

THE COMPOSITION OF SOUTH AFRICAN FINAL MOLASSES

By K. DOUWES DEKKER

In 1955-56 an investigation was carried out by the S.M.R.I. with a twofold purpose, *t.w.*

- (a) to collect more information on the composition of South African final molasses and the factors affecting this composition, and
- (b) to test certain methods of analysis and compare their results.

Each factory was requested to send in two samples, one sample produced in the week of 4th-9th July, the other sample representative of the final molasses produced in the week of 21st-26th November. All factories kindly co-operated by providing the requested samples, except that Chaka's Kraal did not send in a July sample, Umzimkulu did not send in a November sample, and Umfolozi's second sample was produced in January, 1956.

Sucrose and reducing sugars were determined fairly soon after the samples had arrived in order to avoid the danger of deterioration; some other constituents were estimated in 1956. In Appendix A a survey is given of the analytical methods used in this investigation. The analytical results are shown in Tables 1A and 1B.

DISCUSSION

The object of testing two samples from each factory was not only to investigate whether the composition of the cane had changed so much in the July to November period that the change was reflected in the composition of the non-sucrose of the molasses, but also to find out if the changing over of four factories from the sulphitation to the defecation method of juice clarification had affected the composition of the molasses. To get a better insight into the non-sucrose composition, Table 2 was drawn up, in which the various non-sucrose components are expressed as percentages of the total amount of non-sucrose present, *i.e.* the difference between Solids (by drying) and Sucrose (by the Jackson and Gillis method).

The graphs 1 to 7 illustrate the changes for the various factories. In these graphs the factories are arranged geographically, the most northern factory, Pongola, at the left-hand side of the graphs, the

southernmost factory, Umzimkulu, at the right-hand side.

Reducing Sugars (Fig. 1)—The reducing sugar content of the non-sucrose of the July samples is lowest for Pongola, Umfolozi and Z.S.M. and appears to increase distinctly, although somewhat irregularly, from north to south. The abnormally low figure for Natal Estates is associated with the destruction of reducing sugars in the carbonatation process. The geographical effect on the reducing sugar percentages is much less clear in the November data and the gap between the two curves widens perceptibly from north to south. The November data are without exception lower than the July data, the difference between the averages being 16 per cent of the July average.

Our agricultural friends will probably be able to tell us if it is justified to draw the conclusion from these figures that cane cut (in 1955) in the first half of July was not yet quite ripe, particularly so in respect of the factories south of Felixton and increasingly pronounced in a southerly direction, whilst the November cane had properly matured over the whole area.

Crude Protein (Fig. 2)—Here we seem to notice a geographical effect similar to that observed in respect of reducing sugars, but, with the exception of the four most northern mills, the protein content of the November samples is now markedly higher. Proteins are important constituents of the non-sucrose not removed in the clarification process. The data do not reveal any significant difference between the three methods of clarification, *t.w.* defecation, sulphitation and carbonatation, which, in respect of the carbonatation process, is somewhat surprising.

Carbonated Ash (Fig. 3)—On the average the non-sucrose of the November samples contains more ash than that of the July samples. The four most northern mills are again an exception, in respect too of their tendency to contain more ash. The high ash content of Natal Estates non-sucrose attracts attention and is probably due to the low reducing sugar content. The high reducing sugar and low ash content of Sezela non-sucrose favours excellent exhaustion as will be discussed later on.

TABLE 1A

ANALYTICAL DATA OF THE FINAL MOLASSES SAMPLES SENT IN BY PG, UF, ZM, FX, EN, AK, DK, DL AND GL

	PG		UF		ZM		FX		EN		AK		DK		DL		GL	
	July	Nov.	July	Nov.	July	Nov.	July	Nov.	July	Nov.	July	Nov.	July	Nov.	July	Nov.	July	Nov.
Refract. Brix	79.5	81.3	81.8	83.2	84.5	83.4	83.9	83.5	81.2	81.2	82.0	80.2	77.5	77.0	83.0	82.0	81.3	78.4
Brix, dil. 1+1	89.7	88.6	91.5	88.9	94.1	91.6	93.2	90.4	87.0	89.2	89.2	85.4	84.8	83.5	91.3	88.0	90.1	86.4
„ dil. 1+4	92.0	90.8	93.4	91.2	96.3	93.9	95.0	92.4	88.8	91.0	90.5	87.3	96.3	84.8	93.1	90.4	91.3	88.5
„ dil. 1+9	92.8	91.0	95.3	91.8	97.0	94.4	95.7	93.0	89.6	91.3	92.0	87.5	87.1	85.8	94.0	90.8	92.1	89.0
Solids, drying	76.3	77.0	78.8	80.2	82.0	79.5	82.4	81.6	78.1	79.5	79.2	77.1	76.8	76.0	81.2	78.9	79.2	75.5
„ K.F.	76.2	76.6	78.4	80.1	81.2	79.6	81.6	80.5	76.8	78.5	77.9	76.3	76.0	75.8	80.5	77.9	78.0	75.4
Polarisation	35.0	38.6	31.8	35.0	33.6	35.8	34.4	37.2	36.0	35.2	32.0	36.9	30.4	43.4	33.8	36.4	32.2	34.5
Sucrose (J. & G.)... ..	37.9	40.6	34.0	38.1	35.8	36.7	37.0	38.8	36.7	36.2	34.9	37.7	32.4	43.3	35.2	35.8	34.0	35.5
„ (L. & E.)... ..	38.3	40.6	34.0	37.9	36.2	36.7	37.1	39.0	36.9	36.4	35.2	37.6	32.8	43.2	35.1	36.8	34.2	35.2
Red. Sugars	9.6	8.5	13.1	11.5	13.6	11.5	14.6	12.2	12.2	10.5	16.4	12.5	14.7	8.6	14.1	11.4	15.3	12.3
Glucose	2.6	2.4	3.3	2.4	3.5	3.0	3.2	2.0	2.3	2.5	2.5	1.8	2.7	1.8	3.4	2.2	2.7	2.4
Sulph. Ash	17.4	17.0	18.6	16.9	17.8	17.3	16.4	15.8	13.6	16.0	13.7	13.4	13.4	12.0	15.5	14.6	14.9	14.9
Carbon. Ash	14.0	13.2	14.0	13.1	13.6	13.1	12.8	12.0	9.98	12.0	10.4	10.1	9.95	8.95	11.6	11.3	11.2	11.2
SiO ₂ +Insol.+Carbon. Ash...	3.12	2.89	4.54	7.69	4.05	4.73	4.08	6.34	4.61	3.98	4.08	4.93	3.53	3.52	3.57	4.56	4.18	3.87
R ₂ O ₃ % Carbon. Ash	0.55	0.53	1.27	2.71	0.47	0.55	0.82	1.56	1.17	1.11	0.55	0.78	0.48	0.98	0.64	0.77	0.65	0.59
CaO „ „ „	6.61	7.12	11.3	9.66	10.0	12.3	14.9	11.0	13.7	15.0	13.0	12.1	14.2	12.6	14.6	12.0	11.9	9.40
MgO „ „ „	5.03	5.15	7.05	6.43	9.43	9.17	6.44	7.99	11.3	10.2	12.6	10.9	12.5	11.2	10.8	10.9	12.5	13.1
SO ₃ „ „ „	17.6	17.8	17.1	17.4	18.4	18.7	18.1	17.5	22.6	22.3	24.9	20.9	22.4	23.6	22.7	21.7	24.4	26.7
P ₂ O ₅ „ „ „	0.49	0.52	0.75	2.14	0.54	0.76	0.70	1.52	1.34	1.25	0.70	2.14	0.76	0.92	0.87	1.87	0.69	0.83
Cl „ „ „	12.8	13.4	18.8	18.2	19.2	18.4	18.4	17.0	13.2	14.8	14.2	16.3	14.3	14.4	14.9	16.3	13.1	14.9
Na ₂ O „ „ „	0.36	0.45	1.71	1.54	2.43	1.53	1.57	1.59	0.50	0.33	2.22	1.38	0.70	0.67	2.32	2.12	2.05	2.59
K ₂ O „ „ „	49.3	50.6	42.0	40.7	37.6	37.7	35.4	38.7	34.8	34.2	30.4	35.7	32.5	36.2	31.4	32.5	30.8	33.4
Gums	1.64	2.00	2.08	3.94	2.83	3.73	2.52	3.59	3.43	4.39	3.26	3.81	2.11	2.83	2.83	4.21	2.20	2.86
Starch	0.24	0.35	0.28	0.37	0.39	0.54	0.21	0.25	0.51	0.56	0.37	0.54	0.31	0.47	0.42	0.73	0.35	0.53
Protein (N x 6.25)	3.62	4.25	4.06	3.50	3.13	3.25	3.31	3.44	2.69	3.50	2.44	3.25	2.75	2.94	2.44	2.63	2.25	2.88
Wax	0.16	0.17	0.25	1.04	0.45	0.30	0.14	0.59	0.55	0.66	0.16	0.74	0.17	0.19	0.24	0.76	0.14	0.14
True Purity	49.7	52.8	43.1	47.5	43.6	46.1	44.9	47.6	47.0	45.5	44.1	48.9	42.2	56.9	43.3	46.7	42.9	47.1
Refract. Purity	47.7	49.9	41.6	45.8	42.4	44.0	44.1	46.5	45.2	44.6	42.6	47.0	41.8	56.2	42.4	44.9	41.8	45.3
Expect Purity (D.D.)	45.7	45.1	44.4	43.6	43.6	43.5	42.7	42.9	42.1	43.3	40.9	41.7	41.0	43.1	42.2	42.3	41.7	42.6
„ „ (Haw.)... ..	35.30	35.51	34.04	33.83	33.72	33.83	33.03	33.63	32.63	34.35	30.93	32.55	31.41	33.92	32.66	33.68	31.94	33.81
True—Exp. Pur. (D.D.)	4.0	7.7	—1.3	3.9	0.0	2.6	2.2	4.7	4.9	2.2	3.2	7.2	1.2	13.8	1.1	4.4	1.2	4.5
Refr./Sucr. Pur.—Exp. Pur. (Haw.)	12.4	14.4	7.6	12.0	8.7	10.2	11.0	12.9	12.6	10.2	11.7	14.4	10.4	22.3	9.7	11.2	9.9	11.5

TABLE 1B

ANALYTICAL DATA OF THE FINAL MOLASSES SAMPLES SENT IN BY MV, CK, TS, NE, IL, RN, SZ AND UK, AND AVERAGES

	MV		CK		TS		NE		IL		RN		SZ		UK		Average (CK & UK excl.)	
	July	Nov.	July	Nov.	July	Nov.	July	Nov.	July	Nov.	July	Nov.	July	Nov.	July	Nov.	July	Nov.
Refract. Brix	80.4	80.9		81.9	82.3	81.3	83.1	82.2	83.9	83.4	83.2	84.3	80.7	80.9	81.9		81.9	81.5
Brix, dil. 1+1	88.7	88.0		89.7	88.1	87.4	91.0	89.3	90.0	89.9	92.3	92.5	87.5	90.0	89.7		89.9	88.6
„ dil. 1+4	90.9	90.1		91.2	90.3	88.9	92.3	90.7	92.0	92.0	94.2	94.5	89.2	92.2	91.7		91.7	90.6
„ dil. 1+9	91.2	90.6		91.8	90.5	90.1	93.1	91.2	93.0	92.2	94.8	94.9	89.5	92.9	92.0		92.5	91.1
Solids, drying	78.6	78.4		79.5	80.0	78.5	81.5	81.1	81.4	80.4	81.5	81.7	78.4	79.2	79.0		79.7	79.0
„ K.F.	77.5	78.0		78.8	79.1	78.3	81.3	80.9	81.3	79.8	81.0	80.5	77.7	79.1	78.5		79.0	78.5
Polarisation	31.4	35.0		39.2	31.2	36.6	38.2	41.8	32.6	40.0	35.4	40.0	27.4	32.8	29.6		33.0	37.3
Sucrose (J. & G.)	33.1	34.6		39.4	33.4	37.4	39.6	43.1	34.9	41.1	36.5	39.0	30.0	33.5	31.8		35.0	38.2
„ (L. & E.)	33.5	34.6		39.4	34.1	37.3	40.0	43.2	35.2	40.9	37.0	39.0	30.0	33.5	32.3		35.3	38.1
Red. Sugars	15.9	13.5		11.2	16.2	11.3	11.7	6.8	17.0	10.7	15.0	10.2	22.0	15.1	15.4		14.8	11.1
Glucose	2.7	2.6		2.2	2.4	1.7	3.3	2.0	3.6	2.7	3.0	2.4	3.3	2.6	3.3		3.0	2.3
Sulph. Ash	14.8	14.8		13.8	13.9	13.6	15.2	14.9	14.5	14.2	14.8	16.0	12.0	14.6	15.8		15.1	15.1
Carbon. Ash	11.1	11.1		10.2	10.6	10.4	11.8	12.1	11.2	10.6	11.2	11.8	8.93	11.0	11.7		11.5	11.5
SiO ₂ +Insol.+Carbon. Ash	3.62	3.50		4.54	4.95	4.76	0.80	1.09	3.00	4.60	3.81	3.44	5.00	4.16	4.17		3.80	4.27
R ₂ O ₃ % Carbon. Ash	0.61	0.81		0.74	0.60	0.86	0.36	0.40	0.51	1.16	0.57	0.67	0.48	0.40	0.82		0.65	0.93
CaO	10.4	13.4		13.2	9.25	13.1	13.5	10.1	15.0	13.8	10.6	9.74	11.4	11.1	12.6		12.0	11.5
MgO	12.2	11.8		13.6	12.1	10.8	0.64	1.29	7.99	9.67	12.7	13.7	13.7	13.3	11.5		9.80	9.71
SO ₃	23.2	23.4		24.0	23.7	21.6	12.8	7.12	19.3	21.7	23.1	20.1	25.1	24.2	20.8		21.0	20.3
P ₂ O ₅	0.75	0.79		0.87	2.18	2.37	0.29	0.30	1.72	1.92	0.69	0.79	0.62	0.76	0.98		0.87	1.26
Cl	13.9	15.8		13.9	14.3	14.8	19.0	20.0	15.0	16.0	13.8	17.4	12.0	15.0	14.7		15.1	16.2
Na ₂ O	2.08	1.44		1.27	1.32	1.06	8.46	9.65	1.43	1.32	1.69	1.62	1.57	1.28	1.36		2.03	1.90
K ₂ O	34.5	33.1		31.0	35.8	34.0	42.3	40.8	35.0	35.6	34.1	36.2	32.7	33.9	34.8		35.9	36.9
Gums	2.41	4.11		3.83	3.58	4.79	0.63	1.24	3.34	3.90	2.12	3.61	2.16	4.20	3.01		2.48	3.55
Starch	0.38	0.52		0.60	0.56	0.70	0.15	0.20	0.45	0.67	0.33	0.58	0.29	0.47	0.43		0.35	0.50
Protein (N x 6.25)	2.38	2.56		3.13	2.88	3.56	2.44	2.69	3.00	4.00	2.19	2.81	1.81	2.88	3.19		2.76	3.21
Wax	0.19	0.28		0.10	0.94	0.85	0.12	0.08	0.91	0.60	0.15	0.20	0.13	0.14	0.24		0.31	0.45
True Purity	42.1	44.1		49.6	41.8	47.6	48.6	53.1	42.9	51.1	44.8	47.8	38.3	42.3	40.3		44.0	48.3
Refract. Purity	41.2	42.8		48.1	40.6	46.0	47.7	52.4	41.6	49.3	43.9	46.3	37.2	41.4	38.8		42.8	46.8
Expect Purity (D.D.)	41.5	41.9		42.2	40.8	41.9	43.1	44.4	41.1	42.6	41.8	43.3	38.6	41.3	41.9		42.1	42.9
„ „ (Haw.)	31.63	32.66		33.23	31.17	33.29	33.76	35.93	31.18	33.63	32.07	34.40	26.94	31.90	32.90		32.2	33.8
True—Exp. Pur. (D.D.)	0.6	2.2		7.4	1.0	5.7	5.5	8.7	1.8	8.5	3.0	4.5	-0.3	1.0	-1.6		1.9	5.4
Refr./Sucr. Pur.—Exp. Pur. (Haw.)	9.6	10.1		14.9	9.4	12.7	14.9	16.5	10.4	15.7	11.8	11.9	10.3	9.5	5.9		10.7	13.0

TABLE 2

NON-SUCROSE CONSTITUENTS SHOWN AS PERCENTAGES OF NON-SUCROSE PRESENT

	PG		UF		ZM		FX		EN		AK	
	July	Nov.	July	Nov.	July	Nov.	July	Nov.	July	Nov.	July	Nov.
Reducing Sugars ...	25.0	23.3	29.2	27.3	29.4	26.9	32.2	28.5	29.5	24.2	37.0	31.7
Carbonated Ash ...	36.5	36.2	31.2	31.1	29.4	30.6	28.2	28.0	24.1	27.7	23.5	25.6
Gums ...	4.3	5.5	4.6	9.4	6.1	8.7	5.6	8.4	8.2	10.1	7.4	9.7
Starch... ..	0.6	1.0	0.6	0.9	0.8	1.3	0.5	0.6	1.2	1.3	0.8	1.4
Crude Protein ...	9.5	11.7	9.1	8.3	6.8	7.8	7.3	8.0	6.5	8.1	5.5	8.2
Wax	0.4	0.5	0.6	2.5	1.0	0.7	0.3	1.4	1.3	1.5	0.4	1.9
U.O.M.	24.3	22.8	25.3	21.4	27.3	25.5	26.4	25.7	30.4	28.4	26.2	22.9
	DK		DL		GL		MV		CK		TS	
	July	Nov.	July	Nov.	July	Nov.	July	Nov.	July	Nov.	July	Nov.
Reducing Sugars ...	33.1	26.3	30.6	27.1	33.8	30.8	34.9	30.8	—	27.9	34.8	27.5
Carbonated Ash ...	22.4	27.4	24.1	26.8	24.8	28.0	24.4	25.3	—	25.4	22.7	25.3
Gums	4.8	8.7	6.2	10.0	4.9	7.2	5.3	9.4	—	9.6	7.7	11.7
Starch... ..	0.7	1.4	0.9	1.7	0.8	1.3	0.8	1.2	—	1.5	1.2	1.7
Crude Protein ...	6.2	9.0	5.3	6.2	5.0	7.2	5.2	5.8	—	7.8	6.2	8.7
Wax	0.4	0.6	0.5	1.8	0.3	0.4	0.4	0.6	—	0.2	2.0	2.1
U.O.M.	33.1	28.0	33.3	28.1	31.2	26.4	29.8	28.1	—	29.1	26.6	24.7
	NE		IL		RN		SZ		UK		Averages*	
	July	Nov.	July	Nov.	July	Nov.	July	Nov.	July	Nov.	July	Nov.
Reducing Sugars ...	27.9	17.9	36.6	27.2	33.3	23.9	45.5	33.0	32.6	—	33.2	27.8
Carbonated Ash ...	28.2	31.8	24.1	27.0	24.9	27.6	18.4	24.1	28.0	—	25.6	27.9
Gums	1.5	3.3	7.2	9.9	4.7	8.5	4.5	9.2	6.4	—	5.82	9.03
Starch... ..	0.4	0.5	1.0	1.7	0.7	1.4	0.6	1.0	0.9	—	0.80	1.28
Crude Protein ...	5.8	7.1	6.5	10.2	4.9	6.6	3.7	6.3	6.8	—	6.26	7.99
Wax	0.3	0.2	2.0	1.5	0.3	0.5	0.3	0.3	0.5	—	0.72	1.16
U.O.M.	36.3	39.7	23.6	24.2	31.9	32.9	27.6	27.1	35.7	—	28.4	26.2

* Excluding Chaka's Kraal, Natal Estates and Umzimkulu

Gums (Fig. 4)—The non-sucrose of the November samples contains considerably more gums than that of the July samples. Although we are not well informed on the effect of gums on the viscosity of molasses, a high percentage of gums is perhaps not conducive to good exhaustion.

As in respect of crude protein the data do not show a clear difference between the gums content of the non-sucrose of defecation and sulphitation factories respectively. Carbonatation appears to remove gums much more effectively than sulphitation or defecation.

There is no geographical effect.

Starch (Fig. 2)—The term "Gums" is usually applied to a group of polysaccharides which are precipitated by acidified alcohol. Starch is one of them. Although no molasses contains more than two per cent starch on non-sucrose, it is an important impurity due to its effect on the filtrability of raw sugar melts. Starch cannot completely be removed from raw sugar crystals by washing, it is occluded in the crystals in addition to being present in the molasses film coating the crystals. There is usually more starch inside than outside the crystals.

Without exception the November data of the molasses tested are higher than the July data. No difference is perceptible between sulphitation and

defecation mills, but carbonatation appears to remove starch more efficiently.

Wax (Fig. 4)—Wax is also an undesirable impurity in raw sugar due to its effect on filtrability, and is also present in final molasses to a minor extent. A number of factories do not show much difference between the July and November samples, t.w. Pongola, Z.S.M., Entumeni, Doornkop, Gledhow, Melville, Tongaat, Natal Estates, Illovo, Renishaw and Sezela. Other factories show considerably higher data for November, t.w. Umfolozi, Felixton, Amatikulu and Darnall. The latter factories changed over from sulphitation to defecation at some time between July and November, t.w. Umfolozi 1/10/55; Felixton 29/10/55; Amatikulu 24/9/55 and Darnall 10/9/55. The factories which do not show an increase between July and November applied sulphitation during the whole year, with the exception of Illovo and Tongaat which applied defecation in both months and show high wax data for both samples, and Natal Estates which applied carbonatation.

Hence there is little doubt that in respect of the removal of wax there is a fundamental difference between sulphitation and defecation, a difference which so far we have not found in respect of the removal of other non-sucrose constituents (see, however, P₂O₅). It is of course well known that sulphitation syrup is lighter in colour than defecation

syrup, but it has not yet been established that this difference is due to a superior removal of coloured material by the sulphitation process. It might also be that the presence of SO_2 prevents the formation of colour during processing.

Undetermined Organic Matter (Fig. 5)—The data shown are found by deducting from 100 the sum of the percentages of reducing sugars, carbonated ash, gums, crude protein and wax. Starch was not deducted as it is part of "Gums."

The differences from 100 are thus affected by the analytical errors of the other percentages.

U.O.M. contains the organic acids present in final molasses, either originating from mixed juice, or formed during processing. The high data for the Natal Estates samples is perhaps due to the acids formed when reducing sugars were destroyed. Since organic acids bind lime their presence is not conducive to good exhaustion of final molasses.

The data shown in Fig. 5 fluctuate considerably. The July data of the factories north of Chaka's Kraal are higher than the November data; south of Chaka's Kraal there is not much difference.

ASH AND ASH CONSTITUENTS

Sulphated Ash (Fig. 6)—The sulphated ash percentage of final molasses is higher than the carbonated ash figure due to carbonates and salts of organic acids being converted into sulphates during the ashing procedure. Table 1B shows that the average sulphated ash percentage for July (15.1 per cent) is equal to the average percentage for November, and that both averages are considerably higher (31 per cent) than the carbonated ash percentages (11.5 per cent).^{*} Examining the individual factory data it appears that there is also little difference between the July and November data, with the exception of Entumeni and Sezela where the difference is more than 10 per cent.

We noticed that for some ashes the sum of the constituents as given was considerably lower than the carbonated ash figure, for example Pongola (July) where the sum of the constituents amounted to 95.9 per cent, and Natal Estates (November) where the sum was 90.75 per cent. The discrepancy appeared to be due to the presence of carbonates, of which the straight ash of the Natal Estates sample contained 12.5 per cent, and of the Pongola sample 5.1 per cent. Other molasses contained smaller amounts, e.g. Umfolozi (November) 0.79 per cent and Darnall (November) 0.89 per cent on straight ash respectively.

^{*} In averaging, Chaka's Kraal and Umzimkulu were not taken into account, since both factories had provided only one sample.

[†] Unfortunately the July and November curves for SiO_2 in Fig. 6 have been reversed. The dotted curve refers to the July samples, the drawn curve to the November samples.

K_2O — K_2O constitutes 50 per cent of the ash of Pongola final molasses, just over 40 per cent of the ash of Umfolozi final molasses and 30–40 per cent of all other molasses. The difference between July and November samples is not significant.

Na_2O —The ash of Pongola, Entumeni and Doornkop molasses contains considerably less than 1 per cent Na_2O , probably due to the canefields of these mills being situated further inland than those of the other factories, where the Na_2O percentages are 1 to less than 2.5 per cent with the exception of Natal Estates, whose final molasses shows exceptionally high figures, t.w. approximately 9 per cent.

Cl—The percentages vary between 12 per cent (Sezela, July) and 20 per cent (Natal Estates, November). Neither K_2O , Na_2O nor Cl are supposed to be affected by chemical reactions during the clarification process nor to influence the effect of the clarification.

The percentages of the following ash constituents do not reflect directly the corresponding percentages which were present in the juice from which the molasses was produced, since their amounts may have been altered during processing, particularly during clarification operations. These constituents may partly be present in final molasses as finely divided suspended matter.

SiO_2 + Insolubles (Fig. 6)—The July samples do not show any geographical effect.[†] The November samples of Umfolozi, and to a lesser extent of Felixton, Amatikulu and Darnall, show higher data than the corresponding July samples, which we are tempted to associate with the changing-over from sulphitation to defecation. Both Tongaat data are on the high side, the November data for Illovo is higher than the July figure. On the whole it seems safe to conclude that the sulphitation process is slightly more effective in removing SiO_2 + Insolubles than the defecation process. The superiority of the carbonation process in removing silica is quite marked.

P_2O_5 (Fig. 6)—In respect of the removal of P_2O_5 there is no doubt that the sulphitation process is more effective than the defecation process, there being a difference between the November and July samples only for those factories which changed over from sulphitation to defecation between July and November, t.w. Umfolozi, Felixton, Amatikulu and Darnall. Tongaat and Illovo data are high for both months. The similarity between the behaviour of P_2O_5 and wax leaps to the eye and poses the question whether we are concerned here with inorganic phosphates, or with organic phosphate-containing compounds. Honig¹ has pointed out that what is usually

¹ P. Honig, Proc. I.S.S.C.T. 8 (1953) 710

designated as wax is really a mixture of various groups of organic compounds, which could better be classified as lipids. Amongst them we find compounds of fatty acids with phosphoric acid, called phosphatides or phospholipids. If the sulphitation is more effective than the defecation process in removing phosphatides, the effect will probably also be shown by the P_2O_5 figures. Since phosphatides have an undesirable effect on clarification and perhaps also on the quality of raw sugar their behaviour during clarification requires further investigation.

CaO (Fig. 6)—The CaO content of the carbonated ash of Pongola final molasses is low. Proceeding southwards the corresponding figure increases somewhat irregularly up till Felixton/Entumeni and then remains constant between 10 and 15 per cent. Somewhat surprisingly the data do not reveal any specific influence of the particular method of clarification.

MgO—MgO is a major constituent of the ash of Natal juice, and since neither defecation nor sulphitation affect the MgO ion to a large extent, also of the ash of most Natal final molasses. The low data for Natal Estates are due to much MgO being removed in the carbonation process. Differences between other factories are mainly due to the composition of the cane processed. In this respect the low MgO content of the Pongola molasses attracts attention.

SO₃ (Fig. 7)—Somewhat surprisingly the SO₃ curves indicate that there is no appreciable difference in the SO₃ per cent carbonated ash data between sulphitation and defecation factories. Carbonation is effective in removing a high percentage of sulphates.

R₂O₃ (Fig. 7)—The data for Umfolozi and Felixton only seem to indicate that sesquioxides are less

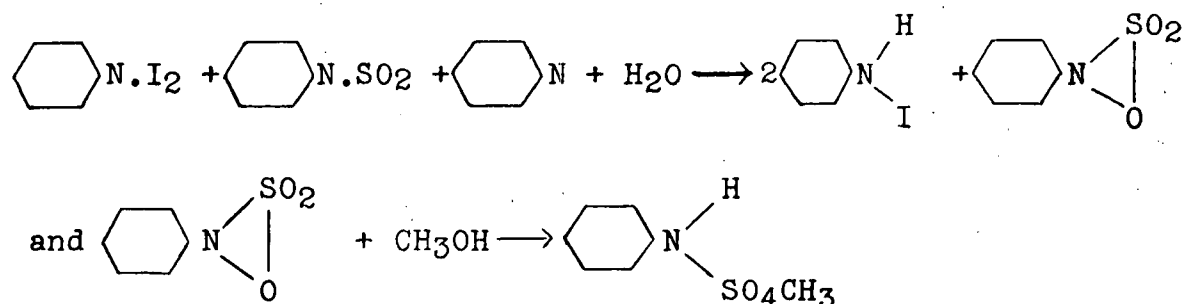
effectively removed by the defecation than by the sulphitation process; the differences for Amatikulu and Darnall are insignificant. Hence there is not sufficient evidence to justify claiming superiority of the sulphitation process in this respect. The carbonation process (Natal Estates) leaves an amount of sesquioxides in clarified juice which is comparable with that of Sezela and Gledhow. There is no geographical effect either.

OTHER DATA

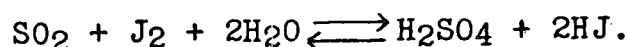
Brix and Solids—Degrees brix were determined by refractometer and gravimetrically at three dilutions, percentage solids was determined by drying and by the Karl Fischer method.

The refractometer brix was found to be much lower than the gravimetric brix, in accordance with many other investigations conducted all over the world. Our data also confirmed the long-known fact that the gravimetric brix depends on the degree of dilution of the sample, the greater the dilution the higher the brix found. The dependence of the gravimetric brix on the concentration of the solution which is tested, is an argument against Honig's suggestion² to substitute apparent target purities for true target purities for judging the exhaustion of final molasses. The use of the Karl Fischer method for the determination of water in final molasses has been discussed by Ghijsen and Suyckerbuyck³ and by Honig and Sattler.⁴

The Karl Fischer method is based on the titration of a small quantity of final molasses suspended in anhydric methanol with a solution of iodine, sulphur dioxide and pyridine in anhydric methanol. The main reaction in the methanol solution appears to take place in two distinct steps as follows:



or simplified



² P. Honig, Proc. I.S.S.C.T., New Delhi (1956).

³ J. J. Ghijsen and H. J. P. Suyckerbuyck, Chem. Weekbl.

46 (1950) 474.

⁴ P. Honig and L. Sattler, Intern. Sugar J. 58 (1956) 92.

As long as water is present in the titrated solution the characteristic iodine colour of the added solution is discharged. Hence the endpoint of the titration is readily noticed visually in a colourless solution, but somewhat more difficult in the dark coloured suspension of final molasses in methanol. In such cases the endpoint of the titration can better be determined electrometrically, as suggested by Almy, Griffin and Wilcox.⁵

The electrical system takes advantage of the fact that the Karl Fischer reagent acts as a depolariser towards a pair of bright platinum electrodes immersed in the reactants and automatically polarised by a small current. The cathode remains polarised so long as an excess of water is present and a potential difference is consequently registered on a potentiometer. The addition of a slight excess of the reagent depolarises the cathode and the recorded potential difference drops to zero because the two electrodes become electrically identical.

Since the Karl Fischer reagent is extremely sensitive to the smallest trace of atmospheric moisture, the whole system of the apparatus must be suitably protected by desiccants, etc. For convenience in actual practice, a methanol-water solution is used for sub-standardisation and back-titration. This is particularly useful because the Karl Fischer reagent, even when stored in cold and absolutely anhydrous conditions, suffers slow but continual auto-decomposition.

It must be realised that the Karl Fischer method in its simplest form can only be applied to liquids which comply with the following requirements:

- (a) they do not react with the reagent;
- (b) they are miscible with the reagent;
- (c) they will conduct an electric current (for the electrometric method).

There are limitations to the method and as a general rule the following groups of compounds give rise to difficulties:

1. Reactive aromatic-amino compounds.
2. Phenolic substances.
3. Highly unsaturated bodies.
4. Compounds containing a reactive carbonyl grouping.

Ghijssen and Suyckerbuyck compared various methods of determining water in final molasses with a "standard method" in which the sample was dried

in vacuo at low temperature (70°) till the weight was constant. For beet molasses they found quite satisfactory agreement between the results of the standard method and the Karl Fischer method, for cane molasses they found slightly lower water percentages by the Karl Fischer method. This is illustrated by the following data:

	Karl Fischer Method per cent	Standard Method per cent
Cuba final molasses	27.1	27.7
Puerto Rico final molasses	25.2	26.2
Mexico final molasses ...	24.1	24.8

They reasoned that the results of the Standard Method were probably somewhat high due to deterioration of non-sugars during the drying procedure and concluded that the Karl Fischer method is rapid, accurate and suitable for routine analysis.

The opinion of Honig and Sattler on the Karl Fischer method for determining water in final molasses is less favourable. They found the Karl Fischer method to give higher results than ascertained by vacuum drying, the difference between the two methods being up to 2.5 per cent. Hence the method could not be recommended.

The Karl Fischer method was explored by the S.M.R.I. in 1955. The apparatus was procured from Messrs. Baird & Tatlock, the end-point being ascertained electrically. Since the preliminary results were satisfactory, the water content of all samples of this investigation was determined both by vacuum drying and by the Karl Fischer method. The results are shown in Tables 1A and 1B, the differences between the water percentages by the two methods are shown in Table 3.

The Karl Fischer method gave slightly higher results, the differences between the results of the vacuum drying method were considerably smaller than as found by Honig and Sattler. Our conclusion is that the Karl Fischer method is quite suitable to assess the water content of final molasses rapidly and sufficiently accurately for many purposes. The costs of the analysis are, however, high.

Sucrose—The sucrose content of the samples was determined by direct polarisation, by Jackson and Gillis method IV, and chemically, using Lane and Eynon's method of determining reducing sugars

TABLE 3

WATER PERCENTAGE BY KARL FISCHER METHOD MINUS WATER PERCENTAGE BY DRYING

July Samples			November Samples		
PG — 0.1%	DK — 0.8%	NE — 0.2%	PG — 0.4%	DK — 0.2%	NE — 0.2%
UF — 0.4	DL — 0.7	IL — 0.1	UF — 0.1	DL ... 1.0	IL — 0.6
ZM — 0.8	GL — 1.2	RN — 0.5	ZM — (-0.1)	GL — 0.1	RN — 1.2
FX — 0.8	MV — 1.1	SZ — 0.7	FX — 1.1	MV — 0.4	SZ — 0.1
EN — 1.3	CK — —	UK — 0.5	EN — 1.0	CK — 0.7	UK — —
AK — 1.3	TS — 0.9	Av. — 0.7	AK — 0.8	TS — 0.2	Av. — 0.6

⁵ Almy, Griffin and Wilcox, Ind. Engin. Chem. 33 (1940) 392.

before and after inversion. Apparent sucrose (direct polarisation) was generally lower than sucrose as determined by the Jackson and Gillis method, but a higher figure was found on three November samples, t.w. Doornkop (+0.1 per cent); Melville (+0.4 per cent) and Renishaw (+1.0 per cent). On the whole the average difference between Jackson and Gillis-sucrose and apparent sucrose was smaller in the November samples (0.9 per cent) than in the July samples (2.0 per cent).

The agreement between the sucrose percentages as determined according to Jackson and Gillis and to Lane and Eynon was quite satisfactory, as can be seen from the average figures, the Jackson and Gillis data tending to be slightly lower. A difference of 0.5 per cent occurred twice, 0.4 per cent five times, 0.3 per cent four times, 0.2 per cent six times, 0.1 per cent six times, and equal results were obtained in nine cases.

Glucose—Not all reducing substances are fermentable. The non-fermentable portion was determined according to a method developed in Java and averaged 20.3 per cent of the total amount of reducing sugars in the July samples, and 20.7 per cent in the November samples respectively. High data for non-fermentable per cent total reducing substances were shown by Natal Estates (28.2–29.4), Pongola (27.0–28.2) and Z.S.M. (25.7–26.1), low data by Amatikulu (15.2–14.4), Tongaat (14.8–15.0) and Sezela (15.0–17.2). The percentage of unfermentable reducing substances is obviously of direct interest to the distiller.

Exhaustion—With one exception (Entumeni) the purities of the November samples are considerably higher than those of the July samples. This tendency is confirmed by the data of the Monthly Summaries. In Table 4 the gravity purities taken from Tables 1A and 1B and calculated with the brix as determined in the 1+1 dilution, are compared with those of the Monthly Summaries. In most cases the agreement is quite satisfactory, as is borne out by the average July data, which differ only 0.1 unit. But in respect of the November samples the S.M.R.I. data are generally higher, the S.M.R.I. average being 1.6 unit higher than the Monthly Summary averages.

The Monthly Summary November average is 3.2 units higher than the corresponding July figure, the difference being smaller however than the difference between the S.M.R.I. figures, i.e. 4.7.

The tendency of the November samples to have higher purities than the July samples can partly be explained by the higher water content, but the available data do not explain why the November samples have not been concentrated in the factory as far as the July samples. Neither do they explain the individual differences shown by Table 4.

To judge the degree of exhaustion of the samples, target purities were calculated both by Douwes Dekker's formula and from the table given by the Hawaiian Experiment Station.

The Douwes Dekker formula gives the true purity which the average Java factory of 1939 would have obtained if a material had been worked having the

TABLE 4
GRAVITY PURITIES S.M.R.I. AND MONTHLY SUMMARIES

		SMRI	Month Sum.		SMRI	Month. Sum.		SMRI	Month. Sum.
July Samples	PG ...	42.3	40.7	DK ...	35.8*	36.4*	NE ...	43.5	43.5
	UF ...	37.2	36.9	DL ...	38.6	37.4	IL ...	38.8	38.9
	ZM ...	38.0	37.5	GL ...	37.7	37.6	RN ...	38.4*	38.9*
	FX ...	39.7	38.5	MV ...	37.3	37.5	SZ ...	34.3	34.4
	EN ...	41.4*	42.8*	CK ...	—	39.5	UK ...	35.5	37.3
	AK ...	39.1	37.6	TS ...	37.9	38.0	Av. ...	38.5	38.4
November Samples	PG ...	45.8	41.8	DK ...	52.0*	43.8*	NE ...	48.3	46.7
	UF ...	42.9†	40.2†	DL ...	41.8	41.4	IL ...	45.7	41.2‡
	ZM ...	40.1	39.0	GL ...	41.1	39.3	RN ...	43.2*	41.6*
	FX ...	42.9	41.4	MV ...	39.3	41.2	SZ ...	37.2	37.0
	EN ...	39.5*	41.5*	CK ...	43.9	43.8	UK ...	—	41.2
	AK ...	44.1	42.6	TS ...	42.8	42.3	Av. ...	43.2	41.6

* Apparent purity

† January sample

‡ October data, analysis of November sample not available

same reducing sugar per cent non-sucrose and sulphated ash per cent non-sucrose ratio's.*

The Hawaiian table gives the expected refractometer/sucrose purities of final molasses of different reducing sugars/carbonated ash ratio's. They are supposed to be saturated at 50°C and to have been

* The Douwes Dekker formula reads:

$$Y_w = 35.89 - 0.0809 x_1 + 0.2605 x_2$$

where Y_w = expected sucrose/dry matter purity.

x_1 = reducing sugars per cent nonsucrose (= dry matter —sucrose)

x_2 = sulphated ash per cent nonsucrose.

concentrated until the viscosity at that temperature is 600 poises. Hence 600 poises is the target viscosity.

The purities of many July samples correspond very well with the Java target purity (Umfolozi, Z.S.M., Doornkop, Darnall, Gledhow, Melville, Tongaat, Sezela and Umzinkulu), three of them even being lower, but the November samples are generally considerably higher. The only exception is Sezela, of which the November purity is merely one unit higher than the target purity.

SUMMARY

Compared with the Hawaiian standards all Natal molasses are badly exhausted, the November samples being worse than the July samples.

The obvious question is, of course: Why is this so? Are our molasses specifically more viscous than Hawaiian molasses, or do Natal factories not concentrate their final massecuites to the required viscosity? An answer to this question cannot be given at this stage, but we hope to investigate the viscosities of Natal molasses in order to find out if they are significantly different from those of other countries.

Such an investigation might also explain why Natal final molasses are less well exhausted towards the end of the season than in July, both according to Java and Hawaiian standards.

Comparison with both Java and Hawaiian standards shows that the exhaustibilities of the final molasses of various factories differ considerably due to differences in non-sucrose composition.

Owing to its high reducing sugar and low ash content Sezela molasses is more readily exhaustible than any other molasses, whilst the purities of Pongola, Umfolozi and Natal Estates are expected to be high. This fact should, however, not detract from endeavouring to reduce the purities further than at present. The Java standards were obtained at a time when hardly any factory was equipped with high-speed centrifuges, and for this reason it should be possible with modern equipment to exhaust Natal final molasses better than required by the Java standard.

The analyses are given of two monthly samples of final molasses of each Natal factory, one having been produced in July, the other in November, 1955. The reducing sugar content of the non-sucrose portion of the November samples is typically lower than that of the July samples which showed a tendency towards higher percentages in the direction from north to south. The opposite effect was observed in respect of the (crude) protein data.

The defecating factories showed higher figures than the sulphiting factories for wax per cent non-sucrose and P_2O_5 per cent carbonated ash. Some constituents appeared to be better removed by the carbonatation than by the defecation and sulphitation processes.

The purities of the samples were judged by comparison with the target purities as calculated by the Douwes Dekker formula and as found from the Hawaiian tables. In respect of the latter criterion all Natal molasses are quite unsatisfactorily exhausted.

The purities of the July samples were on the average but slightly higher than the Java criterion. The purities of the November samples were considerable higher. No explanation could be given for the less satisfactory degree of exhaustion in November.

ACKNOWLEDGMENT

Our sincere thanks are due to the S.A.S.A. Experiment Station who kindly analysed our samples for K_2O , Na_2O (flame photometer) and N (Kjeldahl).

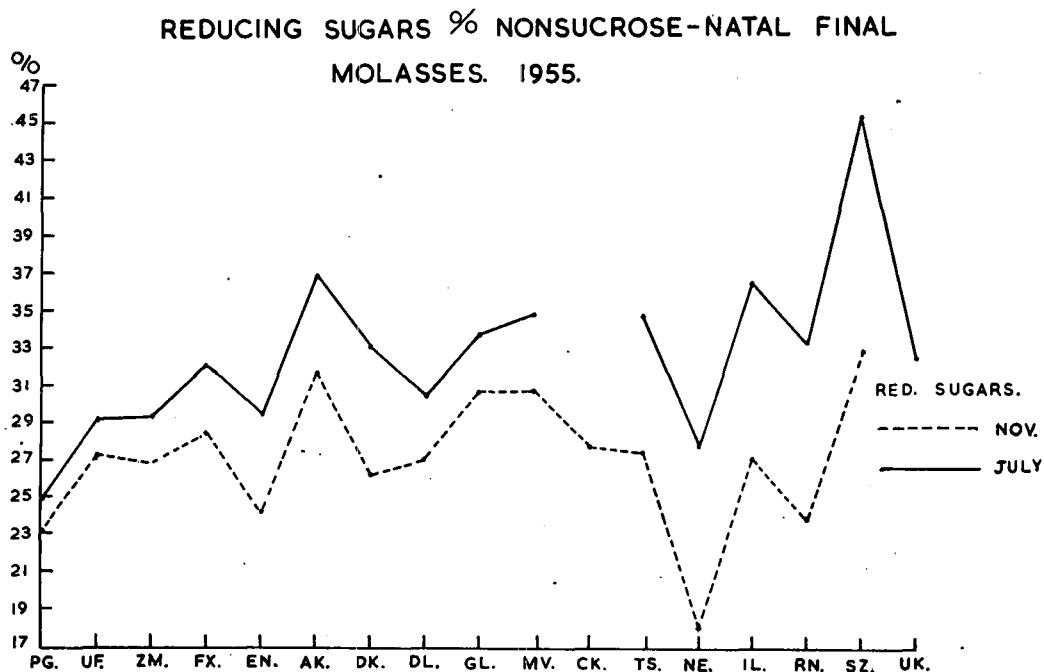


FIG. I.

CRUDE PROTEIN % NONSUCROSE—NATAL FINAL MOLASSES 1955.
 STARCH % NONSUCROSE—NATAL FINAL MOLASSES 1955

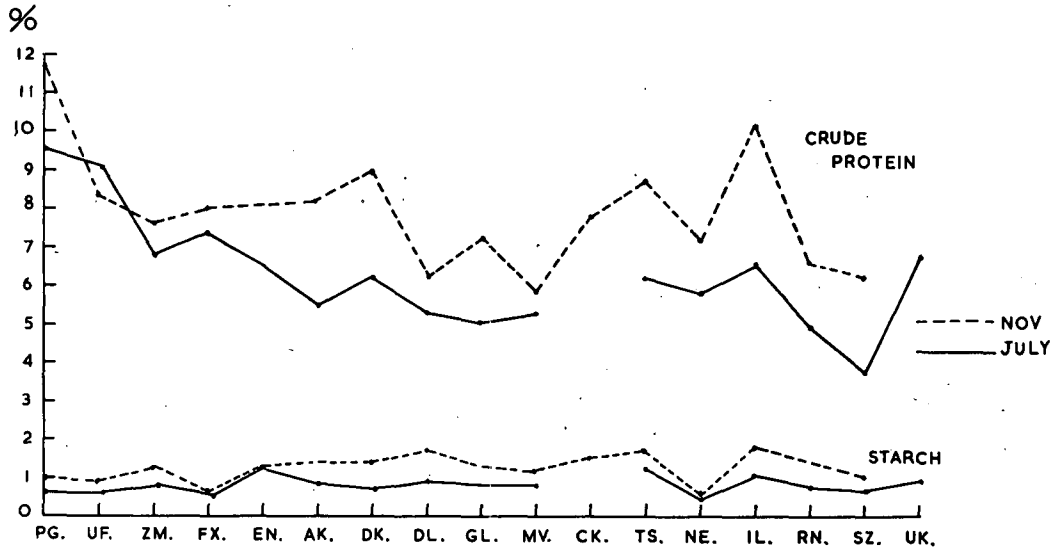


FIG. 2.

CARBONATED ASH % NONSUCROSE—NATAL FINAL MOLASSES
 1955

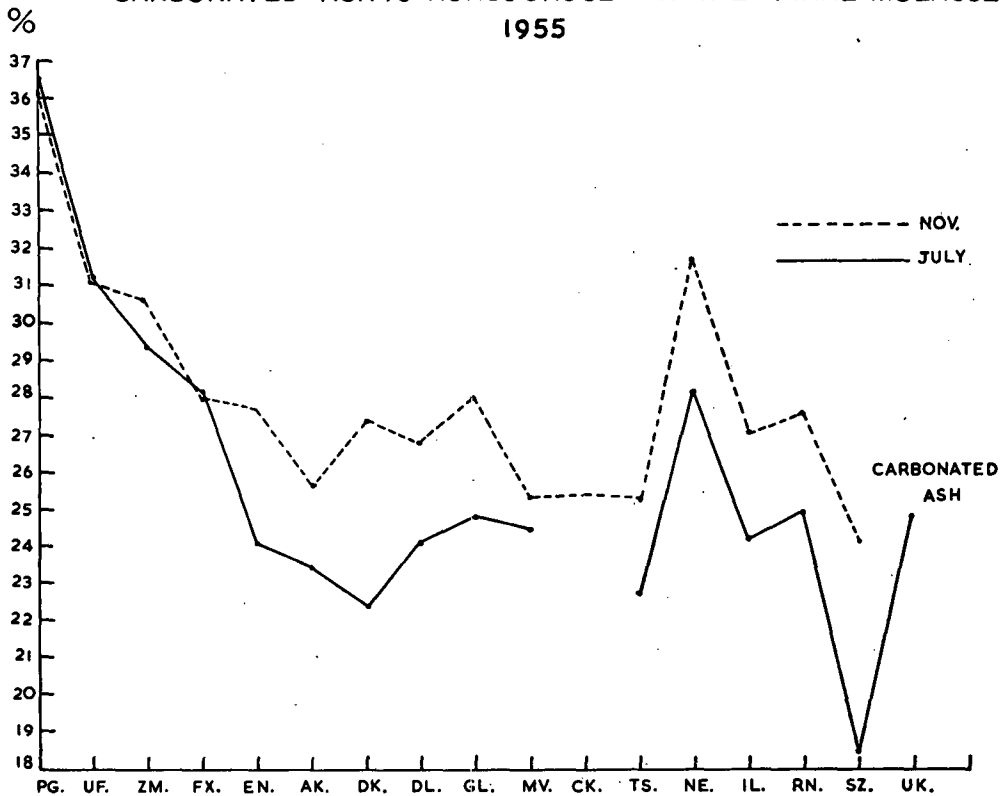


FIG. 3.

GUMS % NONSUCROSE— NATAL FINAL MOLASSES 1955
 WAX % NONSUCROSE— NATAL FINAL MOLASSES 1955

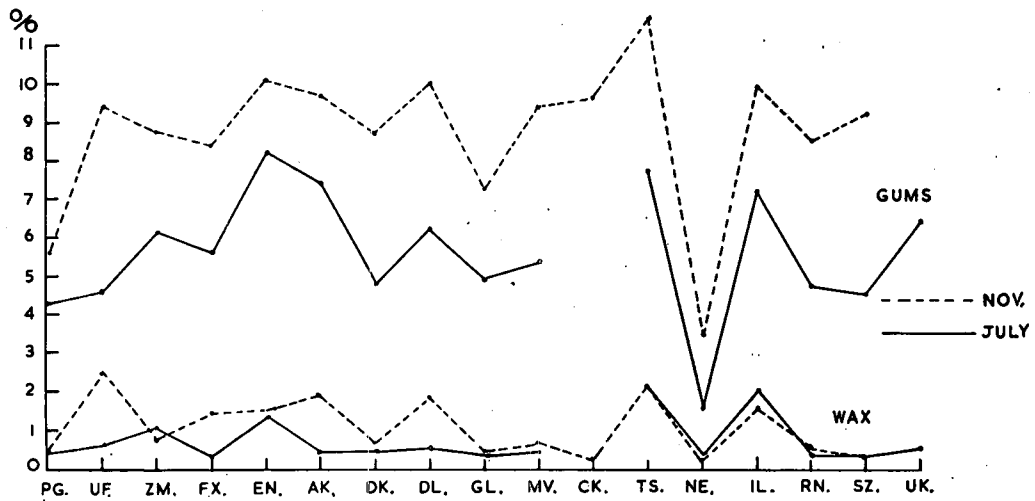


FIG. 4.

U.O.M. % NONSUCROSE — NATAL FINAL MOLASSES 1955.

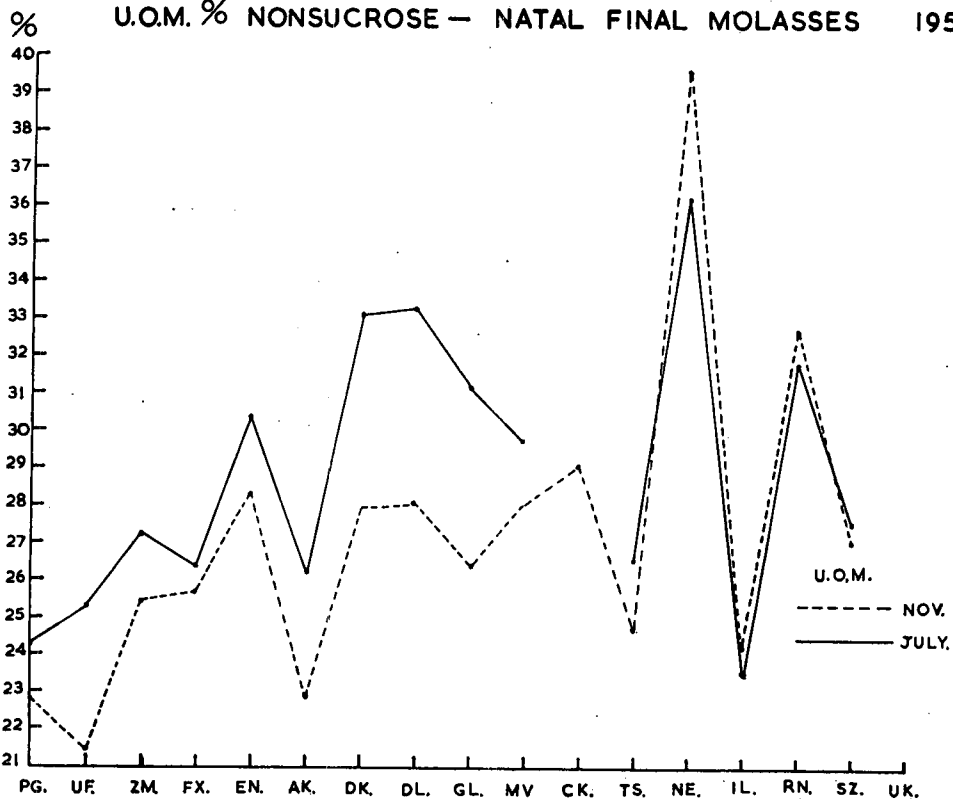


FIG. 5.

SULPHATED ASH % FINAL MOLASSES - NATAL FINAL MOLASSES 1955.
 $\% \text{CaO, SiO}_2 + \text{INSOL AND P}_2\text{O}_5$ % CARBONATED ASH - NATAL FINAL MOLASSES 1955.

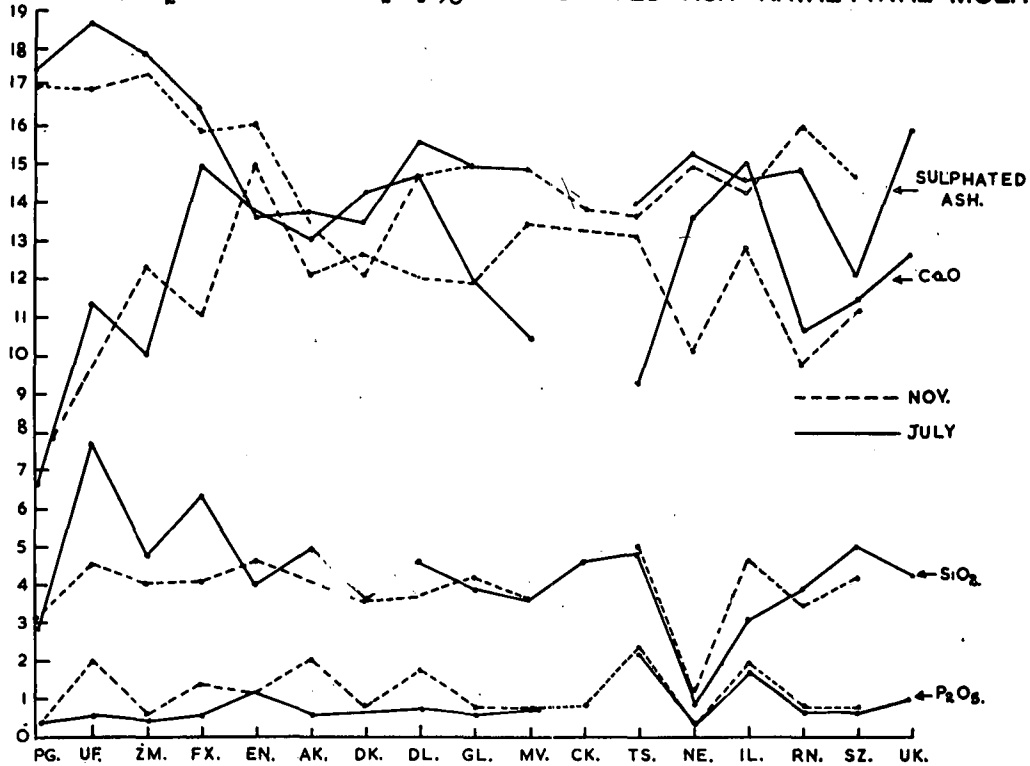


FIG. 6.

SO₃ % CARBONATED ASH NATAL FINAL MOLASSES 1955.
 R₂O₃ % CARBONATED ASH NATAL FINAL MOLASSES 1955.

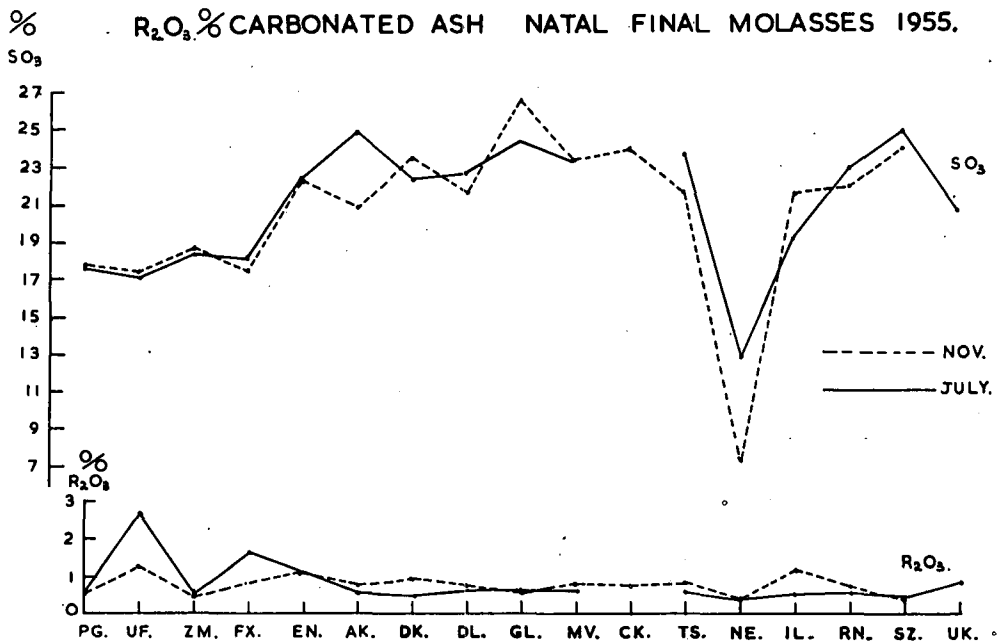


FIG. 7.

APPENDIX A
METHODS USED IN THE ANALYSIS OF
MOLASSES SAMPLES

1. Polarisation

The polarisation of the samples was determined according to the method given in the Recommended Methods of Chemical Control, page 29.

2. Sucrose by Jackson and Gillis Method IV

26.0 g molasses weighed out and transferred to a 200 ml volumetric flask. 50 ml 10 per cent basic lead acetate solution added and solution made to the mark, mixed well and filtered. Two 50 ml portions of the filtrate are pipetted into two 100 ml sugar flasks. To one is added 10 ml of sodium chloride solution containing 231.5 g/l—made to volume and polarised in 200 mm tube at 20°C. To the other is added 20 ml of water. The solution is heated to 65°C in a water bath and 10 ml hydrochloric acid ($d_{20}^{20}/4$ 1.1029) are immediately added. The solution is mixed by rotating and set aside for at least thirty minutes. Cooled to room temperature and made to the mark, mixing the contents by rotating just before the liquid reaches the level of the neck. The solution is shaken and polarised in a 200 mm tube at 20°C.

3. Sucrose by Lane and Eynon Method

26 g of molasses weighed out and transferred to a 200 ml volumetric flask. 35 ml of 10 per cent neutral lead acetate added and solution made up to 200 ml and filtered. 1 g of dry potassium oxalate added to the filtrate, the solution is left to stand for a few minutes and then filtered through No. 40 Whatman filter paper—20 ml of this filtrate is pipetted out and made to 200 ml for the determination of Reducing Sugars—Solution titrated against 10 ml of Fehling's solution, using methylene blue as internal indicator.

50 ml of the filtrate are pipetted out and diluted to approximately 80 ml and brought to pH 4.7 ± 0.2 by the addition of glacial acetic acid. To this 1.6 ml of invertase solution is added (amount depending on the concentration of sucrose and the activity of the invertase solution). The flask is placed in a water bath maintained at 57°C for 15 minutes or left to stand overnight. After cooling the contents of the flask are transferred quantitatively to a 500 ml flask and made to volume. This solution is titrated against 25 ml of Fehling's solution.

4. Solids by Drying

Using vacuum and operating technique designed by Gardiner and Farmiloe,⁶ who found the best value for the ratio of total water to weight of solids was 4.8 and similarly the volume of diluted molasses required was that corresponding to 0.4 g solids. Two

equations were devised to satisfy these conditions:

$$\text{Water to be added} = (\text{weight of molasses taken}) \left[\left(\frac{\text{Percent of solids}}{17.2} \right) - 1 \right]$$

$$\text{Volume of diluted molasses} = \frac{37.4 (\text{weight of diluted molasses})}{(\text{per cent solids}) (\text{weight of molasses})}$$

In equations (1) and (2) uncorrected value of refractometer solids used.

25 g of molasses weighed out into a 200 ml beaker with glass cover and stirring rod. As calculated from equation (1) about 90 ml water are added and the whole is then stirred covered and reweighed. The diluted molasses is transferred to a 150 ml stoppered flask. The calculated volume of diluted molasses, which from equation (2) approximately equals 2.2 ml is transferred by means of a graduated 5 ml pipette, into the centre of dry aluminium powder in an aluminium dish. The stoppered flask being well shaken before aliquots are transferred to the dishes. The weight of the sample being found by difference. The dishes contain 0.8 ± 0.1 g of aluminium powder, the dishes, powder and stirring rods having been previously dried in an oven at 105°C for one hour. After adding the diluted molasses and very careful mixing with the glass rods, which are subsequently left inside, the dishes are placed in the oven with their lids underneath them and dried for approximately 16 hours (overnight) with an air bleed of 100 ml per minute. The starting temperature is 50°C and the temperature is gradually raised to 70°C and kept there for the remainder of the drying period.

The lids are placed over the dishes in the oven recess before they are transferred to the copper cooling block in the desiccator.

5. Glucose

12 g of molasses are washed into a 600 ml Erlenmeyer flask, using approximately 75 ml water. 25 g of yeast are added and fermentation is allowed to proceed for at least four hours at room temperature. The contents of the flask are then quantitatively washed into a 250 ml graduated flask and 25 ml of 10 per cent neutral lead acetate are added. After making to the mark, the neck of the flask is dried with filter paper, $\frac{1}{2}$ g kieselguhr is added and after thorough mixing is filtered. The first few ml are discarded.

50 ml of the filtrate are pipetted into a 100 ml flask and 5 ml of the standard sodium phosphate-potassium oxalate mixture are added (7 g $\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O} + 3$ g $\text{K}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$ to 100 ml). Solution made to the mark, mixed well and filtered. 25 ml of filtrate used for the determination of reducing sugars by the Luff-Schoorl method.

⁶ S. D. Gardiner, *The Analyst* 78 (1953) 709. S. D. Gardiner and F. J. Farmiloe, *The Analyst* 79 (1954) 447. S. D. Gardiner and F. J. Farmiloe, *The Analyst* 80 (1955) 557.

Blank determination is done using water instead of molasses and the result is deducted from the molasses result. Percentage glucose is expressed as invert sugar.

6. Gums

10 g molasses weighed out into 250 ml beaker and diluted with 20 ml water. 150 ml acid alcohol added slowly and the solution is left to stand overnight and then filtered through a Gooch crucible previously dried in the furnace. After washing the crucible is dried in an oven at 105°C for 1 to 2 hours, after which it is cooled and weighed. After cooling and weighing the crucible is ignited in the furnace, cooled and reweighed. The difference in weights gives the amount of gums present.

7. Starch

10 g of molasses diluted with 20 ml of water. 90 ml of alcohol are added and 2 g of kieselguhr. The solution is allowed to stand for 1 to 2 hours before filtering through a paper pre-coated with 2 g of kieselguhr fitted on a 5 cm diameter Buchner funnel. The cake is washed with alcohol and then transferred to a 250 ml beaker with 40 ml of 35 per cent CaCl_2 solution and mixed thoroughly. Mixture is covered and kept boiling for 15 minutes, cooled and transferred quantitatively to a 100 ml flask and made to volume. An extra 1.7 ml of water is added to compensate for the volume occupied by the kieselguhr. The mixture is centrifuged until a clear supernatant solution is obtained. An aliquot is then taken for the determination of starch by the colorimetric method using 2N acetic acid, 10 per cent K.I solution + 0.1 N KIO_3 .

8. Wax

15 g of molasses are weighed out and mixed with 100 ml of water. 20 ml 10 per cent neutral lead acetate is added and 4 g of kieselguhr. The solution is left to stand overnight before filtering through paper previously pre-coated with 2 g of kieselguhr fitted on a Buchner funnel. The cake is dried and extracted in a Soxhlet apparatus using chloroform as a solvent.

9. Karl Fisher Method

The two burettes were filled and zeroed and the electrode system dried. 0.4–0.8 g of molasses was weighed out in a cooled dry titration vessel containing two stirrers. The vessel was put in place and the apparatus switched on with the stirrer set at the slowest speed. Karl Fischer reagent was run into the titration vessel until a permanent excess was recorded. Stirring was then continued for an extra 5 minutes before back titrating with standard methanol.

Standardisation was carried out in exactly the same manner using 2–3 drops of water from a weighing pipette.

10. Brix

Brix at three different dilutions was determined using a specific gravity bottle. Refractometer Brix was determined at 20°C.

11. K_2O , Na_2O

Determined at the S.A.S.A. Experiment Station, using a flame photometer.

12. Crude Protein

Nitrogen was determined at the S.A.S.A. Experiment Station by the Kjeldahl method. The result was multiplied by 6.25.

The Chairman (Mr. Du Toit) said that the paper contained a lot of very useful information and also a challenge.

Dr. Dodds was interested in the relative proportions of non-sugars in the molasses. He wondered if the difference might be due partly to different proportions of the various varieties.

Dr. Douwes Dekker said that it was a most important point as to whether the differences in non-sucrose composition were due to varying proportions of varieties or not. He pointed out, however, that the figures given were average samples taken over a week and contained molasses from many different varieties. To test any such difference would mean testing the mixed juice and bagasse rather than the molasses.

Mr. Narbeth wondered if the large quantities of lime used in the Natal Estates carbonatation process did not introduce large quantities of magnesium oxide and whether this gave any trouble in the process.

Mr. Rault replied that the standard white lime used was not high in magnesium oxide. A new source of limestone had been tried in the last few years, which gave after burning a coloured lime, due to the presence of manganese. It also had a percentage of magnesium content. However, magnesia was apparently nearly all removed by the alkaline reaction during liming, and the mass action of the large quantities of lime used in the carbonatation process.

Dr. Douwes Dekker agreed that the carbonatation process was more effective in removing magnesia than were the other processes. It was also known that the magnesia content of South African juices was higher than that of other countries.

Mr. Alexander said that the brownish colour experienced by Mr. Rault with certain limestones was due to manganese not magnesia.

Mr. Hastilow said that he had found with lime supplied by a local concern that when slaked with purified water this lime was satisfactory, but the silica content was increased if purified water was not used.

Dr. Douwes Dekker said that the specification for good lime was not more than 2 per cent silica.

Mr. Leclezio said that he was particularly struck by the high potash content of molasses in certain cases and he wondered if this was due to the heavy application of potash as fertilizer.

Mr. Du Toit pointed out that the high potash contents were shown at Pongola and Umfolozi where less potassium was used as a fertilizer. The figures were high in these two cases, in one case because the land was alluvial and in the other because it was virgin soil. He wanted to comment on the excellent agreement found at the S.M.R.I. between the Jackson & Gillis method and the Lane & Eynon method. Another interesting point was the geographical distribution of sodium and that the factories nearer the sea had a higher sodium content in molasses than did those far away from the sea. He wondered however why the high content of sodium in Natal Estates molasses should be.

Mr. Rault pointed out that the high sodium content was due to their introducing an appreciable amount of sodium carbonate to get rid of lime salts in their process. This, however, did not lead to their producing more molasses. As far as high potash content was concerned, he had found that potash was high in their molasses ash years ago when little of that element was added in the form of a fertilizer.

Dr. Douwes Dekker said he could not see that the addition of small quantities of sodium carbonate could have any material effect on the sodium content of the molasses.

Mr. Rault said they used quite heavy applications of sodium carbonate which must find its way into the molasses.

Mr. Leclezio asked at what stage the sodium carbonate was added and **Mr. Rault** replied that it was applied immediately after the second carbonation, where it was found that further gassing of the juice under pH 8 did not precipitate more lime so sodium carbonate was then added, which reduced the residual lime salt content in the juice by about thirty per cent. This led to less scaling of the evaporator and, more important still, an immediate improvement in the ash content of the refined sugar.

Mr. Thumann commented on the big differences in the brix which depended on the dilution and this of course must affect the purity figure to 2 or 3°, so that when our purities were compared with those of other countries he would like to know if the same brix dilution were used. Would it not be better, therefore

if the brix method were not used, but solids determined by diluting down strongly and then drying on a paper rosette. He mentioned in this connection that the refractometer brix was nearer to the true brix than any determined by hydrometer. He wanted to know why the big differences in carbonated ash were shown between different factories. Could this be due to clarification? Could the high K_2O be due to soil conditions and could it possibly be due to flooding of alluvial soil. He wondered if some suitable method for eliminating phosphatides could be found.

Dr. Douwes Dekker said as far as brix dilution was concerned, when gravity purities were compared, one had to know the dilution used for brix determination. The recommended method here was a 1 : 1 dilution. However, not all factories used this dilution. As far as the comparison with Hawaii and Java was concerned, in Java the purity was true purity, the brix being determined by drying. **Dr. Douwes Dekker** said that the factory control in Hawaii was completely based on the refractometer brix. He was not sure if it could be universally applied here. It was of course much quicker to work on the refractometer brix rather than on spindle brix. However, refractometers were seldom as accurate as spindles and therefore one had to be careful in their application. Refractometer brix was of course far closer to the true dry matter than was the spindle brix.

Mr. Du Toit remarked that when the potash content was very high it could have a detrimental effect on crystallisation, as shown by a factory in Hawaii where potash was so high that potassium chloride crystallised out with the sugar. This was overcome by mixing the massecuite with final molasses from an area where the potash was low.

Mr. Rault referred to the high brix of 90° shewn for some factory molasses in Mauritius and asked **Mr. Leclezio** why this was so. He suggested that in South Africa we were perhaps not tightening our massecuites sufficiently.

Mr. Leclezio attributed this apparently high brix to the method employed in Mauritius for brix determination; a 10 per cent weight: volume solution was made as contrasted with the 1 : 1 dilution in Natal.

Mr. Boyes pointed out that one objection to the use of the refractometer was that it could only measure up to 85° brix.

Mr. Antonowitz said that the importance of non-sugars in the factories was not fully realised. Chemists were usually tied down to accounting sucrose and so were not able to help the factory superintendent to solve his problems.

Mr. Hugo considered that all countries should use the same dilution for brix determinations.

Mr. Rault asked whether the brix represented the actual centrifugal effluent, if any dilution of molasses was made before the samples were sent to the Sugar Milling Research Institute because the brix was low in many cases.

Dr. Douwes Dekker replied that the factories were specifically asked not to send diluted molasses.

Mr. Leclezio asked what was the percentage of aconitic acid in molasses here.

Dr. Douwes Dekker replied that tests showed that the content was very low and it would not pay to try to recover this from the molasses.

The Chairman stated that this was a very valuable paper which would form an invaluable part of our Proceedings and would no doubt often be referred to in future.