THE COMPOSITION OF SOUTH AFRICAN SYRUPS

By E. W. GRAHAM, B. F. WRIGHT and J. B. ALEXANDER

Recently the Sugar Milling Research Institute has analysed both sugar and molasses samples from the majority of S.A. sugar factories. The variation of composition, particularly of some of the sugars, posed the question as to whether differences were due mainly to clarification or boiling techniques. By analysing syrup samples from all mills it was hoped to gain some insight into this problem.

The investigation in the case of syrup samples was complicated by the fact that syrups deteriorate rather rapidly if unpreserved at the prevailing temperature in Natal and it was found impractical to collect and freeze representative samples from all mills. It was decided therefore to rely on the bacteriocidal properties of mercuric chloride.

During May 1958, 4 half-gallon plastic bottles containing preservative were despatched to each mill together with instructions on how the samples should be collected. In spite of 2 mls. of S.A.S.T.A. preservative[1] being added to each container, some samples arrived at the Institute in an advanced state of deterioration, and had to be discarded. In each of the containers for the December samples was placed 5 mls. of preservative but the CK and FX samples still showed signs of deterioration. The analysis of the syrup samples is shown in Table I which also shows whether the syrup was settled at the factory or not.

Brix

The refractrometric brix was 0.65 higher than the solids found by drying as compared with 2.35 higher found in molasses[2]. The average brix of syrups is considered to be very low and only six of the factories approached the 60° brix value for their syrups. In some cases, the low brixes may be excused because of heavy scaling of the evaporator but in many cases it is felt that considerable improvements could be achieved by raising the brix of syrup to the 60-65° range.

Purity

The average purity of the December samples was 1.3 units higher than that of the June samples and only in cases where there is an increase of reducing sugar content from June to December is it found that there is a drop in the purity figure. In view of the fact that positive deterioration was found in the CK and FX December samples, the possibility of there being some slight deterioration in some of the other samples places doubt on the value of the purity and reducing sugar data.

Polysaccharides

Both the gums and the starch figures increase from June to December; the 60 per cent increase of the starch content being the largest of any of the changes in composition found for any constitutent for which the samples were examined. Similar increases were found in the case of molasses samples and there seems little doubt that the polysaccharide content of canes in Natal increases till the middle and end of the crushing season. Mills that normally were found to have high starch contents in their raw sugar were found in general to suffer the same defect in their syrup.

Lipids

As was found in the case of both sugars and molasses, defecation samples contained considerably more lipids than did their sulphitation counterparts. It is undoubtedly this increased lipid content that adds a great deal to the haziness of defecation clarified juices when compared with sulphitation clear juices. The increase in lipid content from June to December parallels that found for molasses samples.

Protein

The protein contents of samples from the mills further North appear to be higher than those in the South. The single mixed juice carbonatation sample shows the lowest value of all. A slight average increase in protein content was found from June to December.

Ash and Individual Inorganic Constituents

The variation in sulphated ash content of syrups was not found to be great or to vary much from June to December. Contrary to common belief it was found that the average sulphated ash for the sulphitation mills (3.23%) was lower than that of the defecation mills (3.56%). However, since a large proportion of this difference can be accounted for by the potash contents and only about a fifth by the CaO and inorganic P_2O_5 contents it seems likely that the original composition of the raw juice may account for the difference to a large extent. The large difference found between the inorganic phosphate content of defecation and sulphitation syrup samples was also found in the case of sugars and molasses. This too will account partly for the greater clarity of sulphitation juices.

Conclusions

The composition of the syrups from which Natal raw sugars are boiled probably determines to a large extent the impurities which may be found in the raw sugar. It is not suggested, however, that improvements in the boiling and curing of raw sugars will not yield improved results in many cases.

REFERENCES.

[1] S.A.S.T.A. Recommended Methods of Chemical Control, p. 18.
[2] K. Douwes-Dekker: The Composition of S.A. Final Molasses. Proc. S.A.S.T.A. Congress (1957), p. 92.

ANALYSIS OF SYRUP SAMPLES

Factory	Date	Settled or Unsettled	No. of samples	Ref. Brix	Dry Substance	Sucrose	Brix	Red. Sugars % on Brix	Gums % on Brix	Starch % on Brix	Lipid % on Brix	Protein % on Brix	Sulph. Ash % on Brix	K_2O % on Brix	SO_3 % on Brix	Cl % on Brix	CaO % on Brix	MgO % on Brix	SiO_2 % on Brix	P_2O_5 (org.) % on Brix	P_2O_5 (inorg.) % on Brix	Fe_2O_3 % on Brix
PG	June	Unsettled	4	58.5	57.2	51.8	88.5	3.15	0.71	0.071	0.029	0.89	4.06	1.50	0.68	0.44	0.25	0.17	0.067	0.015	0.009	0.004
	December	,,	1	60.4	60.2	53.5	88.6	4.10	0.77	0.131	0.063	0.78	3.99	1.36	0.71	0.39	0.28	0.14	0.081	0.028	0.021	0.003
UF	June																					
	December	,,	1	57.6	57.2	51.5	89.4	3.55	1.11	0.095	0.115	0.85	3.82	1.25	0.64	0.61	0.29	0.21	0.099	0.023	0.025	0.004
ZM	June	,,	4	50.4	49.5	44.3	88.0	3.19	0.97	0.069	0.125	0.70	3.89	1.21	0.55	0.60	0.31	0.27	0.106	0.022	0.024	0.007
	December	,,	1	47.3	46.8	41.1	86.9	4.42	1.23	0.107	0.222	0.73	3.87	0.78	0.57	0.58	0.38	0.31	0.103	0.033	0 030	0.009
FX	June	,,	4	50.8	49.8	43.7	86.1	3.67	1.10	0.062	0.134	0.89	4.01	1.01	0.55	0.55	0.48	0.24	0.091	0.017	0.052	0.010
	December	,,	1	47.3	46.7	41.2	84.4*	7.73*	1.07	0.075	0.117	0.92	4.02	1.04	0.67	0.58	0.47	0.26	0.058	0.030	0.033	0.005
EN	June	,,	1	60.8	60.2	54.6	89.8	2.81	0.75	0.093	0.062	0.61	2.81	0.73	0.62	0.31	0.29	0.28	0.051	0.010	0.009	0.004
	December																					
AK	June	,,	4	53.9	53.5	47.3	87.6	3.68	0.99	0.072	0.109	0.59	3.39	0.81	0.57	0.46	0.32	0.26	0.094	0.020	0.035	0.007
	December	,,	1	50.0	49.5	43.8	87.6	4.14	1.07	0.124	0.121	0.73	3.43	0.77	0.61	0.52	0.44	0.23	0.086	0.027	0.031	0.005
DK	June	,,	2	51.4	49.7	44.5	86.7	3.66	0.57	0.059	0.013	0.63	3.10	0.84	0.67	0.39	0.32	0.25	0 058	0.011	0.004	0.005
	December	,,	1	55.4	55.4	50.4	91.0	3.04	0.74	0.072	0.019	0.74	2.60	0.63	0.57	0.34	0.29	0.21	0.052	0 014	0.002	0.003
GS	June	,,	2	51.1	50.9	45.2	88.6	3.43	0.65	0.042	0.027	0.66	3.41	1.01	0.57	0.42	0.29	0.25	0.055	0 013	0.002	0 005
	December	,,	1	44.1	43.8	38.7	87.8	4.11	0.83	0.065	0.034	0.61	3.43	0.76	0.58	0.42	0.42	0.22	0.046	0.014	0.003	0.004
DL	June	,,	4	55.8	54.7	48.6	87.1	3.87	0.94	0.086	0.115	0.70	3.37	0.90	0.57	0.46	0.26	0.28	0.085	0.017	0.022	0.006
	December	,,	1	52.6	52.2	46.6	88.6	3.98	1.11	0.130	0.149	0.58	3.20	0.78	0.60	0.47	0.37	0.23	0.063	0.030	0.034	0.006
GL	June	,,	4	54.7	53.7	47.1	87.8	3.53	0.75	0.067	0.018	0.64	3.30	0.88	0.64	0.44	0.30	0.26	0.064	0.017	0 004	0.004
	December	,,	1	54.9	54.3	49.2	89.6	3.36	0.98	0.128	0.037	0.60	3.15	0.75	0.61	0.42	0.33	0.26	0.048	0.018	0.007	0.003
MV	June	,,	4	53.1	52.1	46.0	86.7	3.76	0.82	0.064	0.089	0.69	3.85	0.92	0.59	0.45	0.57	0.23	0 060	0.014	0.036	0.007
	December	,,	1	51.4	50.9	45.6	88.7	3.27	1.18	0.099	0.128	0.49	3.49	0.74	0.54	0.43	0.57	0.20	0.047	0.023	0.041	0.007
CK	June	,,	4	52.1	51.1	45.0	86.4	4.15	0.76	0.064	0.026	0.60	3.46	0.76	0.62	0.37	0.44	0.26	0.049	0.015	0.005	0.005
	December	,,	1	40.2	38.5	37.6	84.3*	6.82*	1.09	0.103	0.039	0.82	3.46	0.72	0.73	0.42	0.43	0.31	0.048	0.018	0.005	0.004
TS	June	,,	4	55.3	54.6	48.2	87.2	4.52	0.89	0.084	0.111	0.63	3.10	0.84	0.57	0.39	0.29	0.27	0.075	0.018	0 058	0.005
	December	,,	1	54.5	54.1	48.2	88.4	3.84	1.17	0.132	0.120	0.69	3.07	0.78	0.52	0.43	0.27	0.27	0.059	0.029	0.044	0 005
NE	June		4	58.7	58.5	53.0	90.3	2.63	0.28	0.017	0.014	0.41	2.50	0.76	0.17	0.36	0.28	0.10	0.016	0.004	0 001	0.002
	December																					
IL	June	Unsettled	4	59.4	58.7	51.2	86.3	4.74	0.73	0.063	0.053	0.68	3.87	1.20	0.61	0.50	0 28	0.26	0.090	0.023	0.014	0.007
	December	,,	1	59.8	59.7	52.9	88.5	4.16	1.08	0.098	0.145	0.76	3.50	0.93	0.54	0.50	0.31	0.27	0 071	0.026	0.018	0.006
RN	June	,,	4	59.2	58.5	50.7	85.6	5.62	0.95	0.061	0 019	0.48	3.36	0.88	0.59	0.43	0 27	0 32	0.062	0.014	0.003	0.004
	December	Settled	1	58.3	58.3	51.3	88.0	4.35	1.13	0.072	0 038	0.52	3.45	0.83	0.71	0.50	0.32	0.31	0.059	0.017	0.003	0.003
SZ	June	Unsettled	3	55.2	54.2	47.5	86.1	4.78	0.83	0.055	0.012	0.56	3.28	0.89	0.64	0.40	0.29	0.30	0.090	0.016	0.003	0.005
	December	,,	1	55.8	55.2	49.7	89.1	3.79	1.05	0.091	0.025	0.61	3.24	0.73	0.73	0.39	0.29	0.30	0.060	0.020	0.001	0.005
UK	June	Unsettled	3	51.5	50.9	44.7	86.8	3.80	0.85	0.069	0.088	0.57	3.46	0.97	0.55	0.44	0.35	0.22	0.058	0.015	0.032	0 007
	December	Settled	1	48.4	47.9	43.2	89.3	2.68	1.03	0.145	0.164	0.60	3.20	0.82	0.55	0.44	0.33	0.25	0.055	0.020	0.030	0.005
Average	June			54.8	54.0	47.9	87.4	3.82	0.80	0.065	0.061	0.64	3.42	0.95	0.57	0.44	0.33	0.25	0.071	0.015	0.018	0.006
	December			52.4	51.9	46.5	88.7	3.77	1.04	0.104	0.096	0.69	3.43	0.85	0.62	0.47	0.36	0.25	0.065	0.023	0.021	0.005

* Not included in averages.

Mr. du Toit (in the chair) said this paper was most interesting and full of information. He commented on the considerable difference in the lipid content between defecation and sulphitation processes. He wanted to know if there was much difference in the gum and starch content between the two clarification processes.

Mr. Alexander said that he had not worked this out completely but as far as he could remember there was very little difference as far as starch and gums were concerned between the two processes.

Dr. Douwes-Dekker asked if it would be possible that the deterioration in two cases might mean that the bottles were washed out thus removing the mercuric chloride before the syrup was added.

Mr. Alexander said that notices were made and pasted on the bottles to the effect that these bottles contained the preservative.

Mr. Davies said that although he had not handled the samples himself he thought that due care was taken to make sure that the instructions were properly carried out.

Mr. Rault asked if the amount of mercuric chloride added would not add to the chloride content of the syrup. He also pointed to the fact that Pongola had twice the potash content of the Natal Estates' molasses.

Mr. du Toit said that tests done at the Experiment Station showed similar high potash in soils and juice from Pongola as compared with the older cane producing areas.

Mr. Rault asked if the relationship between potash and sucrose content, found at Pongola, was of general significance.

Mr. du Toit replied that there was a connection but he did not think that Pongola's high sucrose was due to the high potash content. He said that in the case of a very deficient potash soil if the addition of potash gave an increased yield, one could expect also an increase in the sucrose content. This would only be true until the potash deficiency had been satisfied. Extra potash would not increase the sucrose content.

Mr. Galbraith said that the low molasses purity obtained on the South Coast had been said to be caused by a peculiarity in the South Coast cane. He asked Mr. Alexander if he had any information on the subject.

Mr. Alexander said that he had not studied these figures from that point of view, but quoted figures from Dr. Douwes-Dekker's paper on molasses of some years ago. He pointed out that according to the Douwes-Dekker formula, the July molasses samples were only 1.9 units above the expected purity, while the November samples were 5.4 units above the expected purity. He wonders whether this discrepancy might not be attributed, at least partly, to the increased starch and gum content in the November samples.

Dr. Douwes-Dekker said that gums and starch were often accused of lowering the exhaustibility of molasses, but he had never seen reliable proof of this. He wanted to know from Mr. Beesley if he found the amount of these substances was very low in the middle of the year.

Mr. Beesley said that at Illovo final molasses gravity purities for the 1958-59 Season were about 30 degrees for May and June, rising to a maximum of about 35 degrees in October-November and dropping again to about 32 to 33 degrees in February. Unfortunately he did not have any figures for starch and gums for the Season.

Mr. du Toit pointed to the carbonatation process figures for starch and gums as being very low indeed.

Mr. Rault remarked that the high alkalinity ranges of operations during clarification by carbonatation was a cause of larger reducing sugar elimination, leading to a comparatively higher purity of final molasses. This was however compensated by a more energetic removal of other non-sugars not obtainable by the common juice purification processes and resulted in a smaller volume of these final molasses. These non-sugars removed were comprised of organic substances such as gum, wax and proteins, but also of mineral impurities, magnesia, silica, sulphate and phosphates. The potential reduction of sucrose lost in molasses had not been realised through shortage of boiling house equipment by attempting to produce 100 per cent refined sugar by a 3 massecuite system on similar lines to the beet sugar factories, where remelted second sugar liquors are blended with well purified thick juices from the carbonatation process in a continuous flow of liquor. It is unfortunate that in the cane industry, the refining of sugar must be separated from the recovery house as the colour question is always in conflict with the attempt of lowering the purity of molasses.

Mr. du Toit said that at the syrup stage the carbonatation process started with a considerable advantage which could be made use of if there were more plant available. Much better results could be expected because not only were the organic impurities very low but also the inorganic compounds were reduced.

Mr. Boyes said that the paper showed that they were now beginning to record certain impurities which hindered the crystallisation of sucrose—one of these was gums. We had considerable data showing the amount of gums, and should try and go further and find a means of getting rid of gums and starches. He said the use of enzymes in trying to get rid of starch was very costly but he wondered if it would not be an advantage if experiments were conducted to test the effect of enzymes on gums. Commenting further, he added that as large amounts of lime were used, one would expect to find a high lime content in juices, but not considerable quantities of magnesia as shewn by the figures. Magnesia was therefore not removed by the ordinary defecation process, and he asked if it had much effect on the crystallisation of sucrose.

Mr. du Toit said that the magnesium content was most interesting and he had always been of the opinion that the magnesium content of our cane was very high. It was interesting to see that at Pongola magnesium was particularly low in the molasses analysis given some years ago. In the more deficient potash areas lower down the coast more magnesium was taken up by the plant. As far as the elimination of starch and gums by microbial process was concerned, he asked if Mr. Bruijn could throw some light on this aspect.

Mr. Bruijn said that some factory tests carried out at Melville showed that by keeping the juice for a period at a high temperature the starch was reduced by 40 per cent.

Dr. Douwes-Dekker said that, as shown by Mr. Boyes, the use of commercial enzymes to remove starch was probably not economical. Chemists in Australia had investigated the possibility of decomposing starch by utilising the naturally occuring enzymes. They had found that by heating mixed juice at about 5 pH to 70 or 75 degrees some 80 to 90 per cent of the starch was removed. If this method was applicable in the factory, the solution of the starch problem would be simple. The Australian method had been tried out in two South African factories and it had been found in the first instance that the amount of starch removed was much less than in Australia i.e. 50-60 per cent and furthermore that some sucrose was also decomposed. This made the problem more complicated. Another method of getting rid of the starch was to centrifuge the juice in a suitable separator. Tests were carried out some 5 years ago and it was found that the starch could be removed in this way. The experiment had to be discontinued because the sand in the juice eroded the centrifugal and put the machine out of action. The problem therefore was how to get rid of the sand before the mixed juice was passed through the separator. There was however, evidence available that by using a hydrocyclone sand could be removed and tests had been carried out with a view to thus getting rid of sand particles. This now gave us the opportunity to use the centrifuge for the removal of starch. It was hoped to conduct such tests in the coming season. The separator protected by the hydrocyclone it was hoped would compare with the Australian method which is costly because of destruction of sucrose. Admittedly the cost of the machinery was also high but it was hoped that much higher removal of starch would be obtained than by the Australian method.

Mr. Rault asked which were the harmful and which the harmless non-sugar present. It was known that reducing sugar was useful while gums were not, but could the others be so classified ?

Mr. Boyes said that the use of enzymes for removing starch was practical but more research was required to prove whether it was economical. The difficulty was that enzymes only reacted effectively with starch when the granules had gelatinised. This occurred at about 70°C which was considered the upper limit at which the enzymes were effective. The result was that to get appreciable breakdown large quantities of expensive enzyme were required. An overseas firm had been experimenting with heat-resistant enzymes but so far these had a low activity, removing about 25 per cent starch. Another good line of research was to study the different liming techniques for removal of various impurities which were known to affect the recovery of sucrose. Research on Tongaat juices indicated that defecation by hot liming removed starch more effectively than cold liming while the latter gave a superior removal of gums and wax. It would be interesting if other mills studied the quantitative removal of such impurities as silica, starch, wax and gums. In the past the S.M.R.I. had attempted to do this on their own and it was a pity that for obvious reasons data were given for only two months and not for each month of the grinding season as in this way trends could have been discerned.

Dr. Douwes-Dekker said that another possible solution was the breeding of cane with a low starch content and he pointed to the high starch content of N:Co.310

The Chairman said that this subject was worthy of very serious consideration. He thought that perhaps the starch determination should be included besides the sucrose content in canes at least.