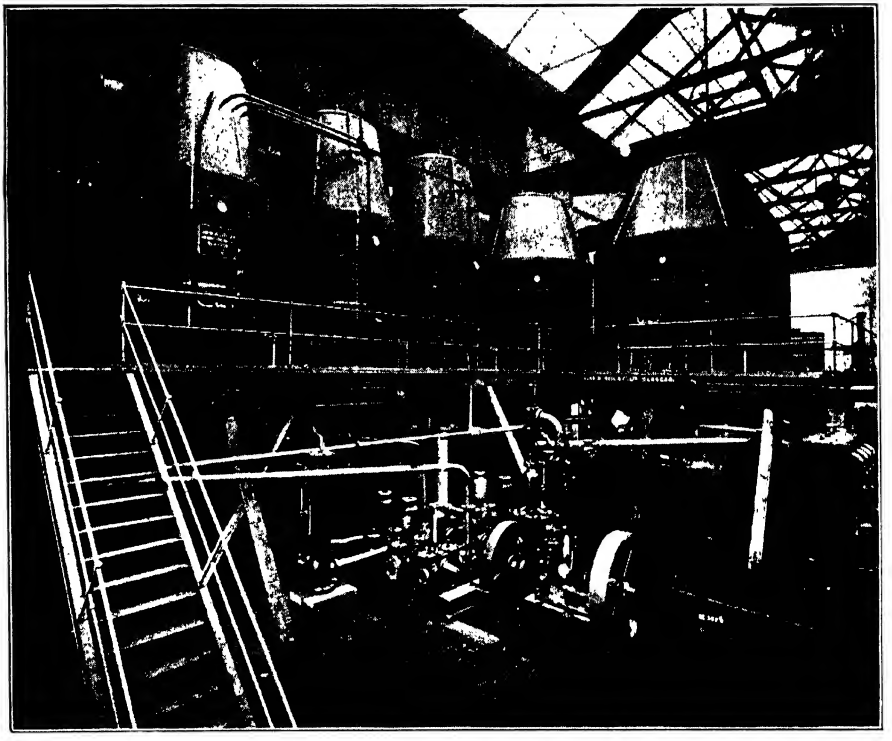

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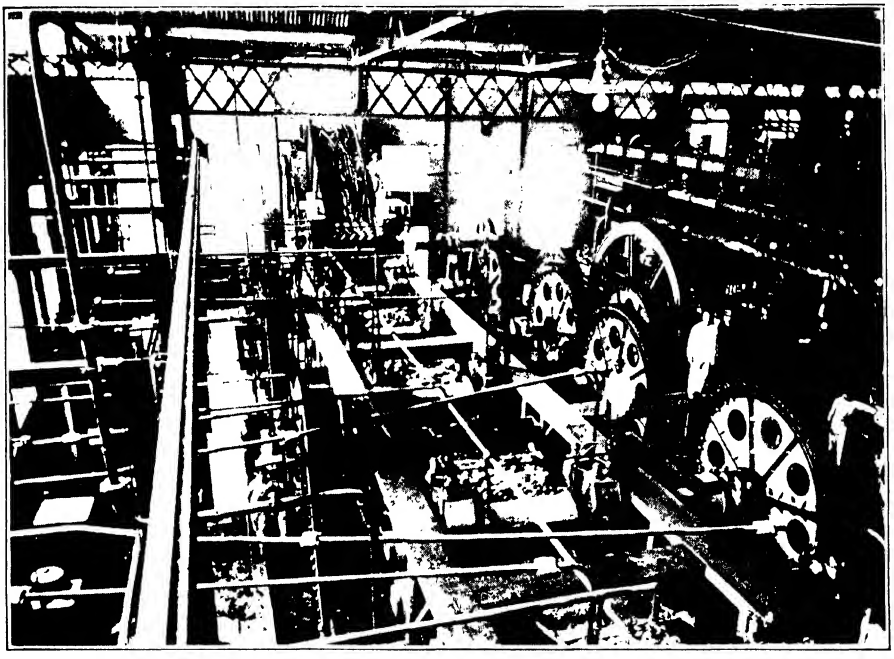

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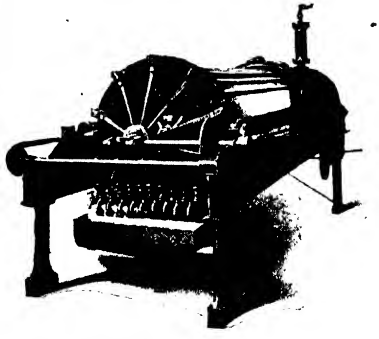

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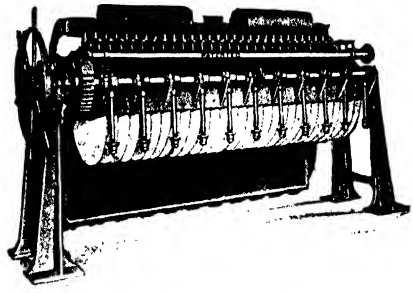

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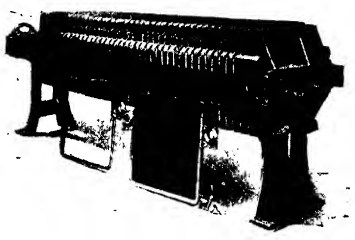

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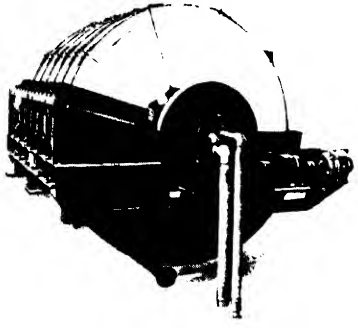

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Our engineers are consequently able, without bias or prejudice, to recommend the general type or types of filters which in their judgment will best serve the needs of the individual mill. Securing our advice in a matter of this sort places you under absolutely no obligation to us, although naturally we hope to convince the prospective purchaser that our own equipment offers the best example of whichever general type may be recommended.

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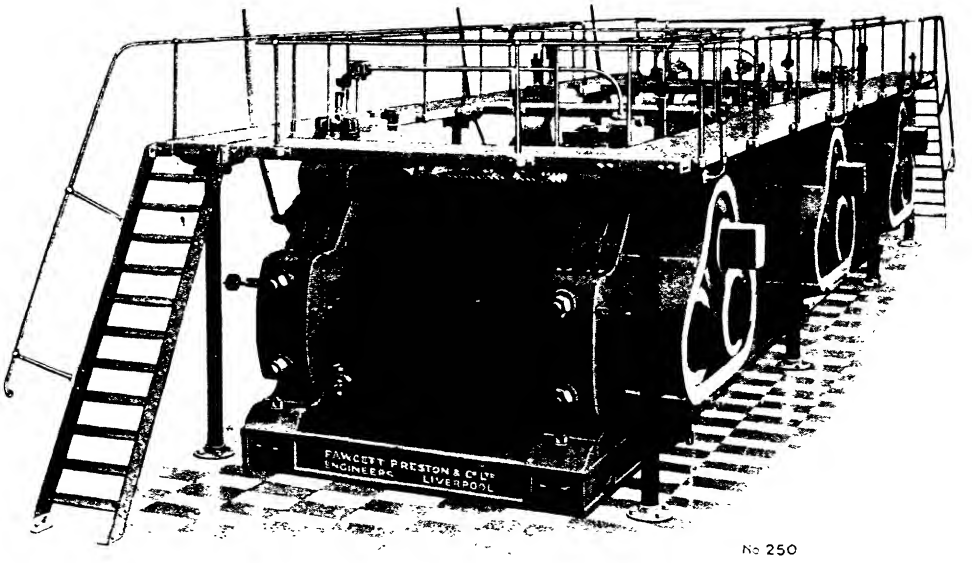

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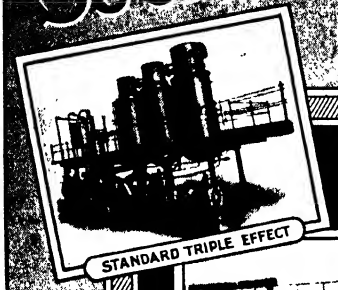

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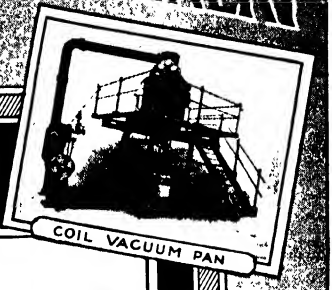

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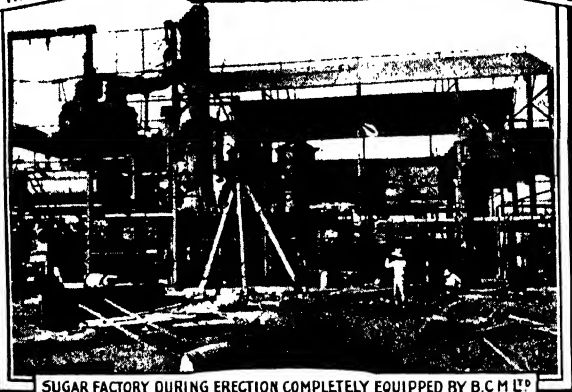

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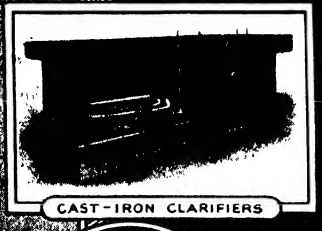

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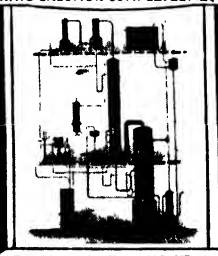

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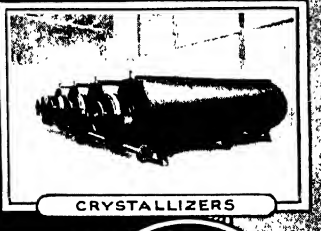

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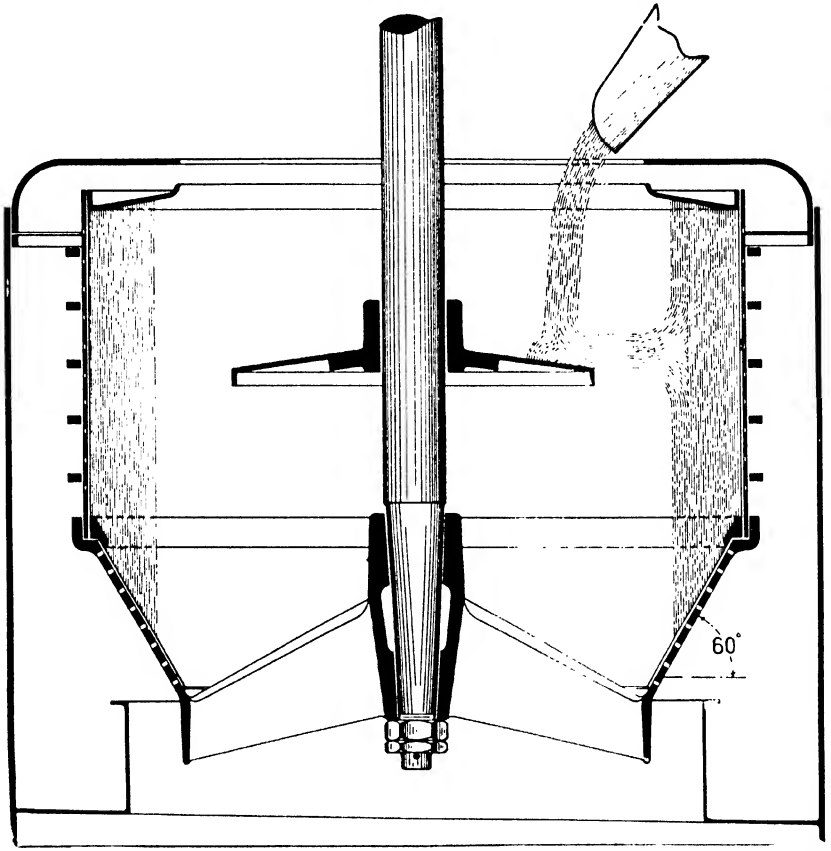

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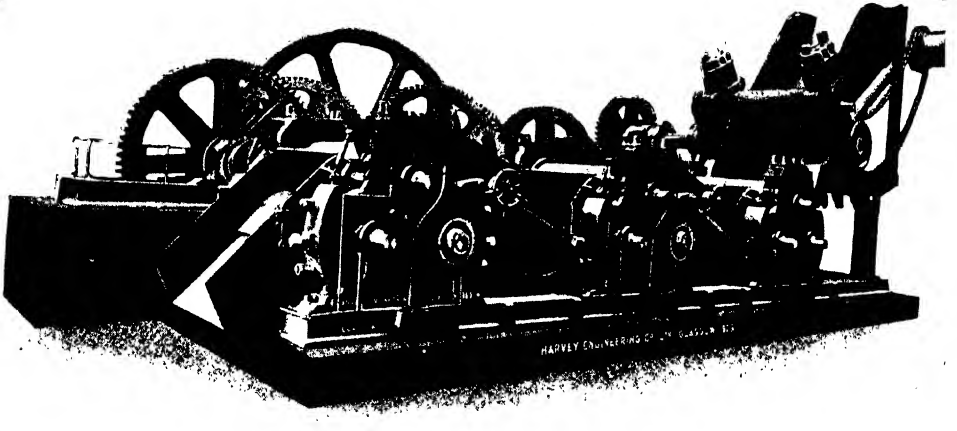

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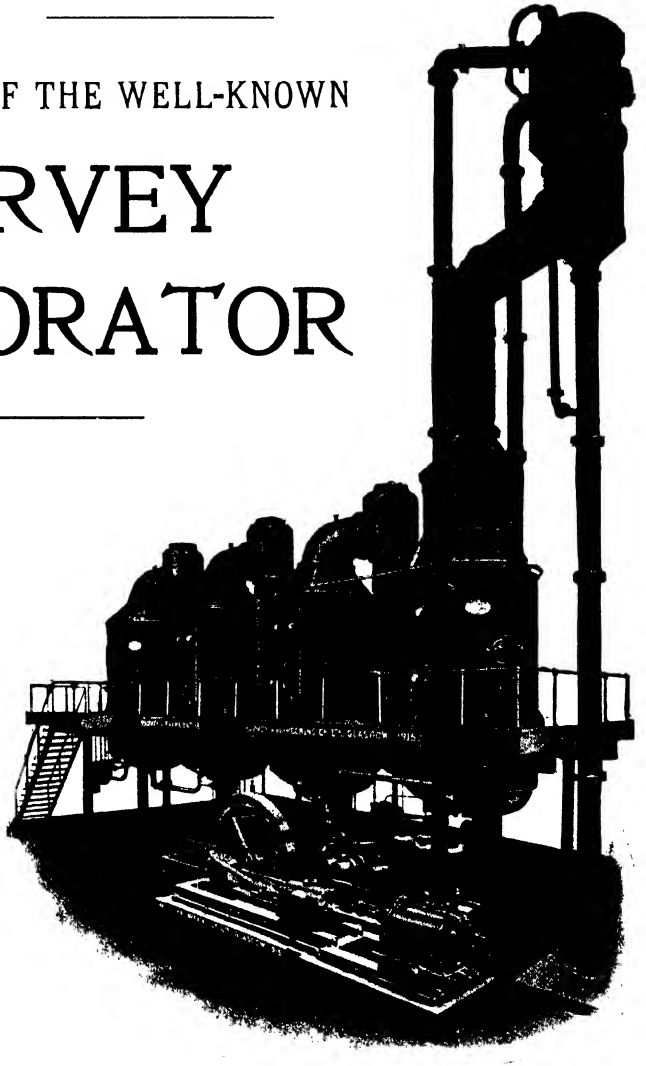

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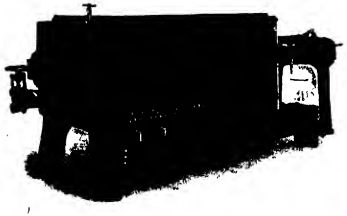

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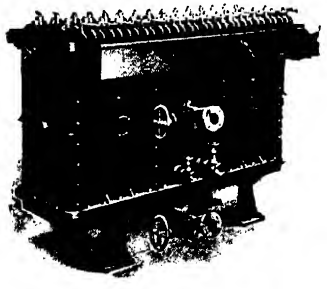

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Successful Application of NORIT in Cane and Beet Sugar Factories.

NORIT AS APPLIED SINCE 1920 IN RAW CANE SUGAR FACTORIES IN LOUISIANA.

(Extracts from an article in the "Louisiana Planter," by W. H. DUNSTONE, Jr., Late Plant Manager of the Sterling Refinery.)

THE FOLLOWING ARTICLE CLEARLY INDICATES THE DETAILS OF APPLICATION OF THE NORIT PROCESS IN ACTUAL PRACTICE.

Economically wrong for cane or beet sugar factories to stand idle from 8 to 10 months out of each year.

"*On the face of the matter, it looks entirely economically wrong for a Louisiana sugar factory, representing a comparatively large capital investment, to operate about two months and to stand idle for ten months out of each year.* On and off for a decade or more, there have been attempts made to take advantage of the idle equipment in the sugar houses, and to refine raw sugars; but in the main, the crop has been ground in the usual way, the house cleaned up, machinery laid by, and no further operations conducted except the drying of third sugars in the summer, and the necessary repairs made for the next season."

"For the past two years the writer has been connected with the *Sterling Refinery*, one of the largest plants operated in Louisiana, and which plant has practically *solved the above mentioned economic problem.*"

"*Sterling originally a raw sugar factory*, where as the demands of the market prompted, plantation granulated, yellow clarified, or 96-test raws were manufactured, *has since the beginning of* 1920 *blossomed out as a full fledged sugar refinery, using* **NORIT**, *a vegetable carbon. This factory has a grinding capacity of* 2,000 *tons daily, grinding on the average* 100,000 *tons a year; and operating as a refinery, has a daily melting capacity of* 170 *tons of raw sugar.* This capacity can be increased to 250 tons per day by a few additions to their present equipment; and this it is contemplated soon to do."

Raw sugar-house plant.

"During the grinding season, sugar cane is unloaded from standard gauge cars by an overhead grab system. The cane is ground by two nine-roller mills and crushers, that are parallel. There are approximately 3,000 horse power of boilers, 1,750 H.P. of which is in Stirling water-tube boilers. The juice is evaporated in a standard quadruple effect which has ample capacity for the cane ground. Liming tanks, four heaters, and Deming settlers comprise the juice clarification end of the house. The mud from the settlers is taken care of by ample mud blow-ups and modern plate-and-frame filter-presses."

"The boiling capacity consists of four pans. Eight 40" machines, nine 36" and twelve 30", under three mixers, constitute the centrifugal equipment. A melter, two granulators, revolving screen, automatic scales, bag sewing machine and electric trucks, make up the complement of a modern sugar factory."

"All this equipment is housed in two large four-storey brick and steel construction, fireproof buildings."

NORIT plant.

"In addition to all the above is the **NORIT** plant. This consists of a three-storey steel and galvanised iron building, containing the following: six 36" plate-and-frame presses, one Sweetland filter, two small bag-filters, three mixing tanks, one wooden cistern, two receiving or storage tanks, a **NORIT** kiln, and necessary pumps."

Successful refining with the aid of NORIT.

"*Sterling has as above mentioned, engaged successfully in the refining of both Louisiana and Cuban raw sugars during the period from February,* 1920, *up to and including the time of this writing, when she is melting Cuban raws exclusively.*"

Combining raw sugar manufacture from cane with raw sugar refining.

A description of the operation of the factory follows:— "As the cane passes on the carrier to mills it is washed with a sprayer or shower suspended over the carrier, using condensation water from the effects. The juice, strained in the usual way is pumped to the liming tanks. Milk-of-lime to reduce the natural acidity to 0·2 c.c. or 0·3 c.c. is added, and also "Filter-Cel" in the proportion of about 4·5 lbs. per ton of cane. The "Filter-Cel" is added in a water suspension of about 15° Bé. The juice is then pumped through the heaters, heating it to about 215°F., to the settling tanks. Decanting the clear juice over the tops of the settlers, it goes to the charge tank of the effects, and is there concentrated to a syrup of about 60° Brix."

(Continued on next page.)

Boiling system in raw sugar end of the house.

"The syrup from the effects is pumped to the pan floor and first and second massecuites boiled therefrom. The first massecuite is boiled at about 72° purity, the high purity of the syrup being reduced by "topping-off" with molasses; and the second massecuite or "boil back" is boiled at about 60° purity by cutting over from the first pan and building up with molasses. These boil-back strikes are dropped into the hot-room cars, left in the hot-room for four or five days and then purged. The resulting run-off ranges from 25 to 28° purity, and is pumped outside as final molasses or black-strap."

"The first massecuite is dropped into a mixer and dried in 36" machines. In the drying, about three gallons of water are applied and the sugar washed to 99° purity. The run off, or molasses, leaving the centrifugals before the water is applied, is run through a separate trough and into separate tanks from that into which the wash, or highest purity molasses which leaves the machines after the water is put on, is discharged. This run-off is used for "topping off" the first massecuites and the balance is boiled up for second massecuites."

"The disposition of the wash is referred to later."

"The second massecuites are dried in 30" machines without washing or with about a half pint of water dashed into the machine when the clutch is thrown out, to facilitate cutting down. This sugar is then conveyed across to a bucket elevator, carried up and discharged into a mingler, where it is "mingled" or mixed to a proper consistency with the wash above referred to as coming from the washing of the first massecuite sugar. This mingled second sugar falls into the mixer containing first massecuite and is mixed with it, so that it gets a double purging, and is washed up with the first massecuite sugar to 99° purity. As previously recited, the run off from the second massecuites is boiled either to thirds, or is pumped out to the final molasses tanks."

Refining process with the aid of NORIT during the grinding season.

"The washed raw sugar of 99° purity then goes to a melter, where with either clear water, or high purity sweet-water, it is dissolved. This liquor, after being strained through strainers made of centrifugal liners, is pumped through a heater where it is brought to 200° F., and discharged into the **NORIT** mixing tanks. **NORIT** in measured quantities is added, the mixture stirred with mechanical stirrers, and then pumped to the top floor through plate-and-frame presses."

Quality of liquors and refined sugars obtained.

"*The filtered liquor leaving the presses is practically water white from the larger initial doses of* **NORIT** *used, and the general average of the liquor leaving the presses in the complete cycle of operations, is such a nearly white colour as to permit of economical operation and the production of a beautiful white, standard granulated sugar, the equal in every way to the best boneblack sugar produced.*"

Filtration and decolorization with the aid of NORIT in one operation.

"At Sterling three **NORIT** mixing tanks of approximately 4,000 gallons each are used alternately. For filtering the liquor, four 36" plate and-frame presses, aggregating approximately 2,700 square feet filtering surface are used. In addition, a Sweetland filter is also used to augment the capacity in a manner described later."

"The **NORIT** is used three times, and occasionally, a part four times, before returning to the kiln to be reburned and re-activated. Two presses are run at the same time; and in the using of the **NORIT** the first time, five tanks of liquor with **NORIT** added, are pumped through the two presses in succession, the total amount of **NORIT** added to the five tanks being the amount which fills the chambers in the presses. It is known that the two presses hold 1,800 lbs. of **NORIT** dry basis. In the second using of the **NORIT** the contents of the two presses are divided between three tanks of liquor which are pumped through the two presses, in succession. The cake in these two presses is in turn air dried, the cake cut down and the **NORIT** used a third time. In the third using the 1,800 lbs. of **NORIT** is divided equally between two tanks of liquor, which are pumped through the said two presses."

Sugar recovery and revivification of NORIT.

"After the completion of the above described cycle, the **NORIT** is dried in the presses and cut down, being now dumped into a wooden cistern of 4,000 gallons capacity. This cistern has a mechanical stirrer, and water is added to the sweet **NORIT**. This mixture is then heated and pumped through another plate-and-frame press installed for this purpose and "sweetened off." **NORIT** has the faculty of retaining the impurities absorbed from the sugar liquor and the sweet-water coming from this press is sent to the melter for dissolving the washed sugar. The sweetened-off **NORIT** cake from this press polarizes from zero to 1·0 per cent.; generally under the latter figure."

Re-burning NORIT.

"This sweetened-off **NORIT** is then sent to the **NORIT** kiln. This kiln consists of two pairs of cast-iron retorts arranged horizontally one above the other, and half the **NORIT** is conveyed through one pair of retorts, and half through the other two. Heat is supplied by burning oil (or coke), and before entering the kiln proper, the **NORIT** is dried in two pans located on top of the kiln, which are heated by the gases from the furnace. The **NORIT** is conveyed slowly through the retorts by conveyors inside, and the lower retorts attain a cherry red colour. The temperature inside the lower part of the kiln chamber is maintained at

(Continued on next page.)

1100° F. The **NORIT** is discharged from the lower retorts of the kiln into a tank of water. This water prevents the dry **NORIT** from flying about, and also acts as a seal on the retorts."

"From this tank which is fitted with a mechanical stirrer, the mixture of water and **NORIT** is either used as such, or is pumped periodically up to a plate and frame press installed for this purpose, and after the press is filled, the cake is well dried with air and then cut down (as in this case). This **NORIT** is now ready to be used over again on fresh batches of liquor, and will be used three times again before it is reburned."

"If the kiln is fed uniformly, its operation is very satisfactory, but as a matter of routine check samples are taken every half-hour, composited and tests made every three hours by boiling a portion of the dry **NORIT** with a 5 per cent. caustic soda solution, to determine if the **NORIT** has been well burned."

"The **NORIT** filtered liquor, in flowing from the presses, passes through a small bag filter. This is a precautionary measure to eliminate the possibility of any quantity of **NORIT** getting by in case of a bad cloth in the press or any bursting out due to bad dressing of the press. It is then pumped to the pan floor and boiled into granulated sugar."

System of boiling refined sugars.

"Four boilings are made as follows:—Two strikes of all first liquor are boiled in succession, the run-offs from these two strikes boiled together for a second, and the run-off from the second held. This operation is repeated until the second run-off from a second boiling is obtained, when the two second run-offs are boiled for the third. This complete operation is repeated and when two run-offs from thirds are obtained a fourth is boiled. These strikes are dried in 40″ machines with good clean water obtained from the tail pipes on the pans. *The first, second and third sugars are all good enough to pack by themselves*, but as two pans are dropping alternately, a mixture is obtained by mixing in the sugar bin. The sugar from the fourth boiling is always held until it is followed by a first with which it is mixed."

"In drying (centrifugalling) these granulated strikes, the wash and run-off are allowed to mix together, except in the case of the last boiling. In drying this strike the wash and run-off are separated; the wash being of higher purity and better colour is boiled back for a second or third granulated, and the run-off of about 89° purity is pumped to the same tank into which is pumped the wash from washing the raw sugar, so that a portion of it is used for mingling the second raw sugars which are double-purged. What is not disposed of in this way is taken into the raw sugar pan along with syrup and boiled for raws. In any event this tank is liquidated every 24 hours."

Handling the refined sugar.

"The white sugar from the centrifugals is elevated to a bin and fed down into the granulators, through a revolving screen, to automatic scales, and into 100 lb. pockets, or is packed in barrels. The sacks are all sewed by a machine, loaded on to platforms, which are picked up by electric trucks, and carried over platform scales for check on weight and count, and are taken direct to freight cars for shipment, or into the warehouse for storage."

"*Thus, in the converting of the juice of the cane into granulated sugar and final molasses, all under one roof, we have the nearest approach to the planters' dream; and all at a commercially possible operating cost.*"

"The sugar produced has without exception been the best from a refining point of view, in facility of washing up, filtration and decolorisation."

Refining of raw sugars during the idle season.

"*Returning again to the refining of raws, carried on after the grinding season*, the methods of operation in the **NORIT** plant and pan-house are the same as described as being used during the grinding. Instead of getting the raw sugar from the pans, sugar in sacks is trucked from freight cars or warehouse, the sugar dumped into the boot of a bucket elevator, carried to the mingler, and the massecuite made for washing the raws. The battery of nine 36″ machines is used for this purpose, and the raw sugar is washed up to 99 purity, the wash and the run-off being separated."

"The wash is used for mingling the raws dumped, as is also the run-off from the last granulated strikes. The run-off, which has a purity of about 76 to 79°, is boiled for a "high remelt," the purity of the massecuite being brought down to about 74° by topping off with run-off from a previous strike of high remelt. These strikes are dropped into the mixer of the 30″ machines and this mixer having a partition, dividing the twelve machines into two batteries of four and eight machines respectively; eight machines are used to dry these strikes. These high remelt strikes are dried without washing, producing a sugar of about 94·5 to 95·0 · polarization, and are conveyed across and dumped in with the raws at the raw sugar elevator. The run-off has a purity of about 48°. This run-off is boiled back on a cut-over from the high remelt pan, and the strike dropped at about 64° purity, called "low remelt." These strikes are dropped into hot-room cars in lieu of crystallizers, and after two or three or more days in the hot-room, are dumped into the smaller part of the above referred to mixer and are dried without washing in the four 30″ machines. This sugar has a polarization of 89 to 90°, and is also conveyed across and dumped into the raw sugar elevator. The run-off of about 35 to 37° purity goes out to the molasses tanks as black-strap."

"The remelt sugars are washed up along with the raws dumped and of course go back into process."

(Continued on next page.)

No pre-filtration of liquor required.

"*It will be noted that in refining raws as practised at Sterling, no pre filtration of the washed sugar liquor is resorted to*, thus eliminating the cost and trouble incidental to using bag-filters or any type of leaf-filters. This of course puts more of a burden on the **NORIT** and in the course of time the **NORIT** accumulates a quantity of mineral matter thus reducing the carbon ratio, and partially clogging the pores of the **NORIT**."

Occasional chemical treatment.

"In order to reduce this ash in the **NORIT**, it is periodically boiled with a 2 per cent. hydrochloric acid solution, and also at times with a 5 per cent. caustic soda solution. The acid treatment is given in the wooden cistern, previously referred to, which is also used for sweetening-off the **NORIT** press cake ; and one of the iron mixing tanks is used for the soda treatment."

"No attempt will be made in this article to make a comparison of all the relative merits of refining with **NORIT** and other methods, notably with boneblack ; but it may be permissible, in passing, to draw attention to the *simplicity of the process, the comparatively small amount of material in process, the short time elapsing from one step in the process to another and the obvious advantages thereof.*"

Yield of refined sugar.

(The sugar obtained is as good as standard granulated sugars obtained by modern bonechar refineries.)

"*It has been demonstrated beyond a doubt that a recovery of 93 lbs. of standard granulated sugar per 100 lbs. of 96-test raws can be obtained ; and also that the process can be conducted on a commercially profitable scale.*"

Advantages of the NORIT application in a raw sugar house.

"A **NORIT** installation in a factory provides the two-fold benefit of permitting the planter to convert his cane into a sugar for which there is a better demand than any other sugar, as well as being a better keeping sugar, should he decide to hold it for more advantageous marketing ; and that of having the equipment available to enter the refining field, buying his neighbour's raws, or foreign sugars, and thus help to carry overhead, keep his organisation together, and enhance the intrinsic value of his factory."

B. SANDMANN, a Louisiana sugar expert and late Superintendent of the Godchaux Refineries "Reserve" and "Elmhall," which former refinery uses bonechar and the latter has applied **NORIT,** says in "Facts about Sugar," *re*

Yield of refined sugars obtained :

"*Now, as to actual figures of yield on granulated sugar on a basis of a 96 per cent. raw sugar the percentage recovered in most plants using* **NORIT** *amounts to 93·33, a figure just as good as the best conducted bonechar refineries can boast of.* It thus appears that there need be absolutely no difference in yield, whether bonechar or vegetable carbons are used as medium for refining raw sugars."

"Sweetwaters."

"Sweetwaters, obtained from **NORIT**, for instance, *are of high purity, and for that reason can be taken right back into the circuit to melt washed sugars, while (as mentioned above) sweet waters originating from bonechar are barred on account of their low purity and general unfitness.* While it may take more water in the beginning to wash raw sugars to a high purity when refining with vegetable carbons, and the amount of affination syrup consequently will be increased, this will be more than compensated for by the quantity of evaporated sweet-water to be taken care of in a boneblack plant."

In *Louisiana,* "*Sterling,*" "*Southdown,*" "*Elmhall,*" and "*Salsburg*" refineries are working the **NORIT** *process* for a number of years.

The **NORIT** *process for refining either cane or beet sugar or for the manufacture of "Golden Syrup," glucose or cornsyrup has been in actual use since* 1911. *Except experimentally no factory has up to now* (1924) *actually applied any other decolorising carbon (with its revivification) in Louisiana, or elsewhere in the world, for the refining of raw sugar, other than* **NORIT**.

The **NORIT** *process is worked in Holland, Belgium, France, Italy, England, Portugal, Germany, Czecho-Slovakia, Denmark, Canada, the United States of America, Venezuela, Hong Kong, Japan, and Mozambique.*

Some factories produce only white standard granulated sugar, others manufacture beside granulated sugar, Würfel cubes and loaf-sugar by the "Adant" process, others again make pressed cubes and pressed loaf-sugar and "Pilet," while also "Golden Syrup" and other kinds of table-syrups are produced by the **NORIT** *process. In Holland, the Scandinavian Islands, Germany, Italy, and the U.S.A., glucose made of maize and potato-starch is manufactured by the aid of* **NORIT**.

(Continued on next page.)

Application of the NORIT Process in Holland.

"NORIT" process at Dinteloord.

Prof. Dr. A. HERZFELD, the eminent German sugar expert and chemist, published in *Die Deutsche Zuckerindustrie* his impressions with **NORIT** in the beet sugar factory Dinteloord where he personally witnessed the success of this process.

The Dinteloord co operative beet sugar factory and refinery, under management of Director Dr. VLEKKE, has a daily capacity of 3,000 tons of roots, and turns out solely white granulated sugar, mainly for the English market, in a quantity of about 4,000 bags of 100 kilos per day.

The **NORIT** plant at Dinteloord consists of : 3 mixing tanks, 2 montejus, 4 filter-presses for sugar liquor, 3 gravity filters, 2 wooden acid tanks, 1 soda-tank, 3 reburning furnaces (1 double and 2 single ones), and 2 small presses for washing the carbon.

Method of applying NORIT.

During the season all the after product sugars at Dinteloord are affinated (washed in centrifugals), melted, treated with **NORIT** and mixed in with evaporator beet syrup from which mixture the white granulated sugar is boiled. This system has the advantage that the refined sugar is boiled from a syrup of high purity and light colour, which liquor, moreover, is absolutely clear and brilliant.

The white granulated sugar produced is excellent, equalling bonechar refined sugar as to whiteness and appearance. The sugar turned out day after day from the beginning of the season to the end is of exactly the same whiteness and brilliancy, and sells in the London market at the same price as bonechar granulated sugars.

After the season, beet sugar from other factories is refined at Dinteloord, and after that, Javan and Cuban raw cane sugar is similarly treated in the same factory. In all these instances **NORIT** is used, and one and the same **NORIT** plant is utilised.

Dinteloord has no bonechar plant, and has refined beet and cane sugars during the past eight years solely by the **NORIT** process.

The sugar-liquors are mixed with this carbon in the mixing tanks, heated to 90° C., and the **NORITED** liquor is then filtered through plate-and-frame presses, montejus instead of pumps being used, whereafter the filtered liquors are run through Danek gravity filters. The use of these gravity filters is only a safety measure.

At Dinteloord there is no trace of **NORIT** to be seen in the factory, which is clean throughout. The **NORIT** is suspended in water, and this suspension is pumped into the sugar liquors. The **NORIT** is used to the amount of 0·25 to 0·50% for beet sugars ; and 1% -1½% if cane sugars are being worked.

Prof. Herzfeld's statement.

Prof. Dr. A. HERZFELD says in his article that the filter-presses ran fast and brilliant, so that the after-filtration through Danek filters appeared not to be necessary. The maximum pressure in the **NORIT** presses is kept at about 2·5 Atm. (about 37½ lbs. per sq. inch).

Regeneration of NORIT.

After the filter-presses are filled with **NORIT**, the cakes are washed with hot water, and the sweet-water used for melting fresh lots of affinated (washed) raw sugars. Compressed air is used for drying the **NORIT** cakes in the presses. These cakes are either used again, or are transported to the revivification plant to be regenerated. The **NORIT** to be regenerated is pumped into the wooden acid-tanks, and either washed with water or occasionally treated with hydrochloric acid by boiling the mixture for about half-an-hour or an hour, after which the acid paste, diluted with water, is pumped through plate-and-frame presses. The cakes are washed in the press with hot water, dried with compressed air, and discharged into the pre-drying tanks of the furnace. The pre-dried **NORIT** is passed through the furnace (patented by the GENERAL NORIT COMPANY, Limited), and there subjected to a heat of 500 to 550° C., whereafter the reburned carbon is discharged into water in a mixing tank.

NORIT can be revivified, and re-used, easily about 50 times, and when used on good quality sugars the revivification can be carried out still oftener up to 100 times.

(Continued on next page.)

Application of the NORIT Process in Czecho-Slovakia.

NORIT process at Radbor.

The first experiments, on the refining of raw beet sugar with the aid of **NORIT** in Czecho-Slovakia, were carried out in the Radbor Sugar Refinery in the off-season of the 1921-1922 campaign. The result of this experiment, which only lasted one week, was such, with regard to the quality of the decolorised liquor and sugar obtained, that the owner of the Radbor Sugar Refinery took the trouble during the season of 1922-1923 to pay a visit to the Dinteloord Sugar Refinery in Holland, which since 1916 has refined both raw beet and cane sugars with the aid of **NORIT**. The result of this visit was that it was decided at once to put a **NORIT** plant into the Radbor refinery.

This decision to instal a **NORIT** plant at Radbor was made during the beet-season of 1922-23. The necessary machinery, including the **NORIT** oven, was erected and completed in 24 days, which demonstrates clearly the ease and simplicity of the process.

NORIT replaces another type of decolorizing carbon.

In previous campaigns Radbor refined its beet sugars with the aid of another decolorising carbon (Carboraffin), which was replaced in 1922 by **NORIT**.

Many factories in Czecho-Slovakia follow suit after Radbor introduced NORIT.

Since then several sugar refineries in Czecho-Slovakia, viz.: Elbe-Kosteletz, Kuttenberg, Louny and Holice followed suit, and introduced the **NORIT** process in their factories.

Holice, which also previously refined its sugars with the aid of another decolorising carbon (Carboraffin), abandoned it and now **NORIT** only is applied there.

NORIT application requires no pre-filtration.

The application of the other decolorising carbon, which filters badly, demands a pre-filtration; and, moreover, this very expensive carbon cannot be revivified. Therefore, for economic reasons its use is necessarily limited to very small percentages, which do not realise the desired result. Filtration and decolorisation with **NORIT** is carried out in *one* operation whilst this carbon is revivified with ease, being used over and over again many times. It may, therefore, be added in larger amount.

Summary of results obtained at Radbor with NORIT.

The following summary of results of experiments made with **NORIT** at Radbor is taken from an important article by Dr. JAR. DEDEK (of the Prague Sugar Experiment Station) and ING. K. ZERT (Chemist to the Radbor sugar refinery), published in the *Zeitschrift für Zuckerindustrie der Czechoslovakischen Republik*.

(1) Decolorisation and filtration with **NORIT** in *one* operation proved to be an easy and reliable procedure. Work with **NORIT** may be compared with ordinary filter-press work Good hard carbon-cakes are obtained.

(2) Regeneration of the used **NORIT**, especially with the use of the **NORIT** oven is carried out with ease, and the oven delivers well reburned **NORIT** having absolutely satisfactory properties. The loss of **NORIT** through reburning, etc., does not exceed 0·03% of the weight of the raw sugar treated.

(3) The decolorisation obtained depends upon the percentage of **NORIT** added, and by using 1 to 1½% on the weight of sugar contained in the liquor, a constant and durable decolorisation of over 60% (up to 80%) was obtained with the first and second liquors (said liquors being treated one after the other without the intermediate revivification of the once used **NORIT**.)

Further notable points mentioned in the same article:

(4) The colour of decolorised liquors equals that of bonechar-treated liquors, and the product made from the treated liquors is irreproachably white.

(5) Cheapness of the **NORIT** plant: Most of the plant was erected from existing machinery (tanks, presses, etc.), the only item necessitating purchase being the **NORIT** oven.

(6) The cheapness of the **NORIT** carbon (its price being about 6½d. per lb. as compared with the price of the other carbon previously used, costing 1s. 4d. per lb.). The cost of regeneration including the loss of carbon, fuel and labour required, came to less than a half-penny per lb.

(7) Saving was effected as the result of the elimination of bag-filtration (and the apparatus for this operation, also for filtercloth, labour, fuel, etc.); and of the elimination of sulphitation, etc.

(8) Economy in full was effected through being able to boil sugar from denser liquors, *e.g.*, 66° Brix., whereas previously those of about 55° Brix. were worked (and from well-decolorized liquors, not necessitating repeated boilbacks). This means per 10 tons of massecuite of 90° Brix., 1·4 ton less water to be evaporated, or a saving of 200-250 kg. (¼ ton) coal, or 28% less total amount of steam necessary for boiling down to crystal.

(Continued on next page.)

Some of the Factories and Refineries in Europe using NORIT.

CZECHO-SLOVAKIA.

At Radbor for refining raw beet sugar **NORIT** only is used, likewise at *Holice*, At *Elbe-Kostelec* and *Kuttenberg* **NORIT** is applied in conjunction with bonechar for refining raw beet sugar, while Louny uses **NORIT** for the filtration and decolorisation of beet juice (syrup from the quadruple effect).

HOLLAND.

Dinteloord, previously a raw beet factory, refines raw beet and cane sugars with the aid of **NORIT** during and after the beet season during the last 8 years, thus refining the whole year round. At the Hollandia and Oud-Gastel refineries raw cane and raw beet sugars respectively are refined with **NORIT**.

BELGIUM.

The *Raffinerie Tirlemontoise* applies **NORIT** in conjunction with bonechar for refining raw beet and cane sugars ; and the *Vieux Lillo* beet sugar factory refines raw cane sugars using this process.

DENMARK.

The *Phoenix* refinery, previously using bonechar, now refines cane sugars solely with the aid of **NORIT**, producing in addition to standard white granulated, cubes and loaf sugars by the " Adant " process, and also manufacturing *Wurfel, Pilet* and press-cubes.

FRANCE.

The *Bernard Freres* refinery produces granulated sugar and cubes from beet and cane sugar by the aid of **NORIT**. This sugar-house has also refined " Jaggery," a very low class of Indian raw sugar, by the **NORIT** process. *Pithiviers* uses **NORIT** for the filtration and decolorisation of the *first* molasses, resulting from boiling beet-syrups to white granulated sugars, as well as its last after-products, which are melted without affination, and filtered and decolorised with **NORIT** in one operation. Pithiviers is producing *white yeast* from molasses only after decolorization with **NORIT**.

PORTUGAL.

NORIT is used for refining raw sugars applying the " concrete white sugar process " to manufacture from raw cane sugars only a white fine-grained sugar, like table salt, thereby producing no molasses.

ENGLAND.

Cantley beet factory applies **NORIT** for refining the *after-products*, obtained in the manufacture of white sugar, so as to boil these into a white also. The use of **NORIT** has been contracted for by many recently projected beet sugar factories. Many, including the larger refineries bought, a number of years ago, licenses for the **NORIT** rights.

Also a number of jam, chocolate, biscuit and other confectionery factories are using **NORIT** to produce white sugar liquors for use in their own manufacture.

GERMANY.

At *Klutzow* and *Walschleben* beet factories **NORIT** is applied for the same purpose, namely the treatment of the after-products for the production of white refined sugar.

Furthermore **NORIT** is used in Canada, United States of America, Hong Kong, Japan, Brazil, Venezuela, Mozambique, Philippines, etc.

GENERAL NORIT CO., LTD.

Head Office: den Texstraat 2, AMSTERDAM, Holland.

See also page iv for other information.

Notes on Sugar Curing Plant.

THE machinery for the sugar curing process which is described in chapter IV, beginning on page 237, forms a very important part of the complete factory equipment, and, owing to its special nature, has to a great extent developed into a special branch of the sugar engineering industry.

We devote ourselves entirely to this branch of engineering, comprising principally crystallizers, centrifugal machines with their accessories, such as strike or distributing mixers, conveyors, molasses and massecuite pumps, sifters, pug-mills, laboratory centrifugal machines and testers, etc.

In the manufacture of plantation white sugar by the process of double curing, the massecuite from the crystallizers is first cured in a battery of ordinary centrifugals as shown by illustration 1348, on page xxxii, or in our SELF-DISCHARGING MACHINE, for the preliminary separation of the thick molasses. If a self-discharging machine is used as in the arrangement shown by illustration 4056, opposite, it can turn out about two to five tons of sugar per hour, according to the size of the basket and to the nature of the sugar. One such machine can therefore prepare the sugar for a considerable number of ordinary centrifugals The self-discharging centrifugal is open in the bottom, and, when the sugar is free-drying, discharges its contents whenever it is stopped, without any assistance. The sugar from the foreworkers or the self-discharging machine is discharged into a mixing mill or pug mill, where it is mixed with the washings from the ordinary centrifugals, and is elevated to the feeding trough for charging the centrifugals of ordinary construction where the sugar receives its final purging and washing in the usual way.

It will be seen from this method that the two kinds of molasses are effectually kept separate, and that, to alter an existing installation to make it suitable for this process, no additional centrifugals are necessary excepting one (or two for large installations) of our self-discharging machines, and consequently much less labour is required than when the machines for the preliminary separation of the thick molasses are of the same ordinary type as used for the final washing.

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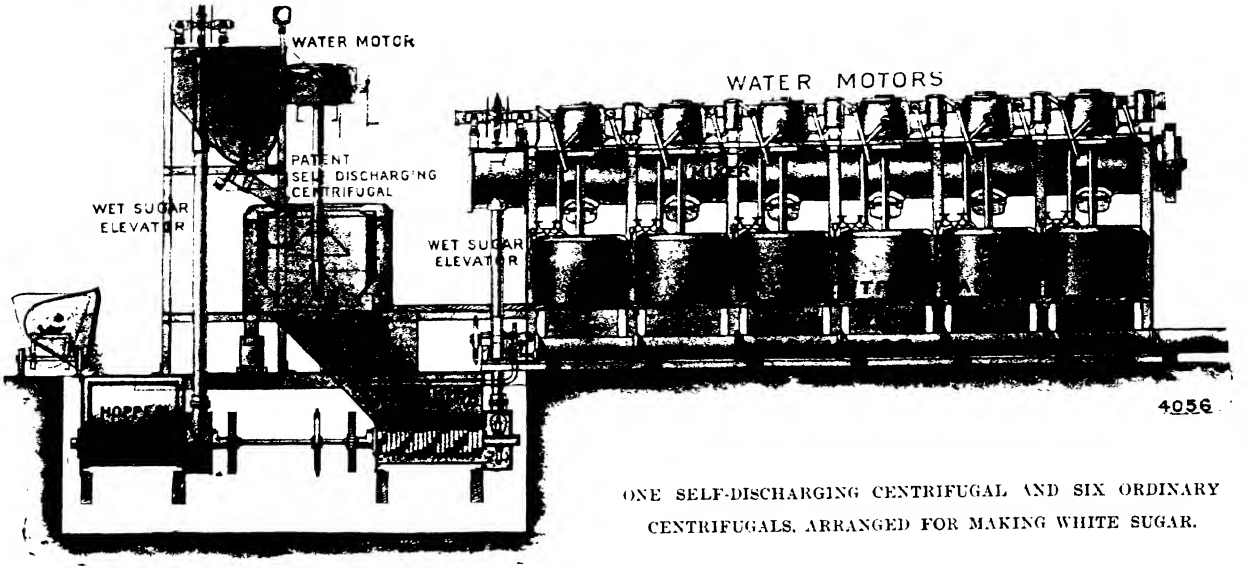

ONE SELF-DISCHARGING CENTRIFUGAL AND SIX ORDINARY CENTRIFUGALS, ARRANGED FOR MAKING WHITE SUGAR.

The self-discharging basket has no discharge valve, and it is charged while running, the massecuite being fed on to a distributing plate as shown, and thrown against the wall of the basket by centrifugal force The slope of the basket bottom is so great that the dried sugar will not lie on it when the basket is at rest, but immediately falls, by its own weight, and is discharged through the large annular opening in the bottom of the basket.

This basket is particularly suitable for raw sugar washing, as shown above, but has also been adopted for general work in refineries and factories, and, where sugar is amenable to treatment, it is the simplest method of obtaining a large output with reduced labour.

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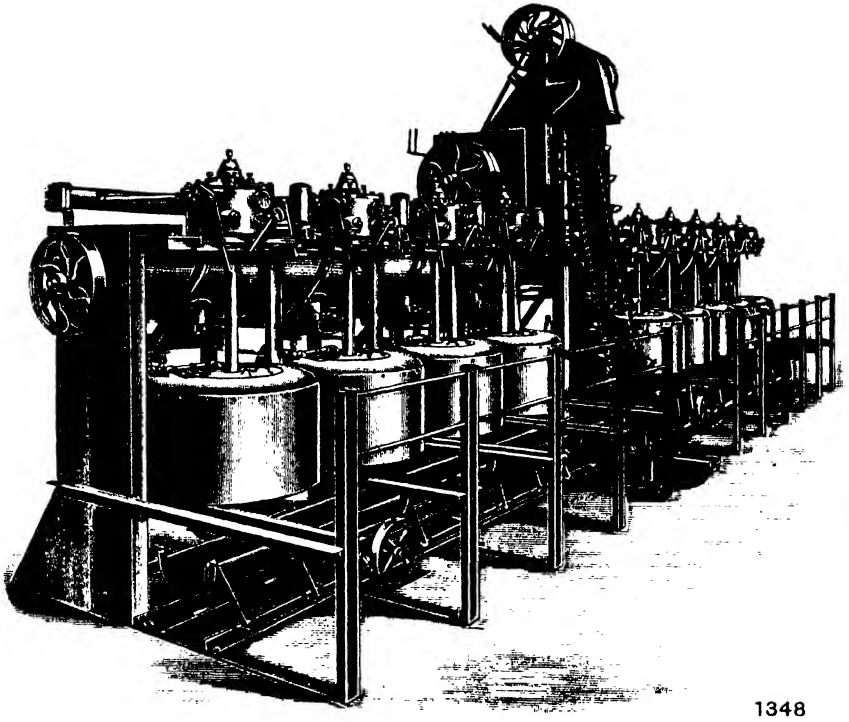

INSTALLATION OF WATER-DRIVEN CENTRIFUGAL MACHINES ARRANGED FOR THE DOUBLE-CURING PROCESS BY MEANS OF TWO BATTERIES OF CENTRIFUGALS OF ORDINARY CONSTRUCTION.

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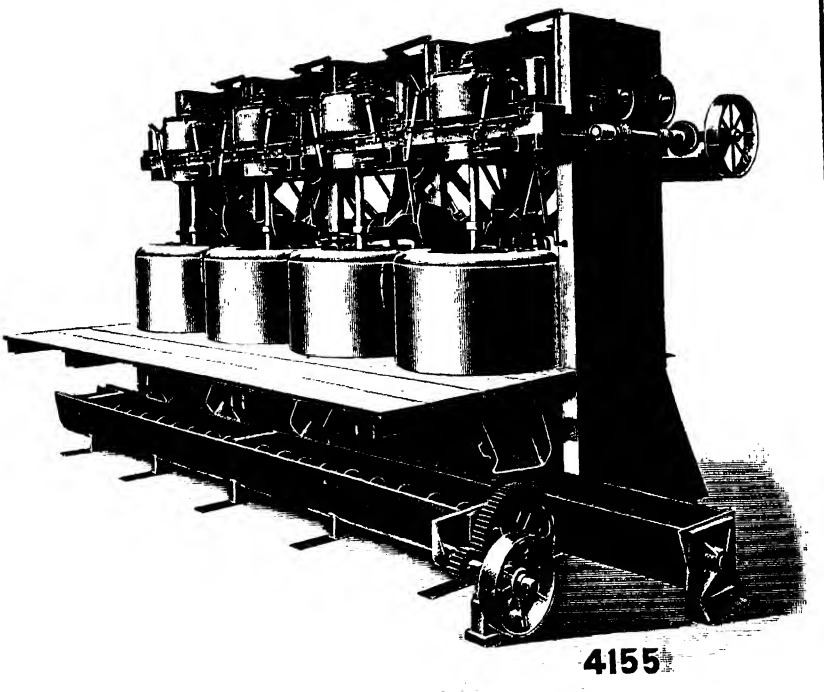

FOUR WATER-DRIVEN CENTRIFUGAL MACHINES ARRANGED WITH TWO FEEDING TROUGHS, TWO CONVEYORS AND SWIVEL SHOOTS FOR DRY SUGAR, SO THAT TWO DIFFERENT CLASSES OF SUGAR MAY BE CURED AT ONE TIME.

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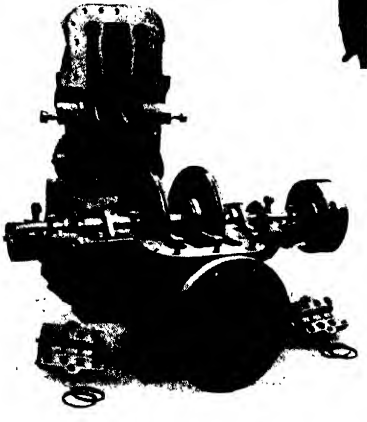

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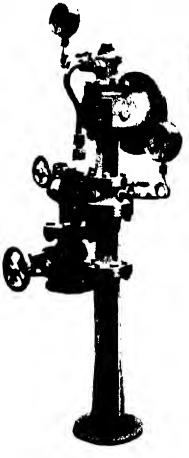

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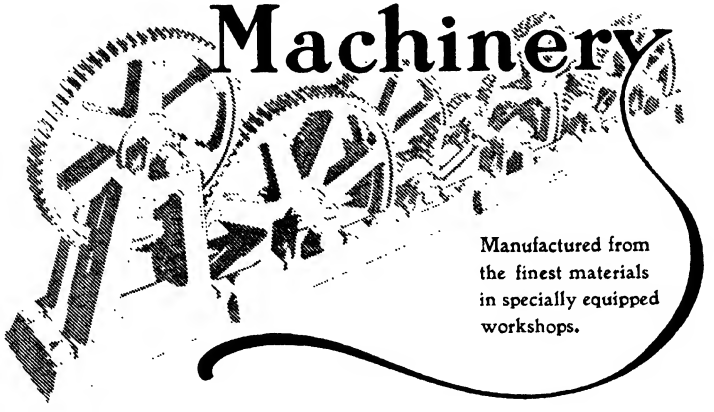
Mirrlees Sugar Machinery



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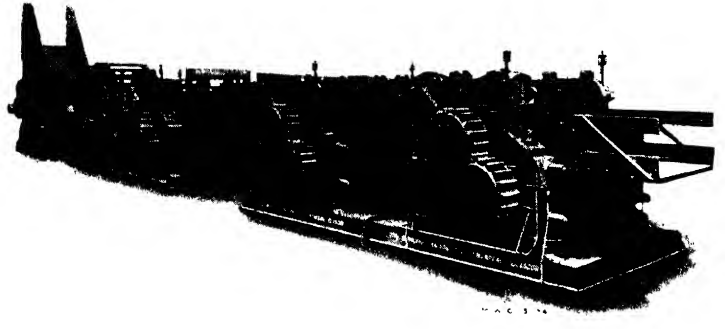

14-roller Cane Crushing Mill, part of a complete 50 ton per hour factory recently built by MIRRLEES.

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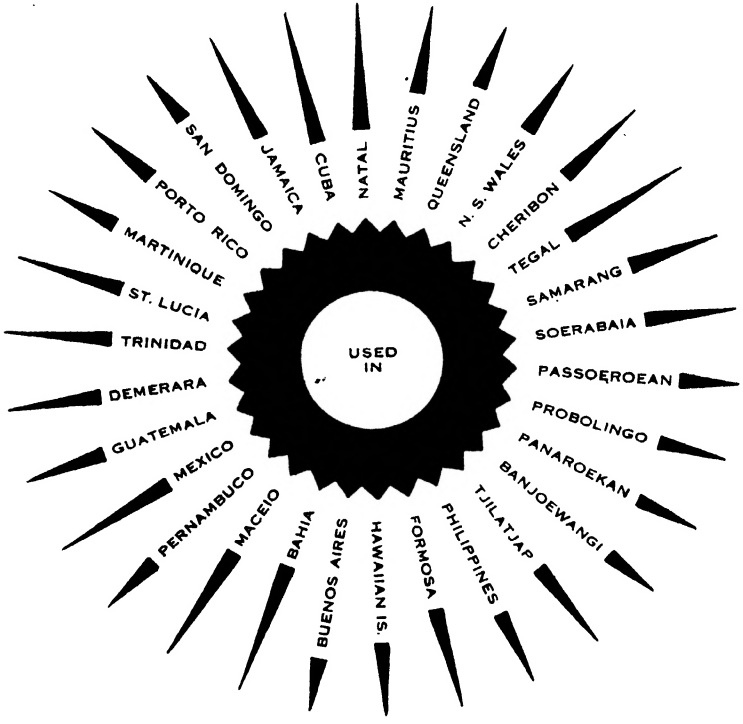

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