A COMPARISON BETWEEN FACTORY AND LABORATORY BOILED RAW SUGARS

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Introduction

There is an inverse correlation between the refining quality of a raw sugar and the amount of non-sugars contained in it. Different non-sugars have, however, a different effect and it also matters whether the non-sugars are present inside the (washed) crystals or in the molasses film coating them. The former group is the more important one. They — as Dr. Honig puts it — syncrystallise together with the sucrose.

Our knowledge about the factors which govern the syncrystallisation is far from complete. We know that the amount of impurities increases when the purity of the mother liquor drops. B-sugar crystals are less pure than A-crystals and the concentration of the impurities increases from the innermost part of a crystal towards its surface. We also know that the growing sugar crystal has a preference for selected non-sugars. For example, reducing sugars syncrystallise to a very limited extent only, but certain ash components, starch and gums are apparently readily absorbed.

One can further imagine that the boiling conditions have an effect on the amount of non-sugars which syncrystallise. In fact, the concentration in a sugar crystal of, for example, starch expressed as a percentage of the concentration of the starch present in the mother liquor in which the crystal was grown fluctuates between fairly wide limits, not only between factories, but also in one factory (see later).

In the course of clarification tests, investigations into the influence of certain non-sugars and the influence of different varieties of cane on the filterability of raw sugar, a laboratory vacuum pan was constructed, which would enable factory type sugar to be produced from small quantities of material. The pan has previously been described by J. Bruijn (1). During preliminary investigations using this pan it was noted that the purity of sugar produced was superior to that produced by a factory from the same material. On the basis of these preliminary results an investigation was started to find out just how much more superior these laboratory produced sugars were, and to enquire why they should be so. The investigation has, however, not yet been completed and an explanation for the difference between the purities of the two types of sugar cannot be given. Nevertheless, it has been decided to publish the results obtained so far, since they should be of interest to anyone trying to improve the purity of his product, being it raw or refined sugar, and since they may stimulate further investigations in other laboratories.

Sampling Procedure

In order to achieve a representative sample of the material about to undergo crystallization at the factory, small stop cocks were fitted on the feed inlet pipes to the pan. From these cocks a continuous drip sample was obtained throughout the boiling period by means of a small portable vacuum pump. In certain factories this method of sampling was unnecessary, as samples could be taken (also as continuous drip samples) by means of the air bleed valve on the centrifugal pumps supplying the pan header feed tanks. In the case of those factories utilizing a magma in their boiling schemes a sample of the magma was obtained through the proof stick of the pan prior to the start of boiling.

Boiling Procedure

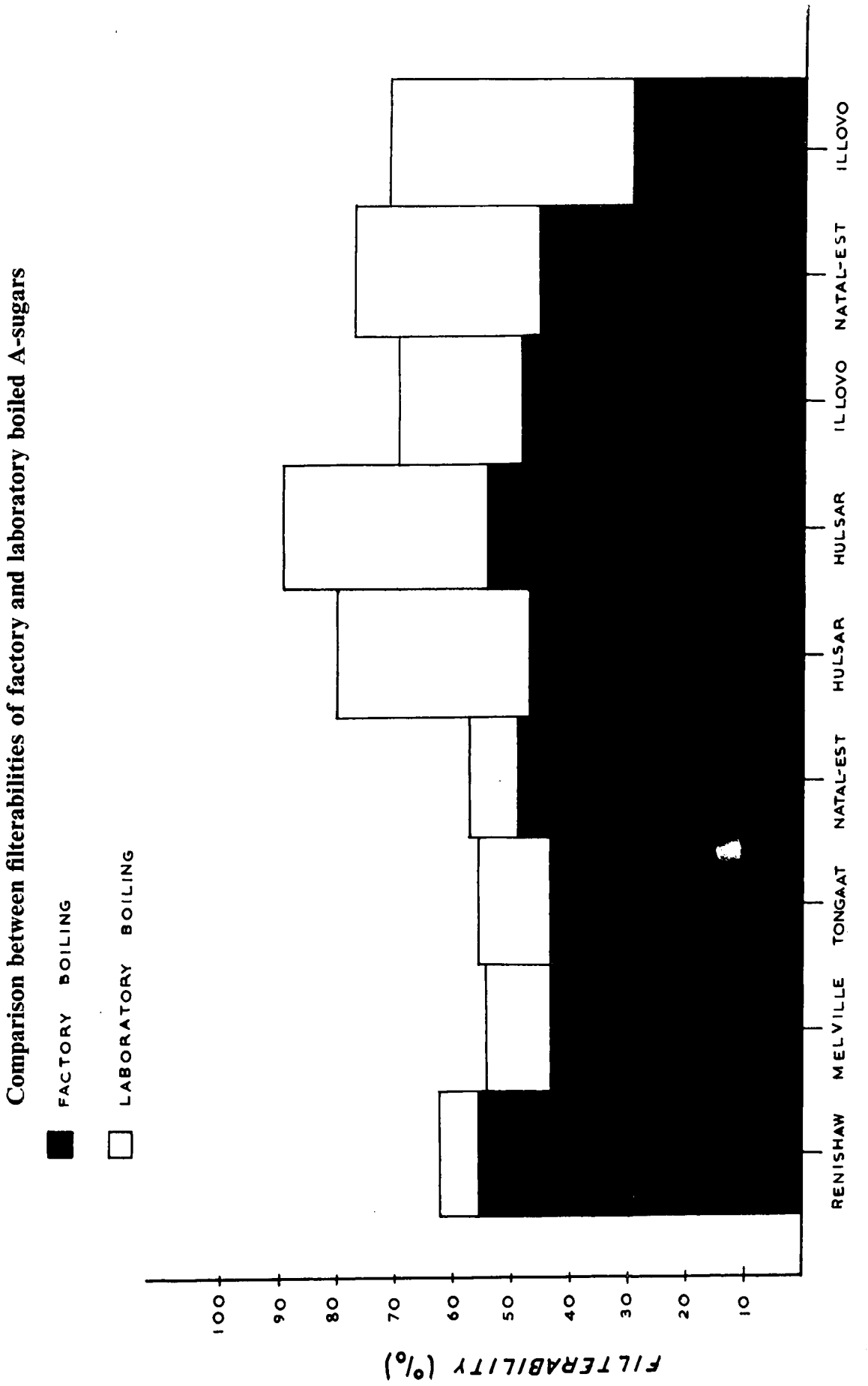
The method followed to produce as identical a boiling as possible, was to observe the factory boiling from the start of boiling operations, and to note each individual operation performed by the pan boiler as well as the conditions of steam, pressure, vacuum, massecuite temperature, conductivity, etc. The period of boiling, volume of massecuite at cut over, volume of massecuite after cutting over and complete boiling scheme were noted, to enable a facsimile of this factory boiling to be made in the laboratory. For this purpose the smaller of the two pans as described by Bruijn was used.

Samples of the sugar produced from both the factory and laboratory boilings were taken on striking the pan and analysed, as well as samples of the run off from these boilings.

Results

To establish a comparison between factory and laboratory boilings, samples of both A-sugars and later B-sugars were produced. In Table No. 1 are shown the results produced by the comparison of A-boilings. It will be seen that in every case the filterabilities of the laboratory produced sugars are higher than those of the factory samples (see Graph No. 1).

It will also be noticed that the difference in filterability between the two types of sugar was smaller for the first group of four boilings than for the second group of five which were boiled at a later date. It is perhaps of significance that in the period of time between the two groups the very vigorous stirrer described in Bruijn's paper was installed in the pan, the former stirrer being regarded as rather ineffective. It should, however, also be appreciated that at the later stage our experience in manipulating the pan and its contents according to a pre-determined programme had increased markedly. For the purpose of exhibiting the difference between factory and laboratory boiled sugars, the last five runs are therefore more appro-


Comparison between filterabilities of factory and laboratory boiled A-sugars
FACTORY BOILING
LABORATORY BOILING
FILTERABILITY (%)
100
90
80
70
60
50
40
30
20
10
RENISHAW
MELVILLE
TONGAAT
NATAL-EST
HULSAR
HULSAR
ILLOVO
NATAL-EST
ILLOVO

GRAPH 1

TABLE 1

Factory and Laboratory A-Boilings

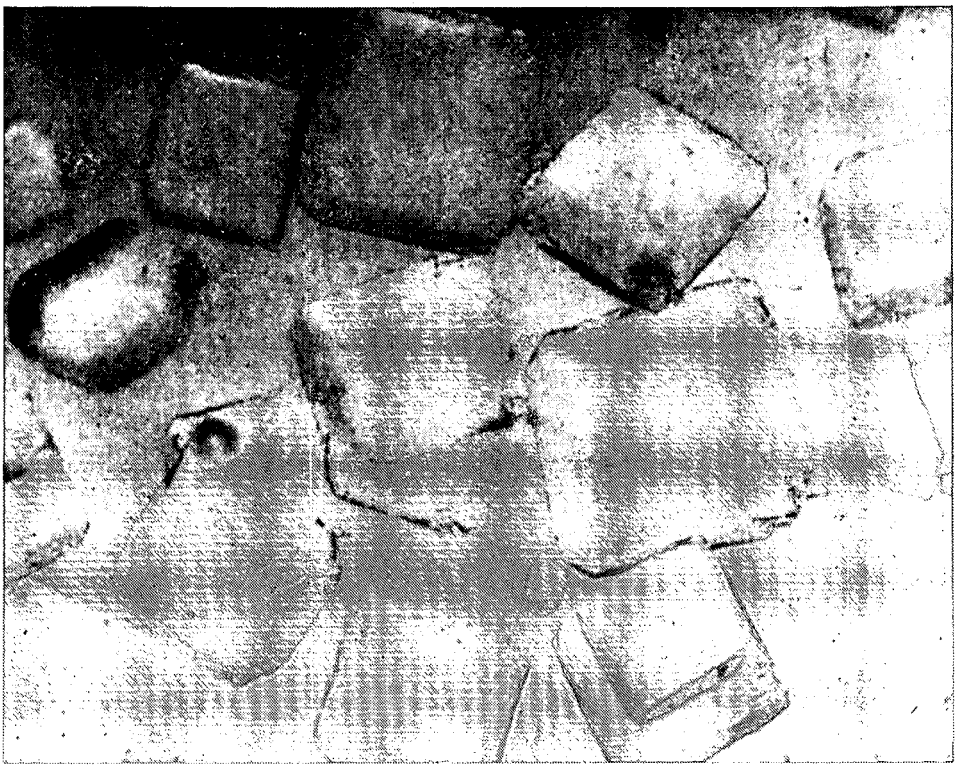
Sample and Analysis	Renishaw		Melville		Tongaat		Natal Estates		Hulsar		Hulsar		Illovo		Natal Estates		Illovo	
	Fact.	Lab.	Fact.	Lab.	Fact.	Lab.	Fact.	Lab.	Fact.	Lab.	Fact.	Lab.	Fact.	Lab.	Fact.	Lab.	Fact.	Lab.
Filterability %	55	62	43	54	43	55	49	57	47	80	54	89	48	69	45	77	29	71
S.G.S. (mm)	—	—	0.83	0.44	0.83	0.40	—	—	0.38	0.50	0.28	0.37	0.49	0.50	0.73	0.40	0.81	0.44
Starch (ppm)	—	—	215	135	330	120	—	—	390	170	310	105	390	35	530	165	170	110
Wax (ppm)	—	—	50	35	65	45	—	—	70	20	50	10	90	14	30	25	70	30
Gums (ppm)	—	—	715	485	1055	733	—	—	1100	750	100 0	550	888	310	700	600	950	750
Silica (ppm)	—	—	80	54	95	60	—	—	35	10	50	20	95	20	65	25	45	17
P_2O_5 (ppm)	—	—	21	20	26	34	—	—	39	6	20	5	34	6	10	10	30	7
Boiling time (hrs.)	—	7.00	—	6.00	—	7.25	—	4.50	4.00	5.25	3.50	4.50	4.33	4.50	5.33	5.50	5.25	5.50
Syrup Brix	52.2		56.8		56.8		62.4		—	—	—	—	67.5		56.1		57.6	
Syrup Purity %	82.3		89.1		87.3		84.9	84.9	—	—	—	—	87.4		87.0		84.7	
Re-melt Brix	—	—	—	—	—	—	—	—	—	—	—	—	—	—	64.1		65.6	
Re-melt Purity %	—	—	—	—	—	—	—	—	—	—	—	—	—	—	94.0		91.0	
Run-off from boiling. Brix	—	—	—	—	—	—	—	—	—	—	—	—	75.3	55.0*	65.2	67.7	50.1	61.7
Run-off from boiling. Purity %	—	—	—	—	—	—	—	—	—	—	—	—	80.3	78.2	74.1	76.8	75.8	73.9

TABLE 2

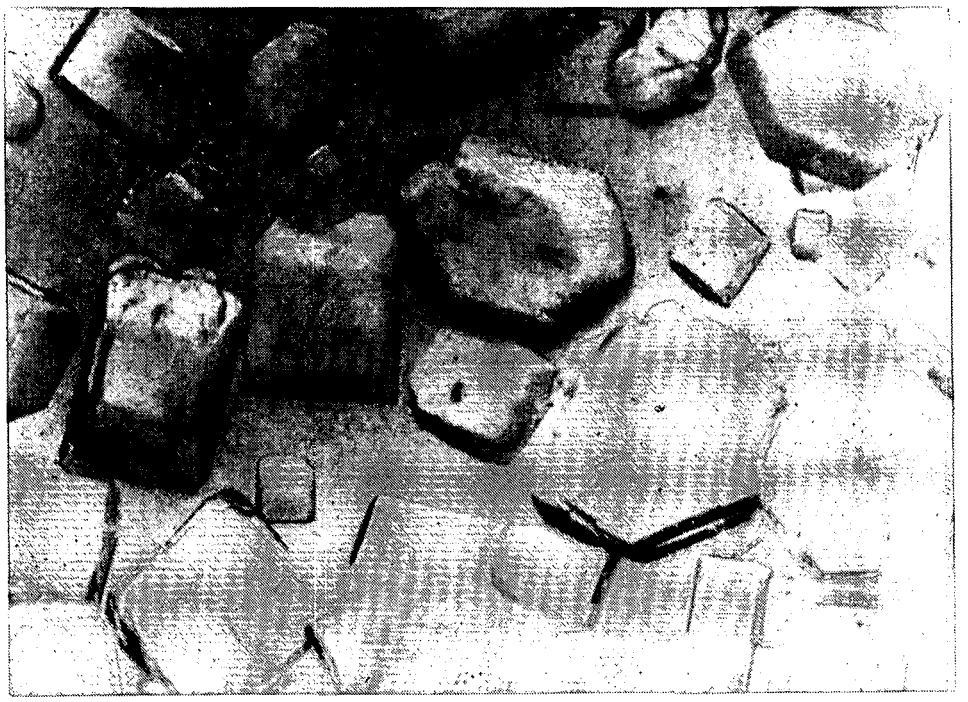
Factory and Laboratory B-Boilings

Sample and Analysis	Natal Estates			Illovo			Tongaat			Illovo		Tongaat		Natal Est.	
	Fact. (1)	Lab.	Fact. (2)	Fact. (1)	Lab.	Fact. (2)	Fact. (1)	Lab.	Fact. (2)	Fact.	Lab.	Fact.	Lab.	Fact.	Lab.
Filterability	15%	58%	16%	12%	44%	13%	21%	62%	18%	4%	30%	30%	57%	3%	61%
S.G.S. (mm)	0.55	0 48	0 60	0.43	0 31	0.44	0.48	0 32	0 45	0 46	0 39	0.48	0 37	0.66	0 35
Starch (ppm)	625	255	625	545	405	—	290	180	310	690	470	310	275	770	370
Wax (ppm)	60	20	70	80	50	90	140	50	180	290	100	100	40	80	30
Gums (ppm)	1900	1050	1900	1850	1400	1700	1400	800	1500	3150	1950	1450	1350	1950	1100
Silica (ppm)	135	35	140	155	55	145	135	40	145	195	65	115	55	255	40
P_2O_5 (ppm)	20	5	25	90	30	80	60	25	65	140	40	50	35	35	12
Boiling Time (Hrs.)	6.33	6.33	—	5.75	5.75	—	7 50	7.50	—	4.83	4 83	9.33	9.33	7.66	7.66
Cooling Time	—	—	21 Hrs	—	—	9 Hrs	—	—	14 Hrs	—	—	—	—	—	—
Syrup Brix	60 2		—	61.7	61.7	—	56.5	56.5	—	—	—	51 8	51.8	6 0	60 0
Syrup Purity	89.4%		—	84 4%	84 4%	—	82.1%	82.1%	—	—	—	84.9%	84.9%	88 0%	88.0%
A Molasses Brix	65.6		—	62.4	62 4	—	60 2	60 2	—	—	—	61 4	61.4	64.7	64.7
A Molasses Purity	77.7%		—	62 8%	62 8%	—	71.0%	71 0%	—	—	—	71.0%	71 0%	76.0%	76.0%
Run-off from boiling Brix	75.0	41.4*	78 7	55 5	47 4*	79 8	62 2	42 0*	—	55.4	51.1*	68 0	76 5	66 0	66 4
Run-off from boiling Purity	57.3%	63.8%	55.9%	56.2%	54.0%	52.6%	53 9%	59.0%	—	55.5%	61.0%	55.6%	61.2%	59.1%	60.8%

*Indicates Molasses has been diluted prior to analysis but after curing).

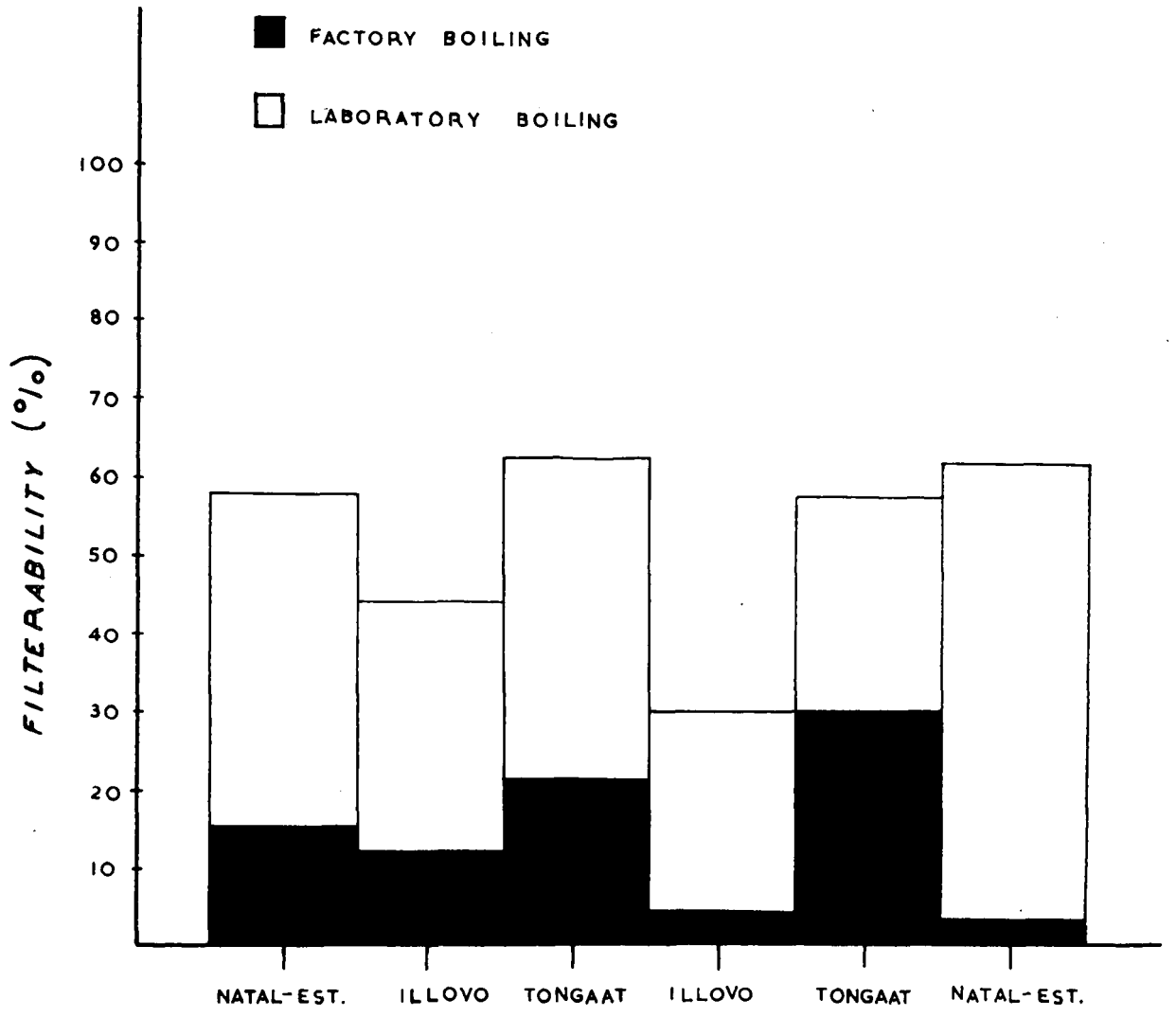


LABORATORY



FACTORY

Comparison between filterabilities of factory and laboratory boiled B-sugars



GRAPH 2

priate. The raw material boiled into sugar in these five boilings was (*a*) a mixture of affination syrup and last run-off from the refined sugar boilings as normally processed in the recovery department of Hulsar refinery and (*b*) and (*c*) syrup from Illovo and Natal Estates, respectively.

The average filterability of the factory boiled sugars of this group was 44% and of the laboratory boiled sugars 77%. The comparative figures for the group of first four boilings was 48% and 58% only. The filterability of the second group of laboratory boiled sugars was thus much better.

The specific grain size of the laboratory boiled sugars unfortunately tended to be smaller than that of the factory sugars. It would, however, be wrong to assume that the size of the crystals had a marked effect on their filterability, for of the Hulsar set of sugars the laboratory sugars are of the larger size, whilst the difference in one of the Illovo sets is negligible. The suggestion has also been made that the difference in filterability could possibly be due to the fact that the syrup samples, as collected at the factories, where they were worked without delay, had to be kept at the S.M.R.I. for some time before they were boiled into sugar, in which period chemical changes might have occurred. However, a separate series of runs showed that sugars boiled in the laboratory from one large sample of syrup at not inconsiderable intervals of time, showed nearly the same filterability.

As far as the non-sugars are concerned it will be seen that the factory samples are always higher. The individual differences will be discussed together with those of the B-sugars.

Photomicrographs of the crystals produced by both A-boilings of an Illovo sample indicate a more uniform and "clearer" crystal being produced by the laboratory boiling. These crystals appear to be free from fissures and inclusions as shown by the crystals in the photomicrographs of the factory boiling.

The results obtained by the comparison of B-boilings are shown in Table No. 2. Here again as with the A-boilings, the laboratory produced sugars show a very much higher filterability than the factory samples (see Graph No. 2). In one case the difference is as much as 58%, while the average increase of the filterability percentages for six samples is from 14 to 52%. In the comparison of B-boilings a second sample was taken of the factory produced sugar, after the massecuite strike from the pan had cooled in a crystallizer for a certain period of time. These samples are marked Factory (2) in the table of results, and indicate no change in filterability due to cooling. The difference in specific grain size is not as marked between factory and laboratory produced samples as in the A-boilings, the factory samples remaining, however, slightly larger. Non-sugar analyses again show a marked increase of the purity of the laboratory boiled over the factory boiled sugars. The average non-sugar contents of the (affinated) A- and B-sugars are shown in Table No. 3. The data for the A-sugars refer to the last five boilings only.

Table No. 3 shows that the laboratory boiled sugars whether A or B, contain about one third of the wax, silica and P_2O_5 present in the factory boiled sugars. A similar figure was found for starch in the A-sugars, but in the B-sugars the starch content of the laboratory boiled sugars is nearly two thirds of the other group.

We do not think, however, that with regard to syncrystallisation of starch we can speak of a typical difference between A- and B-sugars, for it would appear that in view of the variability of the data the number of runs in each group has been insufficiently large to allow us to draw this conclusion.

The laboratory samples contained about two thirds of the gums contained in the factory samples, both taken from the A- and B-boilings. One notices further that the factory B-strikes were better exhausted than the laboratory strikes, in particular when the former had passed the crystallizer. We are not inclined to ascribe to this feat a major effect on the quality difference between the two types of sugar.

Conclusions

The above investigation has shown that there is a remarkable difference in purity between sugars boiled from the same material in factory and laboratory pans. A firm explanation for this difference cannot be offered, although it is obvious that boiling conditions (temperature, supersaturation, etc.) must have played a role in it.

In this context the result of another investigation is of interest, namely that the fraction of the starch present in the mother liquor which syncrystallises with the sucrose varies considerably from boiling to boiling, and from factory to factory (see Table No. 4). There must be some, so far unknown, factor in our factory boiling technique which has an influence on the amount of starch absorbed by the growing sugar crystal. If we could locate this factor it would be possible to produce sugars containing the minimum amount of impurities obtainable under factory conditions, perhaps approaching those of the sugars boiled in the laboratory. This would be a major step forward in our endeavour to improve the refining quality of Natal raw sugar.

Reference

(1) J. Bruijn, The Construction of two laboratory vacuum pans, S.A.S.T.A. Proceedings 38 (1964) 102.

TABLE 3

Non-sugars in the Factory and Laboratory boiled (affinated) sugars

	A - SUGAR			B - SUGAR		
	Factory Sugar (a)	Laboratory Sugar (b)	$\frac{b}{a} \times 100$	Factory Sugar (a)	Laboratory Sugar (b)	$\frac{b}{a} \times 100$
	ppm	ppm		ppm	ppm	
Starch	358	117	33	538	326	61
Gums	928	592	64	1950	1275	65
Starch-free gums	570	475	83	1412	949	67
Wax	62	20	32	125	48	38
Silica	58	18	31	165	48	29
P_2O_5	27	7	26	66	25	38

TABLE 4

Syncrystallisation of Starch in Factory Boiled Raw Sugars

ILLOVO

Strike Number	Sugar		Massecuite	Molasses	Massecuite	$\frac{\text{Starch in Sugar (ppm)}}{\text{Starch in massecuite (ppm on Brix)}} \times 100$
	Filterability %	Starch ppm	Purity	Purity	Starch ppm on Brix	
2780	40	235	87.38	76.42	1300	18.1
2782	31	275	88.10	75.60	1350	20.4
2787	46	245	87.30	74.80	1340	18.3
2761	20	410	87.90	77.90	1295	31.7
2764	25	365	90.30	79.00	1380	26.4
2765	23	410	88.90	77.10	1495	27.4
2830	37	215	87.80	76.00	1355	15.9
2879	—	390	—	—	1350	28.9

RENISHAW

Sample No.	Sugar		Molasses Purity	Massecuite		$\frac{\text{Starch in Sugar (ppm)}}{\text{Starch in Massecuite (ppm on Brix)}} \times 100$
	Filterability %	Starch ppm		Purity	Starch ppm on Brix	
1	62	190	72.0	87.2	1255	15.1
2	—	185	76.4	88.1	1390	13.3
3	—	210	78.2	88.6	1235	17.0
4	—	145	77.7	89.8	1145	12.7
5	—	240	74.9	87.9	1175	20.4

MELVILLE

Sample No.	Sugar		Massecuite		Starch in sugar (ppm) / Starch in massecuite (ppm on Brix) x 100
	Filterability %	Starch ppm	Crystal content	Starch ppm on Brix	
1	61	190	57.8	1380	13.8
2	59	205	56.1	1610	12.7
3	—	265	52.9	1580	16.8
4	—	310	50.6	1620	19.1
5	—	375	50.4	1680	22.3
6	—	310	57.1	1635	19.0
7	—	225	54.9	1545	14.6
8	51	210	58.0	1565	13.4
9	—	290	59.0	1605	18.1
10	37	415	58.7	1550	26.8
11	48	300	55.4	1660	18.1
12	—	295	57.3	1500	19.7

Mr. Chiazzari (in the chair): These figures for filterability vary considerably from factory to factory and factory to laboratory. Do they represent one boiling from each factory or more than one boiling?

Dr. Douwes Dekker: One boiling. The differences do indicate variations in boiling techniques.

Mr. Maine: Would it not be possible to move the laboratory pan to the factories and make the boilings on site so as to get a true comparison in case the syrup sample deteriorates before it is boiled at the S.M.R.I.

Dr. Douwes Dekker: We thought of this but we have found that there is no difference in the quality of the sugar even if the syrup is kept for quite a long time in a refrigerator at the S.M.R.I.

Mr. Boyes: When the paper on the construction of laboratory pans was discussed last year it was agreed that if a boiling was made at a low supersaturation the quality of the sugar could be improved. It would have been interesting to have had the figures for massecuite brix and yields included in the paper.

Dr. Douwes Dekker: There was some dilution, so these figures are not indicative of the brix of the molasses spun off from the massecuite.

Mr. Warne: In table one the brix of the molasses has been diluted owing to the high supersaturation of the run-off because it tended to crystallize. Also, the boilings take so long that it was not always possible to do the purity and brix on the same day. We do, however, try and do the boilings as closely as possible to the way they are done in the factories.

Mr. Boyes: It appears that the molasses were supersaturated and there was not sufficient exhaustion. In practice of course you cannot take too long over boilings.

Mr. Warne: The boiling times in Tables 1 and 2 are as close as we could get to factory boiling times. The pan at the S.M.R.I. has a stirrer with a motor of a certain horsepower and it is not possible to boil beyond a certain brix.

Mr. Alexander: A further difference between laboratory and factory boilings is the method of heating, the laboratory pans being electrically heated so that possibly the temperature at the heating surface could be very much higher than in a factory. Have temperature measurements been made for different parts of a laboratory pan, as I believe was done in a factory pan at Illovo?

Mr. Warne: Work is being done at present on measuring temperatures in the laboratory pan but data are not yet available.

Dr. Douwes Dekker: The pan was designed so that the ratio of heating surface to pan volume would be more or less the same as a factory pan.

Mr. Bruijn: Not only the heating surface but also the amount of heat applied has been kept proportional to the contents.

Mr. Fourmond: Last year Mr. Wiehe carried out comparative tests at Amatikulu between fast and slow boilings. There was no difference in the filterability of the sugars produced. There was, however, better exhaustion with slow boiling.

Mr. Chiazzari: The idea might be worth considering of having a pilot pan — of say 100 cubic feet capacity.

Dr. Douwes Dekker: Before doing this it is necessary to find out the difference between a laboratory boiling and a boiling in a modern factory pan which has been fully equipped with instruments. The aim must be to have a constant boiling technique, which we do not have in factories at present.