

QUANTITATIVE DETERMINATIONS OF SOME NON-SUGARS AND PARTIAL REMOVAL OF ONE IN PARTICULAR— STARCH

By P. N. BOYES

This paper can be divided into two sections. In the first, composited weekly samples of molasses have been analysed for certain constituents. The analysis is by no means comprehensive and those constituents were selected which might affect the viscosity and/or crystallisation of sucrose. The second is more in the nature of a progress report dealing with the removal of one of these constituents—starch.

The Analysis of Molasses

Impurities in cane juice affect clarification, crystallisation and viscosity of massecuites. The effect of these impurities is obvious to all those working in a sugar house and is reflected in the ease of recovery. It is well known at Tongaat that massecuites are "easy" to handle at the beginning of the season and become "difficult" around October/November. The only figures available are the invert sugars and sulphated ash of molasses and the syrup purity. Each of these gives its own seasonal characteristics and demonstrate to some extent the observed changes in recovery, viscosity and crystallisation. The words "gum," "wax" and "glue" are frequently heard but there have never been any actual determinations of these quantities at Tongaat. Technologists throughout the world are now aware that certain constituents e.g. amino acids, have an effect out of all proportion to their quantity on crystallisation and viscosity, and it is most important for each mill to be aware of its own major impurities throughout the season. Analyses of this type are not abstract pieces of research, since they can be used to analyse constituents in mixed juice and draw attention to poor quality cane, as well as forecasting seasonal difficulties.

Impurities are concentrated in molasses and with samples easily obtained and kept, seemed the obvious source for this investigation. Results of analyses are given in Appendix I expressed as a percentage of dry substance in molasses. It can be seen that the summation of sucrose + sulphated ash + invert sugars + gums + wax + protein gives a figure of about 92 per cent. Due to the nature of the determinations both sulphated ash and protein give a figure which is higher than the actual. The important point is

that these six quantities account for the major portion of the molasses constituents.

In order to observe trends more clearly, the sucrose was subtracted from the dry substance leaving the non-sucrose per cent in molasses. The impurities were then expressed as a percentage of non-sucrose. The figures are shown in Appendix II. Several substances—soluble silica, P_2O_5 and K_2O show no particular trend over the season, and it is concluded that their influence is constant and they do not contribute towards seasonal trends. Other substances—gums, starch + proteins give low figures at the beginning of the season, which increase slightly during the season and show a sudden jump on the W/E 28/9/57. This is consistent with factory observation where massecuites increased in stickiness over this period. It will be seen that proteins show the remarkable increase from a minimum of 6.16 per cent to a maximum of 12.20 per cent. Gums show an increase of 50 per cent and starch 25 per cent.

The wax figures are very much lower than the only other results we have for comparison,⁹ where the average for July + November molasses samples was 1.14 per cent on dry substance in 1956. It can be seen that the wax content was high at the beginning of the season in May and June, dropping to a much lower figure during the middle of the season from July to November and showing some increase towards the end of the season from December to January. The filtration in the experimental procedure is very slow and it is very noticeable how the rate varies with wax content of the molasses. The 1956 average result for the industry given in Douwes-Dekker's⁹ report was 0.48 and it can be seen from Appendix I that Tongaat's 1957 figures are sometimes lower. Since the year, from a recovery point of view, was a good one, it is possible that wax is one of the principal culprits in making our massecuites difficult to handle.

Invert sugars are by far the major constituent of the non-sucroses and demonstrate the well-known characteristic of decreasing as the season progresses and the cane ripens. This year we had the opportunity, by continuing the crushing season into January, of observing the various constituents resorting to their start-of-season values.

It was hoped that these obvious trends could be correlated with figures such as the sucrose in cane, app. syrup purity, boiling house performance or molasses per cent cane. No general clear-cut correlation was apparent and the reason for this may be partially explained when the results are subjected to a material balance. It is well known that as the season progresses, the cane ripens with corresponding increase in syrup purity and decreases in non-sucrose load to the boiling house. The molasses per cent cane is therefore a variable figure with a distinct seasonal characteristic. In Appendix III the pounds of the various molasses constituents derived from 100 tons cane are calculated for each week. It is seen that the weekly quantities for gums, starch, protein, sol. silica P_2O_5 and K_2O are remarkably constant. The invert sugars drop from 1,135 lb. to 496 lb., while the sulphated ash shows a tendency to start at 700 lb., increase to 780 lb. and drop to 650 lb. It would appear that the trends in the observed constituents are principally due to the seasonal changes taking place in the sucrose and invert contents of the cane juice. Wax, however, is another constituent which varies considerably.

The investigation will be repeated for the 1958 season and it is hoped that as more figures become available a clearer insight into the role of non-sugars can be obtained. In passing it should be mentioned that a comprehensive formula of molasses exhaustion should take into account protein and gums as well as ash and invert.

The Partial Removal of Starch by the Use of Enzymes

For a number of years refiners have complained of the poor filtrability of South African and Australian raws. There is little doubt that starch is one of the chief offenders. In the past progress was handicapped by the lack of an accurate analytical method for the direct determination of starch. With relatively new analytical procedures, determinations made on monthly Natal raws have revealed a very high starch content—much higher than some countries.² For some reason, still unanswered, cane grown in the vicinity of the Tongaat mill has a very high starch content.

The chemistry of starch is very complex^{3,4} and a recent survey of literature has been made by Buchanan.⁵ Only a few basic facts are necessary at this stage. Starches contain two basic units, α -hexa-amylase and $\alpha\beta$ -hexa-amylase. These exist in starch granules in a form polymerised by subsidiary valencies and esterified. They then occur as polymerised amylase and amyla-pectin, with amylohemiacellulose as a possible third substance. Starch can be broken down by enzymes occurring in malt to soluble starch, dextrin and maltose. These enzymes being selective will not react with other compounds normally occurring in sugar juice. Use is made of this in the brewing and baking industry where malt diastase is well known.

As far back as 1926 Haddon^{6,7} and Feuilherade⁸ were experimenting with a specially prepared imported enzyme, Ubase, for starch removal. The enzyme was very expensive and these two authors had no quantitative method for determining starch removal and so quoted no figures. The author recently tested one of these specially prepared enzymes containing a high α -amylase content which cost \$4.10 per lb. c.i.f. New York. Due to the difficult conditions under which these enzymes have to do their work it is necessary to overdose and it is not surprising to find previous work of this type to be uneconomical.

At this stage it is worthwhile noting the distribution of starch in the various process streams. Table I below is representative of Tongaat and starch is everywhere expressed as parts per million juice, syrup, sugar or massequite.

TABLE I

Starch Content of Tongaat Process Streams in ppm			
Sample	A	B	C
Sugars ...	565	1125	not determined
Molasses ...	2750	3250	6000
Massequite .	1875	3000	not determined
Magma		1850	
Crusher Juice		380	
Mixed Juice		290	
Syrup		800	

From these results it was decided to treat syrup with a suitable enzyme to remove starch prior to boiling. Artificially-made-up solutions of 50° Bx sucrose at 60°C and baker's soluble starch showed that promising results could be obtained using an imported malt. This malt sold at 1s. 3d. per lb. and using a ratio of 10 : 1 starch : malt a partial removal of 50 per cent starch was effected in the artificial solutions. The results were sufficiently promising to warrant two large-scale trials—one lasting ten hours and the second ten days.

Enzyme activity is favoured by a pH of 4.5–7.5 and the enzyme is "killed" fairly rapidly at temperatures in excess of 70°C. The pH of syrup being 6.6–7.0 and temperature 60–65°C, it was thought that reasonable results could be obtained although residence time in the syrup tanks was only half an hour. The enzyme used had a high amylase activity of 200° Lintner—178° β -amylase and 22° α -amylase.

The first trial lasted sufficiently long to boil four pans of A sugar and results were extremely good, reducing the starch from 600 ppm to 280 average in sugar. One very noticeable feature of the trial was the whiteness of the sugar produced.

The second trial was carried out in August and was in some ways disappointing, only 25 per cent starch being removed, thereby reducing the combined A and B sugars from 825 to 600 ppm.

The manufacturers of the malt became interested and from their own experiments using cane starch supplied by the S.M.R.I. and sugars from Tongaat deduced the following: Cane starch has an extremely high gelatinisation temperature of 90°C and at least 37 per cent of the A and B sugar starch contents were still in the ungelatinised state. Gelatinisation is necessary for enzyme activity. A further discovery was the increase in reaction time required for complete conversion of starch in syrups as shown below in Table II.

TABLE II
Reaction Times

Brix	Time for complete conversion minutes
15	30
25	50
50	210

From these results it is obviously better to work with lower brix juices and the natural place for further experiments was the mixed juice. Although mixed juice has a favourable pH for enzyme activity it is heated to 215°F about four minutes after leaving the mills. The enzyme is effective only after the starch has gelatinised and this requires a temperature of 90°C which is well above its survival temperature. In spite of these adverse conditions, experiments carried out in the laboratory showed surprisingly good results.

In one series mixed juice was treated with enzyme and then allowed to boil in four minutes, rapidly cooled, and the solution tested for starch. These results were then compared with samples of mixed juice which had also been boiled for four minutes but the boiling was then continued for twenty-five minutes to allow time for the starch granules to burst and gelatinise. A comparison of results is given in Table III below.

TABLE III

Enzyme Added to Boiling Mixed Juice

	Weight ratio of starch to enzyme	Starch content ppm	per cent removal
(a) No boiling	3.92 : 1	210	34
25 mins. boiling	3.92 : 1	100	66
(b) No boiling	1.33 : 1	110	65
25 mins. boiling	1.33 : 1	77.5	76
Untreated mixed juice		318	

The enzyme is apparently able to live long enough at the boiling temperature to break down a large percentage of starch. The author is unable at this stage to explain this phenomenon, since it is generally accepted that enzyme life is very short above 70°C.

Continuing the experiments, a second series was carried out which simulated Tongaat practice more closely. Mixed juice was treated, boiled, limed to 7.5 pH and settled. The supernatant liquid was then analysed for starch and the experiment carried out in triplicate—see Table IV.

TABLE IV

Treatment of Mixed Juice under Conditions
Simulating the Process

	Weight ratio of starch to enzyme	Starch ppm	per cent removal
Untreated mixed juice		240	—
Sample No. 1	3.92 : 1	40	83.5
Sample No. 2	3.92 : 1	34	85.6
Sample No. 3	3.92 : 1	40	83.5

Comparison with Table III shows that removal has been increased from 66 per cent to 84 per cent. The phosphate floc must have aided in removal of some starch.

These experiments are of an exploratory nature and optimum starch : enzyme ratios have not been determined. It is only fair to say that treatment at a ratio of 3.92 : 1 would cost a factory of Tongaat's size £400 per week. It must be accepted that enzymes only partially remove starch. It must also be realised that removal is not proportional to the amount of enzyme used. A much smaller dosage could effect the same removal. We know that refiners would be very pleased if the starch content of Natal raws could be substantially reduced, but we still have to determine how much starch removal benefits the raw sugar factory.

General

I would like to thank Mr. W. P. Paterson, Chief Chemist, Tongaat, for assistance he has given me in the molasses determinations. Also I would like to thank Mr. D. H. Hill of Havinden (Pty.) Ltd., and Mr. M. Baker-Munton of Munton and Fison Ltd., England, for their interest and co-operation.

Methods used in the Analysis of Molasses Samples

Sucrose	Jackson & Gillis Method IV.
Invert Sugars .	Lane and Eynon.
Dry Substance.	Oven-drying using graded sand—Zerban & Brown, Sugar Analysis, p. 28.
Sulphated Ash.	Methods of Analysis of the Association of Official Agricultural Chemists, 8th edition, p. 535.
Gums	Precipitation from alcohol with correction for ash. Douwes-Dekker, 31st Conf. S.A.S.T.A., p. 105 (1957). Zerban & Brown, Sugar Analysis, 3rd edition, p. 1093.
Starch	Alexander, 28th Conf., S.A.S.T.A., p. 100 (1954)
Silica	4 hrs. digestion of 5 per cent solution at 95°C. Alexander & Parrish, S.A.S.J., 37.9.573 (1953).
Wax	Alexander, 31st Conf., S.A.S.T.A., p. 71 (1957).
N,P,K,	2 mls. of 5 per cent molasses solution used for digestion. Procedure thereafter as for foliar diagnosis using the modified procedure of the Experiment Station, Mount Edgecombe. Cotton. Ind. Eng. Chem. (Anal.), 17.11.734 (1945). Lintner, Plant Physics, 19.-.77 (1944).

References

- ¹ Alexander, Proceedings S.A. Sugar Technologists' Assoc., p. 100 (1954).
- ² Inuma, Abs. Sugar, 53.2.50 (1958).
- ³ Eynon and Lane, Starch—It's Chemistry, Technology and Uses.
- ⁴ Walton, Vol. I—A Comprehensive Survey of Starch Chemistry (1928).
- ⁵ Buchanan, S.A.S.J., 42.1.33 (1958).
- ⁶ Haddon, E., S.A.S.J., p. 629, Oct. (1926).
- ⁷ Haddon, E., S.A.S.J., 12.6.345 (1928).
- ⁸ Feuilherade, Proceedings of the South African Sugar Technologists' Assoc. (1929).
- ⁹ Douwes-Dekker, 31st Conf., S.A.S.T.A., p. 94 (1957).

APPENDIX I

ANALYSIS OF MOLASSES 1957 SEASON

ALL CONSTITUENTS EXPRESSED AS A PERCENTAGE OF DRY SUBSTANCE

No.	Week ending	Dry substance in molasses per cent	Sucrose per cent	Sulphated ash per cent	Invert sugar per cent	Gums per cent	Wax per cent	Starch per cent	Protein per cent	Potash (K ₂ O) per cent	Phosphate (P ₂ O ₅) per cent	Soluble silica (SiO ₂) per cent	TOTAL*
1	18/ 5/57	77.3	42.5	16.0	25.2	5.1	1.0	0.8	4.4	4.0	0.3	0.4	94.2
2	28/ 5/57	78.0	41.0	14.5	25.2	4.9	1.0	0.9	3.8	4.2	0.3	0.3	90.4
3	2/ 6/57	79.2	42.5	15.8	25.3	5.3	0.9	0.9	3.6	3.9	0.3	0.4	93.4
4	9/ 6/57	78.5	42.3	15.9	23.6	5.2	0.8	0.8	3.8	4.0	0.3	0.4	91.6
5	16/ 6/57	79.8	41.7	15.9	21.8	5.3	0.9	0.9	4.7	4.2	0.3	0.3	90.3
6	23/ 6/57	78.7	41.9	16.1	21.5	5.4	0.9	0.9	4.6	4.3	0.3	0.3	90.4
7	30/ 6/57	76.6	42.3	17.0	21.7	5.1	0.9	0.9	3.9	4.5	0.2	0.4	90.9
8	6/ 7/57	78.7	42.3	16.8	19.9	4.6	0.7	1.0	4.6	4.2	0.2	0.3	88.9
9	14/ 7/57	79.2	43.9	16.4	19.3	5.4	0.7	0.9	5.0	4.1	0.3	0.3	90.7
10	21/ 7/57	78.7	43.5	16.1	19.0	5.4	0.8	0.9	5.0	4.3	0.3	0.3	89.8
11	28/ 7/57	78.8	42.4	16.8	19.0	5.8	0.6	0.9	5.5	4.3	0.3	0.4	90.1
12	4/ 8/57	77.7	42.6	17.6	19.1	5.6	0.5	1.0	4.0	4.3	0.4	0.4	89.4
13	11/ 8/57	78.6	44.1	17.2	19.3	5.1	0.5	0.9	4.0	4.4	0.4	0.3	90.2
14	18/ 8/57	79.3	44.6	17.0	19.5	5.2	0.4	0.8	5.5	4.2	0.4	0.3	91.2
15	24/ 8/57	79.4	42.3	17.6	21.0	4.6	0.3	0.8	5.5	4.1	0.3	0.3	91.3
16	1/ 9/57	79.4	44.1	17.4	20.5	4.5	0.3	0.8	5.5	4.6	0.3	0.3	92.3
17	7/ 9/57	79.5	42.7	17.6	19.3	4.4	0.7	0.7	5.5	4.6	0.4	0.2	90.2
18	15/ 9/57	78.6	43.7	17.9	19.3	4.8	0.5	0.9	5.4	4.7	0.3	0.3	91.6
19	22/ 9/57	79.5	43.5	17.8	19.0	4.6	0.3	0.8	4.7	3.9	0.4	0.3	89.9
20	29/ 9/57	78.9	45.9	19.3	17.2	5.5	0.6	0.7	5.8	4.8	0.4	0.4	95.3
21	6/10/57	76.2	48.2	19.3	14.5	7.0	0.5	1.0	6.0	4.5	0.4	0.3	95.5
22	13/10/57	77.7	46.7	18.5	13.7	6.5	0.5	1.0	6.4	4.9	0.4	0.4	92.3
23	20/10/57	77.1	46.3	18.1	15.1	6.1	0.6	1.0	5.9	5.3	0.4	0.4	92.1
24	27/10/57	78.7	43.6	18.8	16.0	6.1	0.6	1.1	5.6	4.7	0.4	0.4	90.7
25	3/11/57	79.5	44.6	18.9	15.9	5.7	0.6	1.0	6.8	5.7	0.4	0.3	92.5
26	10/11/57	78.6	45.4	18.8	16.1	6.2	0.7	1.1	5.6	4.5	0.4	0.3	92.8
27	17/11/57	78.3	44.6	18.4	16.8	6.2	0.7	1.1	6.2	4.1	0.4	0.4	92.9
28	24/11/57	78.8	45.0	17.7	16.9	6.3	0.7	1.0	6.6	4.5	0.4	0.4	93.2
29	1/12/57	77.7	45.0	17.6	16.6	6.3	0.5	1.0	6.0	4.8	0.4	0.3	93.1
30	8/12/57	78.3	43.3	19.2	16.8	6.4	0.8	1.0	6.3	4.5	0.4	0.4	92.8
31	15/12/57	79.8	44.5	18.6	16.7	6.3	1.0	0.9	4.7	4.4	0.4	0.3	91.8
32	22/12/57	78.6	44.6	17.2	17.8	6.2	0.9	0.9	4.7	4.2	0.4	0.4	91.4
33	29/12/57	79.6	43.3	17.0	20.7	6.0	0.7	0.9	5.1	4.3	0.4	0.4	92.8
34	5/ 1/58	79.4	41.8	15.2	21.5	6.1	0.8	0.8	5.5	4.1	0.4	0.4	90.9
35	11/ 1/58	79.4	39.7	—	23.8	5.8	0.7	0.7	5.1	4.2	0.4	0.3	—

* Summation of sucrose per cent + sulphated ash per cent + invert sugar per cent + gums per cent + wax per cent + proteins per cent

APPENDIX II

ANALYSIS EXPRESSED AS A PERCENTAGE OF NON-SUCROSE IN MOLASSES

No.	Week ending	Dry substance per cent molasses	Sulphated ash per cent molasses	Clerget sucrose per cent molasses	Non-Sucrose per cent molasses	SOME IMPURITIES EXPRESSED AS PERCENTAGE of NON-SUCROSE IN MOLASSES							DATA FOR CORRELATION PURPOSES				
						Gums per cent	Wax per cent	Starch per cent	Protein (NX 6.25) per cent	Invert per cent	SOL.SiO ₂ per cent (4 hrs. digest. at 95°C)	P ₂ O ₅ per cent	K ₂ O per cent	Sucrose in cane per cent	Apparent syrup purity	Boiling house performance	Molasses per cent cane
1	18/ 5/57	77.3	12.36	32.95	44.35	8.94	1.80	1.40	7.76	43.8	0.65	0.42	6.90	11.70	85.0	94.75	2.92
2	26/ 5/57	78.0	11.30	31.97	46.03	8.30	1.63	1.47	6.52	42.6	0.59	0.43	7.06	11.69	85.2	97.39	2.62
3	2/ 6/57	79.2	12.50	33.62	45.58	9.12	1.56	1.52	6.16	43.5	0.60	0.44	6.72	11.66	85.1	98.01	2.78
4	9/ 6/57	78.5	12.45	33.18	45.32	8.92	1.45	1.32	6.64	40.8	0.70	0.44	7.02	12.28	85.5	98.96	2.91
5	16/ 6/57	79.8	12.65	33.27	46.53	9.04	1.55	1.49	8.05	39.1	0.52	0.43	7.18	12.41	85.6	98.93	2.80
6	23/ 6/57	78.7	12.70	33.03	45.67	9.22	1.53	1.52	7.93	37.1	0.56	0.44	7.37	12.55	86.0	96.11	2.40
7	30/ 6/57	76.6	13.05	33.37	43.23	9.02	1.59	1.63	6.94	38.5	0.80	0.44	7.92	12.73	86.4	95.40	2.54
8	6/ 7/57	78.7	13.20	33.34	45.36	7.97	1.26	1.68	7.98	34.5	0.60	0.40	7.36	12.73	86.4	97.83	2.84
9	14/ 7/57	79.2	13.00	34.31	44.89	9.50	1.25	1.65	8.78	33.9	0.50	0.44	7.22	12.67	86.7	97.80	2.78
10	21/ 7/57	78.7	12.65	34.29	44.41	9.60	1.42	1.67	8.87	33.6	0.60	0.45	7.74	12.97	86.5	98.31	2.66
11	28/ 7/57	78.8	13.25	33.40	45.40	10.00	1.10	1.63	9.50	32.9	0.65	0.60	7.43	13.42	86.5	99.08	2.36
12	4/ 8/57	77.7	13.65	33.08	43.92	9.89	0.87	1.69	7.13	33.8	0.67	0.73	7.61	13.59	87.0	98.84	2.88
13	11/ 8/57	78.6	13.50	34.64	43.96	9.17	0.85	1.64	7.12	36.7	0.58	0.73	7.57	13.93	86.9	98.31	2.91
14	18/ 8/57	79.3	13.50	35.33	43.97	9.33	0.66	1.47	9.94	35.1	0.55	0.73	7.60	14.22	86.9	98.71	2.92
15	24/ 8/57	79.4	13.95	33.49	45.91	8.00	0.54	1.36	9.53	36.2	0.47	0.56	7.14	14.08	86.9	100.18	2.67
16	31/ 8/57	79.4	13.80	34.99	44.41	7.94	0.61	1.51	9.85	36.9	0.51	0.61	8.16	13.97	87.5	98.96	2.59
17	7/ 9/57	79.5	13.95	33.97	45.53	7.94	1.28	1.26	9.60	34.1	0.43	0.64	8.13	14.47	87.4	99.70	2.60
18	14/ 8/57	78.6	14.01	34.34	44.26	8.45	0.95	1.63	9.60	34.2	0.49	0.61	8.36	14.41	87.4	99.35	2.72
19	22/ 9/57	79.5	14.15	34.27	45.23	8.08	0.58	1.49	8.29	33.5	0.50	0.73	6.82	14.40	88.0	98.43	2.62
20	28/ 9/57	78.9	15.20	36.15	42.75	10.14	1.10	1.35	10.65	31.6	0.67	0.75	8.89	13.65	88.5	98.92	2.43
21	6/10/57	76.2	14.65	36.90	39.30	13.55	0.89	1.94	11.66	30.3	0.56	0.81	8.74	13.37	88.4	98.09	2.42
22	12/10/57	77.7	14.40	36.28	41.42	12.10	1.02	1.93	12.08	25.7	0.67	0.80	9.18	13.99	89.0	98.91	2.33
23	18/10/57	77.1	13.95	36.67	40.43	11.70	1.14	1.98	11.28	28.9	0.72	0.82	10.15	14.01	88.6	99.27	2.31
24	27/10/57	78.7	14.75	34.38	44.32	10.80	1.06	1.94	9.86	28.5	0.70	0.72	8.37	13.94	88.9	100.03	2.24
25	2/11/57	79.5	15.00	35.44	44.06	10.40	1.00	1.89	12.20	28.8	0.55	0.75	10.25	13.74	88.9	100.40	2.00
26	10/11/57	78.6	14.75	36.75	41.85	11.65	1.24	2.05	10.44	30.3	0.66	0.69	8.40	13.71	88.9	99.58	2.22
27	16/11/57	78.3	14.35	34.67	43.63	11.00	1.31	1.91	11.18	30.2	0.66	0.64	7.27	13.34	88.9	100.23	2.18
28	23/11/57	78.8	13.95	35.44	43.36	11.50	1.22	1.79	12.00	30.7	0.68	0.81	8.20	13.25	99.1	99.01	2.49
29	30/11/57	77.7	13.65	35.00	42.70	11.45	0.99	1.74	11.00	30.1	0.64	0.77	8.66	13.07	88.4	100.14	2.09
30	8/12/57	78.3	15.00	35.92	42.38	11.85	1.39	1.79	11.55	31.0	0.71	0.82	8.30	13.25	88.0	99.63	2.33
31	14/12/57	79.8	14.80	35.58	43.22	11.54	1.84	1.71	8.67	30.9	0.61	0.83	8.13	13.22	88.0	99.61	2.23
32	21/12/57	78.6	13.50	35.54	43.06	11.20	1.72	1.72	8.71	32.8	0.66	0.76	7.75	13.02	87.3	99.58	2.33
33	28/12/57	79.6	13.55	34.55	45.05	10.60	1.18	1.54	9.03	36.5	0.67	0.71	7.61	12.71	87.3	100.42	2.04
34	4/ 1/58	79.4	12.05	33.22	46.18	10.40	1.32	1.45	9.46	37.9	0.65	0.71	7.02	12.76	86.3	101.01	2.24
35	11/ 1/58	79.4	—	31.50	47.90	9.60	1.17	1.20	8.48	39.4	0.52	0.67	6.97	12.08	85.3	100.74	2.54

APPENDIX III

MATERIAL BALANCES OF MOLASSES CONSTITUENTS

POUNDS OF IMPURITIES IN MOLASSES PER 100 TONS CANE PROCESSED

No.	Week ending	Gums	Wax	Starch	Protein	Invert	Sol. silica	P ₂ O ₅	K ₂ O	Sulphated ash
1	18/ 5/57	231	46.7	36.4	201	1135	17.5	11.1	179	722
2	28/ 5/57	204	39.3	35.5	157	1030	14.2	10.4	170	593
3	2/ 6/57	231	39.5	38.6	156	1100	15.3	11.2	170	695
4	9/ 6/57	237	38.4	34.9	174	1080	18.3	11.6	184	725
5	16/ 6/57	236	40.4	38.9	210	970	13.5	11.2	187	708
6	23/ 6/57	203	33.6	33.4	174	815	12.3	9.6	162	610
7	30/ 6/57	198	35.1	35.9	153	845	17.5	9.7	175	665
8	6/ 7/57	206	32.5	43.3	206	890	15.5	10.2	190	750
9	14/ 7/57	237	31.2	41.2	219	885	12.4	11.2	180	723
10	21/ 7/57	227	33.6	39.4	210	794	14.1	10.6	183	675
11	28/ 7/57	214	23.6	35.0	204	705	14.0	12.7	159	625
12	4/ 8/57	232	21.9	42.6	181	856	16.9	18.4	192	788
13	11/ 8/57	238	21.6	41.8	182	880	16.9	18.6	199	786
14	18/ 8/57	239	16.9	37.6	255	902	14.1	18.6	195	788
15	24/ 8/57	196	13.3	33.4	233	855	11.4	13.9	175	745
16	1/ 9/57	179	13.5	34.8	226	844	11.7	14.0	188	716
17	7/ 9/57	188	30.2	30.0	227	796	10.3	15.1	193	725
18	15/ 9/57	203	22.8	39.2	231	823	12.2	14.7	201	763
19	22/ 9/57	192	13.6	35.2	197	792	11.8	17.3	162	740
20	29/ 9/57	211	22.8	28.0	222	657	13.9	15.6	185	740
21	6/10/57	258	17.0	36.8	222	535	10.7	15.5	166	710
22	13/10/57	234	19.6	37.3	233	496	12.9	15.4	177	672
23	20/10/57	219	12.1	36.9	210	540	13.4	15.3	190	645
24	27/10/57	215	21.1	38.6	196	565	13.9	14.8	166	662
25	3/11/57	184	17.6	33.2	215	506	9.8	13.2	181	600
26	10/11/57	217	23.1	38.2	194	563	12.2	12.9	156	655
27	17/11/57	210	24.9	36.2	212	575	12.5	12.2	138	626
28	24/11/57	249	26.4	38.8	260	664	14.7	17.4	178	696
29	1/12/57	205	17.2	31.0	197	538	11.5	13.9	155	572
30	8/12/57	235	27.6	35.4	228	611	14.1	16.3	164	701
31	15/12/57	223	35.2	33.0	167	595	11.8	16.1	157	666
32	22/12/57	226	34.5	34.5	175	657	13.3	15.4	156	630
33	29/12/57	195	21.7	28.4	166	766	12.3	13.1	140	608
34	5/ 1/58	216	27.4	30.2	196	766	13.5	14.8	145	540
35	11/ 1/58	234	28.6	29.3	206	960	12.3	16.3	170	—

Mr. A. C. Barnes said that he considered this was a very interesting paper. Immature cane contained more starch than did mature cane. It was therefore possible that much might be removed by more careful topping. **Mr. Boyes** had tackled the reduction of starch from a factory point of view but it might be possible to reduce the starch by dealing with the cane itself. He had discussed this with **Mr. Boyes** and asked him if he had been able to determine the starch in cane from any particular fields at Tongaat, for another possibility was that there might be a deficiency of trace or minor elements which might affect the amount of starch.

Dr. Dick asked about the different starch content in different varieties.

Mr. Boyes said that up to now the only work he had done was the analysis of starch in cane subjected to different amounts of fertilizer and irrigation. At present there was insufficient information to draw conclusions.

Mr. Alexander said that some determinations of the starch content of the various cane varieties had been made and that the quantities of starch found in different varieties varied considerably. N:Co.310 was found to contain even more starch than Uba of the same age. The age of the cane was important,

as on ageing, the starch content of canes tested increased and did not decrease as was assumed in some quarters. In S.M.R.I. tests most starch was found in the mature portion of the stalk rather than the green portion. He asked whether in the experiments the results of which were reported in Table III, a separate control had not been run.

Mr. Boyes replied that this had not been done, but from the data presented this could be thrashed out. From Table IV it could be seen that the clarified juice after treatment contained 40 p.p.m. of starch which would result in a syrup of 150 p.p.m. Under operating conditions the juice is boiled for three hours and still contains 800 p.p.m. starch in the syrup. Clearly therefore the removal of starch had been affected by something more than heat and flocculation and this something was obviously the action of the enzyme.

Mr. Alexander considered that perhaps the same quantity of starch would have been removed by the ordinary defecation treatment under laboratory conditions.

Mr. Hastilow said that last year Mr. Alexander had shown very little correlation between starch content and filtrability. He gave a much better correlation when wax and silica were compared with filtration rate.

Mr. Rault said that in the days of the Uba Cane it was thought that the poor work at the filter presses was due to the high wax content of Natal juices. He had tested samples of filter cakes from various mills as well as from his factory working with carbonation. Their wax content was definitely much higher than it is now. The good filtrability of carbonation sugars, could not be attributed to their freedom from starch, which was detectable in appreciable amounts.

Mr. Johnson asked if Mr. Boyes had found much difference in starch content between the A, B and C Massecuites.

Mr. Boyes referred to Table I, and quoted the figures shown for A and B Massecuites as well as for molasses.

Mr. Alexander said that it was incorrect to suggest that last year's figures proved no correlation between filtrability and starch—many other factors could influence this correlation. The influence of only the three impurities, starch, wax and silica, had been considered in tests carried out so far.

Dr. Douwes Dekker said that the figures in the paper would have to be studied and he thought that in time one might be able to draw reliable conclusions from them. The figures affecting filtration are very complicated and this subject was now being studied by the S.M.R.I. He asked Mr. Boyes if he could give any indications if the protein content depended on the time of the season. He wondered if the type of cane from certain areas was responsible for the sudden jump in protein content.

Mr. Boyes said that in 1956 he carried out tests and found that in that season the rise in protein content was more regular, rising as the season progressed. He had no figures to show if the protein content depended on the area from which the cane was drawn.

Mr. Rault said the nitrogen or its equivalent protein content of the molasses tested by him from various sulphitation and the one carbonation factory had seldom shown the present high range of figures, listed in the product of the Tongaat Factory. He wondered if the clarification by simple lime defecation was less effective in removing nitrogenous impurities from cane juice.

Dr. Douwes Dekker said that in recent comparisons simple defecation and sulphitation processes showed no appreciable difference in protein content of molasses.

Mr. Boyes said that he had never been able to reduce the starch content of the syrup in the factory long enough to study the effect on the process. From tests done previously they were not able to find any difference in the starch content of sulphitation as compared with simple defecation.

Dr. Douwes Dekker bore this out.

Mr. Barnes said that in the following paper it was shown that Tongaat was not the biggest offender as far as starch content over all sugar was concerned.

Dr. Douwes Dekker said that if we wished to find correlation between climatic factors and the condition of cane it must not be forgotten that there are certain process conditions which would affect the starch content of sugars.

Dr. McMartin said that in plants generally reducing sugars were first formed in the leaf during daylight and almost immediately transformed into sucrose and then into starch. During the hours of darkness this was transformed into sucrose and transported down into the stem. This sucrose was usually later transformed into starch because the plant could store more carbohydrate in this form. Some plants e.g., the potato, had special organs, tubers, to enable them to store starch but the sugarcane did not have this provision and differed from most plants in storing its carbohydrate as sucrose. It might be that N:Co.310 which had a high sucrose content might simply be a variety with a high carbohydrate manufacturing capacity, and hence high in starch content as well as sucrose. Australia and South Africa both produced high sucrose contents and these countries had a high starch content; this might be due to the same reason—high capacity for total carbohydrate manufacture.

Dr. Dick thought that the high starch content recorded for Formosa might be due to the popularity there of N:Co.310.