

# REPORT OF COMMITTEE ON STANDARDIZATION OF CHEMICAL CONTROL

Three meetings of the Committee for Standardization of Chemical Control were held during the year, on 28th August, 1947, 21st October, 1947 and 23rd March, 1948.

## NEW SCHMITZ TABLES.

It was realized for some time that the tables used by us for finding the sucrose or pol from saccharimeter readings and brix were slightly in error for the conditions under which we work. These old tables were apparently calculated for the old normal weight of 26.048 and a temperature of 17½°C. Nearly all saccharimeters in use in our industry are fitted with the Ventzke Scale, but a few have the new International Sugar Scale, and all new saccharimeters will probably be fitted with this latter scale. It was therefore decided to draw up two sets of tables: one applicable to the Ventzke Scale 26.026 grams and the other to the International Sugar Scale 26.000 and also to correct to 20°C. The tables were therefore calculated from the formula:—

$$\text{Sucrose or pol} = \frac{S \times N}{99.718 \times \text{Sp.Gr. } 20^{\circ}/20^{\circ}\text{C}}$$

where S = saccharimeter reading.

N = normal weight, i.e. 26.026 for Ventzke Scale and 26.000 for the International Scale.

99.718 = the weight of 100 ml. of water at 20°C. weighed in air with brass weights.

The two sets of tables were drawn up and printed in two different colours—the tables for the International Sugar Scale in red and the tables for the Ventzke Scale in green.

After the necessary consultations with the South African Sugar Association, these tables were distributed and brought into use during September, 1947.

## SAMPLING.

Because of the importance of sampling in chemical control, the revised chapter on sampling is herewith given in detail, and it is hoped that members will comment on it and suggest improvements:—

**General Remarks.** The reliability of the results of chemical control depends to a large extent on the methods of sampling. The greatest care should therefore be taken to ensure that all samples and sub-samples prepared for analyses are as nearly as possible representative of the bulk by making the amount of sample proportionate to the total amount, by paying attention to the state of cleanliness and by strict periodic supervision.

With regard to the state of cleanliness, the Committee for Standardization of Chemical Control advises that vessels and receptacles made of copper are generally very suitable for sampling of sugar products and are readily kept clean.

Sample cans should be provided in duplicate sets so that as one set is in use, the other may be cleaned.

With regard to supervision, the committee recommends that a record of all sampling devices be kept in each laboratory; that all sampling devices and receptacles be inspected at least once in twenty-four hours, and that the results of the inspection and the time when inspected be logged and kept as a permanent record of sampling conditions throughout the season.

**Cane.** The nature of cane makes it very difficult to obtain representative samples, so that hand samples of cane should only be resorted to in cases of necessity. Hand samples, are however, sometimes sent in from fields by planters to get an idea of the maturity of the cane and its sucrose content. When cane is sampled for this purpose the committee recommends that it be done by taking a large number of stools from random points in the fields and sub-sampled at the laboratory if necessary.

**Bagasse.** Since representative sampling and sub-sampling of bagasse are matters of considerable difficulty it is advised that routine sampling of bagasse for pol and moisture determinations be done at all factories in the following manner:—

A sampler in the shape of a dust shovel with a long handle should be provided. The length of the shovel edge should be one fifth of the length of the last mill discharge roller.

Regardless of chokes or other irregularities of crushing one shovelful of bagasse should be taken each quarter-hourly period from each fifth interval of length across the discharge chute as the bagasse emerges from the last mill.

The five portions should be mixed rapidly and intimately on a clean, dry wooden surface and then coned and quartered. Two opposite quarters should be rejected, and the wooden surface swept clean where these portions lay. The remaining two quarters should be intimately mixed.

From the remainder sufficient bagasse should be taken to fill completely a receptacle provided with a close fitting lid. The receptacle should hold 1200 to 1500 grams of final bagasse.

At the end of each hour four such samples should be mixed rapidly and intimately in a wooden box in the laboratory, and this should constitute the hourly sample for analyses from which 520 grams for the pol test and 100 grams for the moisture test are drawn at random.

**First Expressed Juice.** This should be continuous and automatic. The fundamental purpose of any device should be to give a continuous sample of the juice as it flows from all points along the whole width of the crusher rolls. The construction should be such that the flow is not likely to be interrupted by large pieces of bagocillo or chips of cane. The rate of delivery should be controllable to accord with the size of sample required. The whole arrangement should be of such a form that it may be readily cleaned and kept clean.

Any device or arrangement that fulfils the above conditions may be considered as suitable, but if the constructional details of the crusher housing permit, the following is particularly recommended:—

A cylindrical tap is constructed from a 6-inch length of three-quarter-inch brass tubing, one end of which is provided with a short handle to adjust the height and angle of the face of the tap which is in contact with the juice to be sampled. The tap outlet is cut at an angle of 45° and is referred to above as the face. This tap is inserted into the flow of juice and held in position by a brass housing bolted to the juice chute; this housing is provided with a packing gland to prevent any leakage of juice and is sufficiently tight to hold the tap in any desired position.

A greater or less amount of juice can be sampled by lowering or raising the tap into the flow of juice, while a greater degree of control can be obtained by turning the cut face of the tap towards or away from the flow of juice.

Angle-iron deflectors should be bolted upon the inside of the juice chute to direct the flow of juice towards the point where the tap is inserted, while some suitable form of screening should also be provided to prevent blockage of the tap by bits of cane trash etc.

Cleanliness of the apparatus is maintained by the installation of a steam system, the working head of which is two short lengths of pipe suitably drilled, and fitted "V"-shaped across the sampler and below it in such a position as to direct the steam on to the sampler but not to dilute the sample with condensed steam.

As far as possible the rate of flow and the size of the receiving container should be so proportioned that the container is not more than filled during the period of taking the sample. If it should happen

that a container is filled before the sample is completely taken, the full container should be put aside and another substituted. At the end of sampling the contents of the two containers should be well mixed together before a sub-sample is taken to the laboratory.

**Crusher Juice Sample Signalling Device.** In order to indicate to the sample taker the time when the beginning and end of any consignment of cane has reached the crusher rollers, an automatic signalling device should be installed. The following is a description of a simple device which is recommended:—

A vertical wheel (18 inches to 24 inches in diam.) is driven through a reducing gear by the carrier, either from a sprocket meshing with the chain, or from one of two successive idlers coupled together with sprockets and chains. The gearing is so proportioned that the wheel completes one quarter revolution while any part of the load on the carrier travels from a marked point before the first cane knives to the first crusher rolls. Around the periphery of the wheel, a number of small holes (quarter-inch to half-inch and about 1 inch apart) are drilled radially.

When the beginning of the consignment to be sampled is passing the marked point, a peg is placed in the hole nearest the top of the wheel. At a point 90° from this, in the direction of the travel of the wheel, there is placed an electric switch, which is tripped by the peg as it passes. This switch lights a lamp at the signalling point, and another lamp in the laboratory to indicate that the sampling bucket should be placed in position. The end of the consignment is indicated in the same way.

**Mixed Juice.** Mixed juice should be sampled at or near the point where it is weighed. It should be either continuous or proportionally representative of every scale-tank weighed. If scale-tanks are sampled, the sample should be representative of the juice at every depth in the tank, and preferably should be taken from the outflow throughout the emptying of the tank. The following is a method of doing this which is recommended wherever constructional details of the scales and receiving tanks allow:—

A semi-circular trough of about 1-inch diameter is so placed that one end is in the outflow of juice during discharge, and the other leads into a small box of about 2 gallons capacity. One such trough is provided for each scale-tank, both discharging into the same box. The diameter of the trough and the size of the box are so proportioned that the latter is nearly filled during one complete discharge of a scale-tank. When discharge is completed the contents of the box are stirred and a portion removed by means of a cup or a stick. The box is then emptied

by means of a plug provided for this purpose. The same amount should be taken to represent each full scale tank and an adequate portion for each partly-filled tank.

**Last Expressed Juice.** A catch sample should be drawn at the same time as the bagasse is sampled along the full length of the roller and the juice sub-sampled in the laboratory.

**Sulphited Mixed Juice.** A catch sample of mixed juice for acidity and sulphur dioxide determination should be taken from the juice tempering tanks.

**Clarified Juice and Filtered Juice.** These samples should be taken automatically wherever possible, or catch samples may be taken at frequent intervals.

**Filter Cake.** This sample should be taken from the truck which is receiving the filter cake.

**Syrup.** This sample should be taken automatically wherever possible or catch samples may be taken immediately before the syrup enters the pans.

**Masseccuite (from pan).** A catch sample is required as the masseccuite comes from the pan, avoiding the first runnings and steamings.

**Masseccuite (from crystallizer).** A catch sample from the top of the crystallizer is taken as required.

**Molasses.** (a) *Run-off Molasses from centrifugals.* A catch sample is required as nearly as possible representative of the bulk of the run-off.

(b) *Molasses prepared for boiling.* A catch sample is required.

(c) *Molasses for sale.* A representative continuous or catch sample should be taken as each tank car is being filled.

(d) *Final Molasses.* Samples should be taken from the discharged side of the molasses pump (or from the scales if molasses is weighed) continuously, or catch samples should be taken as frequently as required for representative sampling.

**Sugar.** The importance of a correct procedure for sampling of raw plantation white or refined sugar cannot be over emphasized. Whether the sample is used for the purpose of recovery calculations or for the assessment of value in commercial transactions, any carelessness or error will completely vitiate the most careful work of the analyst.

The two primary requirements are that:—

1. The sample shall be thoroughly representative, and
2. After the sample is taken, it shall undergo no change until used for analysis.

The actual procedure used will vary according to whether the sugar is sampled:—

- (1) as it is produced,
- (2) on filling the package,
- (3) from the closed package.

1. It is general to sample sugar from each lot or strike. Mechanical samplers can be devised for "chain and bucket" elevators, "grasshopper" conveyors, or hoppers or chutes. Their form will naturally vary according to the conditions and the equipment in use. The principles to govern their design and use are covered in requirements 1 and 2 above, but the following points should be observed—

- (a) In any intermittent sampling device the portion taken at each operation shall be at least in the proportion of 10 grams per 200 lbs. of sugar.
- (b) The receptacle into which the sample portion is delivered shall not have a capacity greater than 5 Kgms of sugar, and shall have an opening to atmosphere no greater than 3 sq. cms.
- (c) The container shall be emptied, or replaced, at least as frequently as every 1½ hours or for every 50 tons of sugar sampled.
- (d) The time elapsing between the emptying or replacing of a receptacle from a mechanical sampling device and the mixing and sub-sampling of the sugar contents, shall not be greater than one half-hour.

(2) The sugar sampled at the time of filling the package can best be taken by the workman at the scale or the closing machine. Again the amount taken per package shall be at least in the proportion of 10 gms. per 200-210 lbs. of sugar.

The samples shall be placed in a seamless receptacle 10½ inches high and 7 inches in diameter, fitted with a funnel shaped concave lid with a hole 1¼ inches in diameter. The funnel shall have a depth of 1¾ inches.

3. Samplings from a closed package, in the case of jute, hessian or cotton bags, shall be done with a trier. The trier shall be the "short" trier as used by the United States Treasury Department and specified by the United States Bureau of Standards. The dimensions of the trier shall be as follows:—

Length overall ... ..	40.6 cms.
Length to spoon ... ..	22.9 cms.
Length to shank ... ..	17.8 cms.
Length of handle ... ..	26.7 cms.
Width of spoon... ..	2.7 cms.
Depth of spoon... ..	0.8 cms.
Diameter of handle ... ..	3.8 cms.

An illustration of the trier will be found on page 506 of "Cane Sugar Handbook" Spencer and Meade. Eighth Edition.

In taking the samples, 100 per cent. of the packages should be sampled; the trier should be plunged into the middle of the package and withdrawn filled with sugar. If the trier has the correct length, all the layers of sugar, from the outside to the centre of the package should be correctly represented. Great care should be taken not to obtain a surface sample, because of the variations caused by drying or absorption of moisture.

The total contents of each trier should be emptied into a receptacle as described under (2), and the trier left clean for the next sample, the whole operation being conducted as rapidly as possible.

The unit of sample may be a strike, lot or truck-load, as circumstances warrant.

After taking the sample the receptacle should be conveyed to the laboratory and the contents mixed and sub-sampled within one half-hour of completion of the sample.

*Mixing and Sub-Sampling.* The contents of one, or at the most three, of the above receptacles shall be mixed and sub-sampled with the utmost despatch, to avoid changes in moisture content.

The contents of the receptacle are passed through a wire screen of three-eighths-inch mesh onto a metal top table. All lumps on the screen are broken up and added to the sample. Bits of bag, fibre, bagasse, string etc., are discarded.

Mixing should be by the standard procedure of "coning and quartering," cones between each quartering being made and flattened three times, leaving the final two quarters of such quantity as to *completely fill* a screw cap bottle, or a half pint milk bottle with a new waxed disc closure, holding about 250-275 gms. sugar. The final two quarters should therefore amount to no more than 350 gms. sugar.

Any further mixing and sub-sampling of the bottles from a number of "lots" shall be done in the above manner, omitting the screening.

The analyst should discard the top 20-30 gms. of sugar before weighing a portion for analysis.

### EXPERIMENTAL.

**Mixed Juice in Scale-tanks.** Some experiments were undertaken by members of the committee to explore the possibilities of horizontal layers of juice being found in the mixed juice scale-tanks and to what extent these layers may differ in density and sucrose.

Five samples of mixed juice were taken from three tanks whilst being emptied from the scales at Natal Estates and the saccharimeter readings taken.

The results were as follows:—

First tank.	Second tank.	Third tank.
61.4	51.6	54.0
61.4	51.6	54.0
61.5	51.5	53.9
61.4	51.6	54.0
61.4	51.6	54.0

Other results obtained are:—

#### Corrected Brix of Mixed Juice at different levels in Juice Scale-tanks (Howe scales) at Esperanza.

Top*... 7.10	Top ... 15.70	Top ... 15.36
Centre* 10.32	Centre 15.60	Centre 15.26
Bottom*13.96	Bottom 15.50	Bottom 15.16
Top ... 15.12	Top ... 15.60	Top ... 16.06
Centre 15.42	Centre 15.70	Centre 15.96
Bottom 15.76	Bottom 16.20	Bottom 16.16
Top ... 14.22	Top ... 14.11	Top ... 15.46
Centre 14.52	Centre 14.11	Centre 15.06
Bottom 14.62	Bottom 14.13	Bottom 15.26
Top ... 15.42	Top ... 15.74	Top ... 15.62
Centre 15.12	Centre 15.74	Centre 15.52
Bottom 14.87	Bottom 15.90	Bottom 15.42

\* (Mill Stoppage)

#### Analysis of Mixed Juice in different levels in Juice Scale-tanks at Esepranza.

Test No.	Corrected brix.	Per cent. sucrose.	Purity.
1. Bottom	16.44	14.73	89.6
Centre ...	16.34	14.68	89.8
Top ...	16.54	14.78	89.4
2. Bottom	15.94	14.32	89.8
Centre ...	15.67	14.13	90.2
Top ...	15.44	13.81	89.5
3. Bottom	14.94	13.32	89.2
Centre ...	14.84	13.27	89.4
Top ...	14.64	13.10	89.5
4. Bottom	15.77	14.10	89.4
Centre ...	16.17	14.46	89.4
Top ...	16.17	14.44	89.3
5. Bottom	15.07	13.56	90.0
Centre ...	15.17	13.58	89.5
Top ...	15.37	13.78	89.7
6. Bottom	15.31	13.78	90.0
Centre ...	15.57	13.91	89.3
Top ...	15.57	13.98	89.8
7. Bottom	13.97	12.43	89.0
Centre ...	14.17	12.60	88.9
Top ...	14.27	12.80	89.7
8. Bottom	14.37	12.73	88.6
Centre ...	14.17	12.51	88.3
Top ...	14.07	12.33	87.6

Test No.	Corrected brix.	Per cent. sucrose.	Purity.
9. Bottom ...	12.91	10.86	84.1
Centre ...	12.51	10.60	84.7
Top ...	12.21	10.32	84.5
10. Bottom ...	16.14	13.92	86.2
Centre ...	16.31	14.19	87.0
Top ...	16.24	14.10	86.8

**Samples taken half-hourly at Z.S.M. & P. and composited.**

Analysed once every four hours. Average daily analyses shown in following table.

**Analysis of Mixed Juice in Juice Scale-tanks at Sezela.**

Test No.	Corrected brix.	Per cent. sucrose.	Purity.
Top ...	14.9	13.26	89.0
Middle ...	14.9	13.46	90.3
Bottom ...	14.8	13.36	90.3
Top ...	15.3	13.94	91.1
Middle ...	15.4	13.97	90.7
Bottom ...	15.4	13.94	90.5
Top ...	13.6	11.72	86.2
Middle ...	14.2	12.33	86.8
Bottom ...	14.3	12.47	87.2
Top ...	15.6	14.07	90.2
Middle ...	15.7	12.14	89.9
Bottom ...	15.6	14.16	90.8

Last expressed juice.		Total last mill juice.		Purity difference.	
Brix.	Purity.	Brix.	Purity.		
4.07	77.4	2.20	73.6	3.8	
3.80	79.2	2.15	75.3	3.9	
3.43	78.1	2.28	74.6	3.5	
3.44	79.1	2.09	73.7	5.4	
3.95	79.7	2.36	76.7	3.0	
3.57	79.8	2.52	76.7	3.1	
3.83	79.4	2.24	77.7	1.7	
4.43	77.6	2.24	75.9	1.7	
4.27	78.9	2.17	76.0	2.9	
4.17	79.4	2.35	76.2	3.2	
4.14	78.7	2.34	76.5	2.2	
4.10	80.2	2.27	76.2	4.0	
4.28	79.0	2.25	78.7	0.3	
3.87	78.3	2.15	74.2	4.1	
3.72	78.0	2.10	76.2	1.8	
4.35	79.5	2.14	77.6	1.9	
4.05	79.5	2.16	73.6	5.9	
4.21	78.6	2.09	74.6	4.0	
4.15	79.0	2.21	78.7	0.3	
4.12	78.6	2.25	74.7	3.9	
4.02	77.6	2.35	75.3	2.3	
3.52	78.5	2.05	75.9	2.6	
3.79	76.8	2.06	72.3	4.5	
3.97	78.3	2.21	76.9	1.4	
3.82	89.3	2.29	76.0	3.3	
3.74	79.7	2.24	78.1	1.6	
3.62	79.0	2.14	78.0	1.0	
3.93	78.9	2.24	75.0	3.9	
3.80	78.2	2.23	77.1	1.1	
3.35	78.8	2.10	78.1	0.7	
3.61	78.1	2.30	73.5	4.6	
3.80	78.9	2.22	76.1	2.8	
3.47	78.1	2.25	74.7	3.4	
3.58	78.5	2.30	77.0	1.5	
3.92	77.3	2.24	75.4	1.9	
3.74	77.0	2.19	76.2	0.8	
3.79	76.5	2.00	74.0	2.5	
4.14	78.7	2.17	73.7	5.0	
3.90	79.2	2.26	75.2	4.0	
3.52	78.4	2.07	73.9	4.5	
3.73	79.1	2.10	77.6	1.5	
3.40	78.5	2.28	72.4	6.1	
Average	3.86	78.6	2.21	75.71	2.89

There are appreciable differences in brix and sucrose of juices from different layers in the same scale-tank. An exceptionally wide range in density was found in the first test at Esperanza where a mill stoppage occurred during the period of the test. It is therefore obvious that in order to get a true sample from each tank, it is necessary to take proportionate quantities of all the layers in the tank. The procedure recommended for sampling mixed juice will ensure this. There does not seem to be any definite trend however, and low results can be obtained at the bottom, middle or top of the tank depending on the analysis of the incoming juice. It is therefore likely that this type of error will cancel out over a period of time and where the juice flow was rapid such as at Natal Estates the contents of the scales were very uniform.

**Last Expressed and Last Mill Juice.** The recommendation for sampling last expressed juice, of course, applies to the juice expressed by the top and the last roller and not to juice from the last mill as a whole. This mistake has been made in the past at some factories, and purities lower than that of the last expressed juice reported. Some tests were done at both Z.S.M. and P. at Empangeni and at Esperanza during the past season, to see to what extent the juices differ. The following results were obtained:

*N.B.*—Total juice sample taken in all cases immediately after sampling the discharge roller.

## REYNOLDS BROS. LTD., ESPERANZA.

TOTAL LAST MILL JUICE.							LAST EXPRESSED JUICE.						
Test No.	Corrected Brix.	Per cent. Sucrose.	Purity.	pH.	Test No.	Corrected Brix.	Per cent. Sucrose.	Purity.	pH.				
1	...	1.82	1.30	71.4	6.1	1	...	2.98	2.29	76.9	5.7		
2	...	2.22	1.60	72.1	5.6	2	...	4.00	3.19	79.8	5.3		
3	...	2.07	1.44	69.6	5.9	3	...	3.50	2.57	73.4	5.3		
4	...	2.16	1.47	68.1	5.8	4	...	3.92	2.79	71.2	5.5		
5	...	2.02	1.47	72.8	6.2	5	...	3.53	2.75	77.9	5.8		
6	...	1.97	1.35	68.6	6.1	6	...	3.20	2.39	74.7	5.8		
7	...	1.87	1.23	65.8	6.0	7	...	3.20	2.34	73.1	5.7		
8	...	2.12	1.46	68.9	5.9	8	...	3.80	2.93	77.1	5.6		
9	...	2.11	1.47	69.7	5.9	9	...	2.97	2.19	73.7	5.7		
10	...	2.07	1.48	71.5	6.2	10	...	3.60	2.65	73.6	5.6		
11	...	2.13	1.48	69.5	6.0	11	...	3.25	2.47	76.0	5.8		
12	...	2.01	1.46	72.7	6.3	12	...	2.82	2.12	75.2	5.9		
13	...	1.83	1.23	67.2	6.2	13	...	2.96	2.17	73.3	5.8		
14	...	1.93	1.42	73.6	5.9	14	...	3.56	2.73	76.7	5.6		
15	...	1.77	1.23	69.5	6.0	15	...	2.91	2.14	73.5	5.6		
16	...	1.75	1.23	70.3	6.0	16	...	3.21	2.42	75.4	5.1		
17	...	2.17	1.55	71.4	5.8	17	...	3.51	2.58	73.5	5.5		
18	...	1.97	1.35	68.5	6.1	18	...	3.13	2.32	74.1	5.8		
19	...	1.81	1.26	69.6	6.3	19	...	2.63	1.93	73.4	5.9		
20	...	2.27	1.60	70.5	6.0	20	...	3.48	2.63	77.1	5.7		
Average	...	2.00	1.40	70.00	6.02	Average	...	3.31	2.48	74.93	5.64		

These tests bear out results obtained at Empangeni some years ago, when the average purity of the last expressed juice for the whole season was found to be 80.5 compared with an average of 75.0 purity for the last mill juice.

**Filter Cake.** It was felt that samples of filter cake can best be taken from the truck into which it was discharged. Mr. Rault submitted some figures to illustrate the variability of sucrose in filter cake if the samples are taken across a rotary filter. Four samples of filter cake were taken across a Mauss filter and each analysed.

Average of four tests.	Highest result.	Lowest result.
0.80	1.75	0.14
0.51	1.40	0.14
1.05	1.75	0.42
0.91	1.68	0.63
0.89	1.19	0.70
1.03	1.40	0.63
0.79	1.05	0.49
0.82	2.10	0.14
1.21	2.73	0.56
0.37	0.56	0.21
0.84	1.26	0.42

The question arose how long a sample of filter cake will keep before deterioration will seriously affect the sucrose result. This will, of course, depend on the type of cake, and the conditions at the factory, but the following results may be of interest. On keeping samples of Oliver Campbell filter cake from the Maidstone factory for eight hours no difference in pol could be found. The same was found for this type of filter cake at Esperanza, as the following results will show:—

2/9/47.		3/9/47.		4/9/47.		5/9/47.		5/9/47.		6/9/47.	
Time.	Per cent. sucrose.										
8. 0 a.m.	1.6	8. 0 a.m.	0.5	6. 0 p.m.	0.6	12.30 p.m.	0.2	5. 0 a.m.	0.1	6. 0 p.m.	0.5
8.30	1.6	8.30	0.5	6.30	0.6	1. 0	0.2	5.30	0.1	6.30	0.4
9. 0	1.5	9. 0	0.5	7. 0	0.6	1.30	0.2	6. 0	0.1	7. 0	0.4
9.30	1.9	9.30	0.4	7.30	0.6	2. 0	0.3	6.30	0.1	7.30	0.4
10. 0	1.7	10. 0	0.5	8. 0	0.6	2.30	0.2	7. 0	0.1	8. 0	0.4
10.30	1.5	10.30	0.5	8.30	0.8	3. 0	0.2	7.30	0.1	8.30	0.4
11. 0	1.7	11. 0	0.5	9. 0	0.9	3.30	0.3	8. 0	0.2	9. 0	0.5
11.30	1.3	11.30	0.5	9.30	0.7	4. 0	0.2	8.30	0.1	9.30	0.4
12. 0 noon	1.5	12. 0 noon	0.5	10. 0	0.7	4.30	0.2	9. 0	0.1	10. 0	0.5
12.30 p.m.	1.5	12.30 p.m.	0.3	10.30	0.6	5. 0	0.3	9.30	0.1	10.30	0.4
1. 0	1.5	1. 0	0.5	11. 0	0.7	5.30	0.3	10. 0	0.1	11. 0	0.4
1.30	1.6	1.30	0.5	11.30	0.7	6. 0	0.2	10.30	0.2	11.30	0.4
2. 0	1.6	2. 0	0.5	12. 0 a.m.	0.7	6.30	0.2	11. 0	0.1	12. 0 m.n.	0.5
2.30	1.7	2.30	0.3	12.30 m.n.	0.7	7. 0	0.3	11.30	0.1	12.30 a.m.	0.4
3. 0	1.5	3. 0	0.5	1. 0	0.6	7.30	0.2	12. 0 noon	0.2	1. 0	0.4
3.30	1.6	3.30	0.5	1.30	0.6	8. 0	0.3	12.30 p.m.	0.1	1.30	0.4
4. 0	1.5	4. 0	0.5	2. 0	0.6	8.30	0.2	1. 0	0.1	2. 0	0.4

It was decided at Esperanza to continue the tests until a zero pol was obtained. The results were interesting. In six tests the time varied from ten to twenty-six hours. In nearly all cases it was found that once a definite drop in pol started, deterioration

was very rapid. Thus in the first test a sample of filter cake analysing 0.5 per cent. sucrose still gave 0.4 per cent. after twenty-one hours, but five hours later no pol was obtained.

(Reynolds Bros. Ltd., Esperanza). OLIVER FILTER CAKE TESTS.

No. 1 Test.		No. 2 Test.		No. 3 Test.		No. 4 Test.		No. 5 Test.		No. 6 Test.	
Time.	Per cent. sucrose.										
3.30 p.m.	0.5	5. 0 a.m.	0.5	8. 0 p.m.	0.4	9. 0 a.m.	0.6	4. 0 a.m.	0.3	12. 0 m.n.	0.6
4.30	0.4	6. 0	0.5	9. 0	0.3	10. 0	0.5	5. 0	0.4	1. 0 a.m.	0.6
5.30	0.4	7. 0	0.6	10. 0	0.3	11. 0	0.6	6. 0	0.4	2. 0	0.6
6.30	0.4	8. 0	0.5	11. 0	0.2	12. 0 noon	0.6	7. 0	0.4	3. 0	0.6
7.30	0.4	9. 0	0.5	12. 0 m.n.	0.1	1. 0 p.m.	0.6	8. 0	0.4	4. 0	0.6
8.30	0.6	10. 0	0.4	1. 0 a.m.	0.1	2. 0	0.6	9. 0	0.4	5. 0	0.6
9.30	0.6	11. 0	0.6	2. 0	0.1	3. 0	0.6	11. 0	0.3	6. 0	0.6
10.30	0.5	12. 0 noon	0.5	3. 0	0.05	4. 0	0.5	11. 0	0.4	7. 0	0.6
11.30	0.4	1. 0 p.m.	0.6	4. 0	0.05	5. 0	0.5	12. 0 noon	0.3	8. 0	0.6
12.30 a.m.	0.5	2. 0	0.5	5. 0	0.05	6. 0	0.4	1. 0 p.m.	0.4	9. 0	0.6
1.30	0.6	3. 0	0.5	6. 0	0.0	7. 0	0.4	2. 0	0.5	10. 0	0.5
2.30	0.5	4. 0	0.5	—	—	8. 0	0.3	3. 0	0.5	11. 0	0.7
3.30	0.5	5. 0	0.4	—	—	9. 0	0.3	4. 0	0.4	12. 0 noon	0.7
4.30	0.5	6. 0	0.3	—	—	10. 0	0.3	5. 0	0.4	1. 0 p.m.	0.7
5.30	0.5	7. 0	0.0	—	—	11. 0	0.0	6. 0	0.4	2. 0	0.7
6.30	0.5	—	—	—	—	—	—	7. 0	0.2	3. 0	0.6
7.30	0.5	—	—	—	—	—	—	8. 0	0.1	4. 0	0.5
8.30	0.5	—	—	—	—	—	—	9. 0	0.0	5. 0	0.2
9.30	0.5	—	—	—	—	—	—	—	—	6. 0	0.1
10.30	0.5	—	—	—	—	—	—	—	—	7. 0	0.0
11.30	0.4	—	—	—	—	—	—	—	—	—	—
12.30 p.m.	0.4	—	—	—	—	—	—	—	—	—	—
1.30	0.3	—	—	—	—	—	—	—	—	—	—
2.30	0.2	—	—	—	—	—	—	—	—	—	—
3.30	0.1	—	—	—	—	—	—	—	—	—	—
4.30	0.1	—	—	—	—	—	—	—	—	—	—
5.30	0.0	—	—	—	—	—	—	—	—	—	—
26 hours		14 hours		10 hours		14 hours		17 hours		19 hours	

**Trash and Tops.** Preliminary experiments on the sampling of trash and tops in consignments of cane were carried out, but unfortunately the number of tests done on a single consignment were not enough to give an estimate of the standard error of sampling. Where tests were done in duplicate, using at least 5 lbs. of sample per ton of cane and not less than 100 lbs. sample per consignment, the estimates agreed reasonably well. It was found that the labour requirements for estimating tops and trash were about 1 to 1½ man-hours per 100 lb. sample.

**INVERTASE.**

Samples of the commercial invertase concentrate were obtained during the season and it was immediately taken into use at Darnall where the rapid invertase method of sucrose determination (inversion at 55—60°C.) was compared with the Jackson and Gillis No. 4 method under routine conditions on all mixed juice samples. It was found that the invertase method could be used quite conveniently for routine purposes, but it did involve the extra work of deleading all samples and taking the refractometer brix of the inverted solution. There were no real differences in sucrose per cent. mixed juice between the two methods as the following results will show:—

**Darnall, Mixed Juice Analyses, Sucrose per cent. Juice. Comparison between Jackson and Gillis No. 4 and Rapid Invertase methods from 8th September, 1947, to 2nd December, 1947.**

J. & G.	In-vertase.	J. & G.	In-vertase.	J. & G.	In-vertase.
12.13	12.11	12.25	12.25	12.44	12.41
12.49	12.49	12.29	12.29	12.64	12.63
12.29	12.27	12.87	12.84	12.53	12.53
12.41	12.41	12.57	12.55	11.21	11.21
12.45	12.39	12.62	12.62	11.85	11.88
11.97	12.03	13.04	13.04	10.98	10.95
12.73	12.71	12.63	12.60	11.93	11.93
12.90	12.95	12.69	12.69	11.88	11.88
12.26	12.28	13.51	13.53	12.21	12.18
12.64	12.64	12.29	12.29	11.90	11.82

J. & G.	In-vertase.	J. & G.	In-vertase.	J. & G.	In-vertase.
12.48	12.54	12.86	12.86	12.74	12.76
12.68	12.70	12.85	12.85	11.69	11.67
12.92	12.95	12.89	12.89	11.55	11.57
12.90	12.87	13.17	13.17	12.49	12.51
12.39	12.39	11.68	11.70	12.40	12.37
12.14	12.14	11.89	11.92	12.29	12.35
12.74	12.72	12.12	12.16	12.12	12.12
12.80	12.80	11.50	11.50	11.95	11.98
12.82	12.80	11.89	11.89	12.50	12.50
12.76	12.76	12.03	12.09	11.86	11.89
12.60	12.60	11.90	11.90	12.27	12.25
12.32	12.29	12.58	12.56	12.15	12.12
12.22	12.21	12.33	12.43	12.11	12.11
12.03	11.96	11.76	11.76	12.47	12.47
12.16	12.16	12.46	12.44	11.90	11.93
11.90	11.88	12.21	12.24	11.36	11.34
11.19	11.17	13.06	13.06	10.90	10.92
12.20	12.17	13.16	13.23	11.06	11.06
10.89	10.92	11.93	11.93	11.89	11.92
11.98	12.00	12.12	12.10	12.71	12.71
11.35	11.35	12.13	12.13	12.05	12.05
11.19	11.19	11.37	11.39	12.32	12.37
11.60	11.63	11.85	11.88	12.51	12.49
12.42	12.40	11.71	11.69	11.88	11.91
11.45	11.48	10.75	10.78	12.08	11.98
11.85	11.90	12.03	12.03	11.60	11.60
11.61	11.64	12.21	12.18	11.89	11.87
12.98	12.96	11.65	11.65	11.85	11.85
12.74	12.72	11.98	11.94	11.13	11.16
12.81	12.83	11.38	11.34	11.90	11.88
12.15	12.20	11.46	11.42	10.74	10.80
12.51	12.52	11.61	11.62	10.70	10.79
12.10	12.10	11.75	11.76	11.24	11.21
12.79	12.79	11.40	11.32	10.98	10.99
12.56	12.56	12.63	12.67		

Total No. J. & G. Samples=134 Av.12.12.  
Total No. Invertase Samples=134 Av.12.12.

Samples of molasses from various factories were also analysed by these two methods at Darnall. Again the differences were small. The average results from the 16 factories were in fact again identical for the two methods.

Factory ...	UF.	EM.	FX.	AK.	DL.	NG.	GL.	CK.	NE.	TS.
Sucrose per cent. J. & G. method No. 4 ...	38.11	40.48	37.62	36.12	36.42	34.21	34.58	33.74	37.05	38.20
Sucrose per cent. Rapid invertase method ...	38.32	40.55	37.41	35.72	36.02	34.71	34.66	33.84	37.13	38.30
Factory ...	IL.	RN.	ES	SZ.	UK.	MV.	Average of 16 factories.		Ref. Molasses.	
Sucrose per cent. J. & G. Method No. 4...	36.63	37.00	34.04	35.93	34.63	34.04	36.18		37.74	
Sucrose per cent. Rapid invertase method ...	36.40	36.93	34.22	36.03	34.38	34.21	36.18		38.14	

The activity of the invertase preparation was determined at the Experiment Station and k, the velocity coefficient, measured according to the method of the Association of Official Agricultural

Chemists. One bottle of Sumasuco invertase concentrate dated 15/4/47 was kept in a refrigerator from the time of its arrival. Another bottle of the same date was kept at room temperature. There

was a slight mycelial growth in this bottle but the invertase solution which was kept in the refrigerator was still clear in March 1948. Periodic determinations of the activity of the invertase solutions gave the following values for K.

Date.	Invertase kept in refrigerator.	Invertase kept at room temperature.
7th July, 1947 ...	—	0.472
25th August, 1947 ...	0.469	0.462
17th January, 1948 ...	0.488	0.457
6th March, 1948 ...	0.475	0.435

The invertase in the refrigerator therefore kept very well and although there appears to be a gradual loss of activity of the invertase exposed to room temperature, the loss is small.

### MOLASSES ANALYSES.

The molasses collected for sucrose determinations by the Jackson & Gillis No. 4 method and the invertase method (55—60°C.) were also analysed for brix, pH, reducing sugars, certain inorganic constituents, etc. The results were as follows:—

### FURTHER REVISION.

While this report deals mainly with the sampling of factory products and certain tests carried out during the year, the revision of our existing methods of chemical control has proceeded further. The Chapter on Reagents has been revised and changes in the general methods of analyses considered. It is felt that a wider field should be covered in the recommendations for the analysis of products.

The committee will endeavour to draw up the necessary specifications but the assistance and co-operation of members not serving on the committee are most essential if a really comprehensive set of methods, covering all the essential control work in the factory, is to be evolved.

### ACKNOWLEDGEMENTS.

The committee acknowledges with thanks the assistance of Mr. F. W. Hayes who drew up the recommendations for the sampling of sugars. Thanks are also due to the laboratory staffs of Darnall, Esperanza, Empangeni, and staff on the Central Board for carrying out many of the analyses here given.

### SUMMARY.

Three meetings of the committee were held during the year. The proposed revised methods of sampling are given in detail and tests were carried out to find out to what extent layers of juice differing in brix and sucrose existed in mixed juice scale-tanks. Differences in composition between last expressed

### MOLASSES ANALYSES.

Factory	UF.	ZM.	FX.	AK.	DL.	NG.	GL.	CK.	NE.	TS.	IL.	RN.	EZ.	SZ.	UK.	MV.	Average of 16 factories. Molasses.
Brix	91.30	92.00	85.00	88.50	89.50	90.80	87.50	86.40	85.50	92.50	89.00	89.30	87.00	90.00	82.00	90.00	88.52
Reducing sugars per cent.	9.90	10.12	11.00	12.36	11.64	12.17	15.06	19.43	8.12	11.83	11.50	14.20	10.88	14.71	8.31	11.02	12.02
pH	5.8	5.9	5.8	6.0	5.9	5.6	6.2	5.7	5.2	6.1	6.1	5.6	5.9	5.7	6.2	5.9	5.85
Volume of precipitate 20 per cent. solution after 8 hours settling, expressed as a percentage	4.7	2.3	4.7	4.6	4.6	2.0	8.1	2.3	Nil	9.3	7.0	5.5	2.3	7.0	7.0	6.0	4.84
Ash per cent.	13.00	12.70	11.30	11.20	11.90	13.65	12.00	11.05	11.10	11.90	11.55	11.10	11.00	11.20	11.25	11.30	11.70
Silica (SiO <sub>2</sub> ) per cent. ash	1.16	1.10	1.60	2.20	2.80	1.80	3.75	3.65	1.25	3.30	3.85	3.40	3.50	4.20	2.40	3.70	2.73
Lime (CaO) per cent. ash	12.20	11.70	11.80	13.80	14.00	11.50	12.80	14.70	17.00	14.50	13.00	11.70	12.20	12.80	12.60	13.10	13.09
Magnesia (MgO) per cent. ash	7.97	9.78	11.95	9.96	11.41	10.50	11.04	9.41	1.81	9.96	10.32	11.22	10.64	10.64	9.27	9.78	9.73
Phosphate (P <sub>2</sub> O <sub>5</sub> ) per cent. ash	0.84	0.60	0.32	0.32	0.40	0.64	0.48	0.72	0.36	0.40	0.64	0.60	0.72	0.72	0.52	0.64	0.56
Potash (K <sub>2</sub> O) per cent. ash	35.01	33.35	31.52	32.77	31.69	31.36	28.21	28.00	36.30	31.11	31.32	30.28	28.46	27.81	28.00	28.00	30.82
Nitrogen (N) per cent. ash	0.10	0.15	0.11	0.08	0.13	0.14	0.18	0.15	0.20	0.17	0.14	0.14	0.11	0.08	0.07	0.07	0.13
Sulphates (SO <sub>3</sub> ) per cent. ash	16.46	16.81	18.52	22.29	21.33	20.92	20.58	22.16	3.43	22.64	20.10	21.13	21.51	23.67	19.21	21.61	19.52

and last mill juice are shown. There can be big differences in the sucrose of filter cake in different parts on a rotary filter. Samples of Oliver Campbell filter cake kept for a surprisingly long time, but once deterioration became noticeable the fall in pol was rapid.

From preliminary experiments the sampling of trash and tops from consignments of cane seems reasonably accurate, but rather time and labour consuming.

There were no real differences under routine test conditions at the time between the Jackson and Gillis No. 4 method of sucrose analysis and the rapid inversion method, using invertase, when applied to mixed juice and molasses.

The invertase solutions kept well. Further analytical data on the molasses collected for the sucrose tests are also given.

Committee for Standardization of Chemical Control:—

J. L. du TOIT (*Convener*).

W. BUCHANAN. J. D. MILLAR.

G. C. DYMOND. G. S. MOBERLY.

W. G. GALBRAITH. J. RAULT.

The PRESIDENT said the Report represented a considerable amount of discussion by the Chemical Control Committee and he invited questions on anything connected with the revision of present methods.

Mr. DUCHENNE asked if the committee would consider the testing of sucrose in molasses by estimation of invert sugar before and after inversion, as an alternative method to the Jackson and Gillis method at present used. He had employed such a method for two years, and had found it safe and accurate, provided no lead acetate was used in clarifying the 2 per cent. solution.

The PRESIDENT replied that the Chemical Control Committee would go into the matter.

Dr. DODDS read a letter from the S.A. Bureau of

Standards suggesting standards to which various grades of sugar should conform. He would send, with the permission of the Association, a copy of the latest methods of sampling and analysis.

Mr. VAN WYK remarked on the statement in the report that 100 per cent. of the bags in a lot of sugar should be sampled. He wondered if it were really the intention to thus sample a shed full of sugar, and if it were really necessary to sample 100 per cent. of the bags. In sampling fertilisers a different method was used and it was recommended that ten samples should be taken at as uniform spaces as possible in a heap.

Mr. MOBERLY said that, as every bag is sampled as it came from factory or refinery, this sampling method applied only to certain small lots.

Mr. VAN WYK thought that as the Bureau of Standards now have a sub-committee working on the sampling of commercial products, the Association should be represented by one of its members on that sub-committee.

Dr. DODDS agreed that the Association should be so represented. He also pointed out that there was an important difference between the manufacture of sugar and of fertilizers. Superphosphate for instance was made in very large batches, whereas sugar coming through the factory never accumulated in large quantities and was never thoroughly mixed.

Mr. DU TOIT pointed out that with a high-grade product such as sugar, extreme precautions were necessary to get a reliable sample. One could not allow any part, which might differ radically from the rest, to be missed in sampling.

The PRESIDENT said that for white sugar, which is very pure, such sampling would not be necessary, and this reference was to sampling at a refinery where it was found necessary to sample each bag.

The methods reported on were recommended methods applicable to South African factories and the refinery, and they were continually being revised as information was received and analyses made.