

BRIX DETERMINATION

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One of the most important single parameters used in the control of the process department of a raw sugar factory or a sugar refinery is purity. For a solution, purity is defined as the percentage ratio of sucrose to soluble solids (brix) in that solution. Because speed in the analyses is essential if the results are to be used for process control the past practice in South African sugar factories was to determine pol instead of sucrose while brix was determined indirectly using a hydrometer. The disadvantage of these analyses is that appreciable errors can obtain and the errors are usually greater the lower the purity of the product analysed, e.g. for final molasses it is not uncommon to find that the brix result obtained using the hydrometer is ten units too high.

The presently accepted method⁴ for determining the "true" brix of a solution is to dry a sample under vacuum at 60-65°C using an extender such as sand, for approximately sixteen hours. This method takes far too long to be of practical value in process control but it does permit an evaluation of the correctness of the results obtained by other techniques.

Some years ago reliable sugar refractometers became commercially available and it was generally accepted that a more correct analysis for brix in sugar liquors could be obtained with the refractometer than with the hydrometer. Although the refractometer has been adopted for brix determination in some other countries its application in the South African sugar industry has been rather limited. This was possibly an indirect result of the present cane payment system which requires that the first expressed, last expressed and mixed juice be analysed using the hydrometer. Since the hydrometer was used on these juices it was probably felt that it might as well also be used for other products. However, during the last few seasons a number of South African sugar factories have used refractometer brix measurements for control purposes and it seems likely that this practice will become more widely adopted in the future.

Preliminary studies made on the juice extract† produced in the direct analysis of cane had shown that insoluble matter affected the refractometer reading giving rise to a biased error. Two techniques were found suitable for removing such insoluble matter viz. centrifuging at 30,000 g for 20 minutes or filtration through filter paper with an average pore diameter of 1 micron (see Appendix). A number of measurements have been made on different sugar liquors to determine the magnitude of the effect of the insoluble matter present on the refractometer brix as well as the correctness of these brix data. The correct brix was taken to be equal to the solids by drying result obtained on the filtered

samples. Hydrometer brix readings obtained on some filtered as well as unfiltered juices have been included with the refractometer data which have been presented below. The analyses on cane extracts and first expressed juices were carried out in October/November, 1968 on Mount Edgecombe samples while those on mixed and clarified juices were done on Illovo samples in January/February, 1969. In October/November a number of the first expressed juice samples at Mount Edgecombe were heavily contaminated by soil from the fields while in January/February the juices at Illovo were comparatively free of such insoluble matter.

Analyses on cane extracts¹

From 47 sets of determinations it was found that the solids by drying results were always lower than the refractometer brix results on filtered samples. The averages for the 47 sets of data were as follows:

Solids by drying : 5.419%
 Refractometer brix : 5.558°

The standard deviation between the two analyses was 0.060.

The effect of the insoluble matter on the refractometer brix reading was obtained from 102 sets of comparative measurements.

Refractometer brix filtered extracts : 5.656°.
 Refractometer brix unfiltered extracts : 5.833°.

From an inspection of the individual pairs of corresponding data it was apparent that the brix obtained on the filtered extract was, with only one exception, always lower than that obtained on the original extract. The differences ranged from 0.03 to 0.55 with an average difference of 0.177 and a standard deviation from the mean of 0.071. The exceptional result is not considered to be highly significant and probably resulted from an error in the reading of the unfiltered sample, which was sometimes difficult to read accurately. The effect of the above noted difference on important parameters which are calculated using the brix of extract was as follows:

(a) Fibre % cane

When the brix of filtered extract was used to calculate fibre % cane the latter was higher than when calculated using brix of unfiltered extract by, on average, 0.538 units.

(b) Pol % cane

When the brix of filtered extract was used to calculate pol % cane the latter was lower than when calculated using brix of unfiltered extract by, on average, 0.023 units.

(c) Purity of extract

When the brix of filtered extract was used to calculate the purity of extract (which is equivalent to the purity of absolute juice) the latter was higher than when calculated using brix of unfiltered extract by, on average, 2.67 units.

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†The method of cane extraction was described by Buchanan S.A.S.J. 1966, 50, 1049.

TABLE 1

No. of sets of data averaged	Ref. Brix unfiltered samples	Ref. Brix filtered samples	Hydrometer Brix unfiltered samples	Purity from Ref. Brix on unfiltered samples	Purity from Ref. Brix on filtered samples	Purity from Hydrometer Brix
97	19.85	19.60	20.35	86.56	87.72	84.34

TABLE 2

No. of sets of data averaged	Ref. Brix unfiltered samples	Ref. Brix filtered samples	Hydrometer Brix unfiltered samples	Purity from Ref. Brix on unfiltered samples	Purity from Ref. Brix on filtered samples	Purity from Hydrometer Brix
89	19.87	19.62	20.14	86.32	87.52	85.06

Analyses on first expressed juice samples

Refractometer brix measurements were made on both filtered and unfiltered samples of first expressed juice while the hydrometer brix was measured on the unfiltered samples as is the accepted practice with the present cane payment system. The averages of 97 sets of data together with the corresponding purities were as set out in Table 1.

Inspection of the individual sets of data showed that the refractometer brix on filtered juice samples was lower than that on unfiltered samples with only two exceptions, the average difference being 0.25° brix, with a standard deviation of 0.136. The hydrometer brix was higher than the filtered mixed juice refractometer brix in all but one case, with an average difference of 0.75° brix, and a standard deviation of 0.847. The hydrometer brix was so much higher than that of the "filtered" refractometer brix on eight samples that the resulting purity difference exceeded 10 units. These very large differences probably obtained by virtue of an abnormally high insoluble matter (soil) content of these first expressed juice samples. If the data of these "abnormal" samples is omitted from the averages the compara-

tive figures from the remaining 89 sets of data are as set out in Table 2.

The overall pattern was similar to that of Table 1, indicating the general validity of the relations shown between these parameters.

Analyses of mixed juice

Twelve sets of data were obtained, the averages of which were as set out in Table 3.

It was surprising to find that the hydrometer brix on the filtered mixed juice gave the best indirect measure of the brix but standard deviation figures showed the refractometer brix on the filtered juice to be the more consistent figure. The difference between the refractometer brix of the filtered and unfiltered mixed juice was 0.03° which was unexpectedly small.

Analyses of clarified juice

The averages found for 16 sets of data are as shown in Table 4.

The refractometer and hydrometer brixes on the filtered clarified juice were almost identical and not appreciably lower than the corresponding data for the unfiltered clarified juice.

TABLE 3

	Dry Solids	REFRACTOMETER RESULTS						HYDROMETER RESULTS					
		Filtered (a)	Filtered (b)	(b-a)	Std. Dev.	Unfiltered (c)	(c-a)	Std. Dev.	Filtered (d)	(d-a)	Std. Dev.	Unfiltered (e)	(e-a)
Brix	12.43	12.72	0.29	0.12	12.75	0.32	0.13	12.67	0.24	0.24	12.92	0.49	0.20
Purity	83.27	81.37	-1.90		81.18	-2.09		81.69	-1.58		80.11	-3.16	

TABLE 4

	Dry Solids	REFRACTOMETER RESULTS						HYDROMETER RESULTS					
		Filtered (a)	Filtered (b)	(b-a)	Std. Dev.	Unfilt. (c)	(c-a)	Std. Dev.	Filtered (d)	(d-a)	Std. Dev.	Unfilt. (e)	(e-a)
Brix	13.19	13.47	0.28	0.11	13.49	0.30	0.11	13.48	0.29	0.13	13.52	0.33	0.13
Purity	85.22	83.44	-1.78		83.32	-1.90		83.38	-1.84		83.14	-2.08	

Discussion

The effect of insoluble matter on refractometer brix has been reported on previously by Clayton^{2, 3} and by Rhodes⁵, both of whom also found that the insoluble matter elevated this brix reading. Comparison of the dry solids and refractometer brix of filtered (or centrifuged) cane extract showed that the latter method gave the most accurate indirect measure of the brix. In view of the magnitude of the resultant influence on the fibre % cane and purity of the extract calculated using the brix reading it is clearly advisable to filter the extract prior to reading the brix. Unfortunately no dry solids data were obtained on the first expressed juice samples. However it seems reasonable to assume that had these analyses been carried out the average result would have been a little lower than the refractometer brix on filtered juice. It is noted that the hydrometer brix on unfiltered first expressed juice was appreciably higher than either of the refractometer brix readings. This pattern still obtained when the data relating to the samples which contained abnormally high insoluble matter content were omitted from the average.

The averages of the rather small number of sets of data obtained on mixed juice showed the hydrometer brix of filtered juice to be the closest of the results of the indirect measurements to the true brix while the hydrometer brix on unfiltered mixed juice showed the biggest error. Nevertheless, bearing in mind the relatively small number of sets of data available and the standard deviations shown in Table 3, it is quite possible that a slightly different picture would emerge with more results. The data on clarified juice showed the hydrometer and refractometer readings to be in close agreement while filtration of clarified juice had a very small effect on the brix readings.

The conclusion which is drawn from these data is that with these relatively high purity juices the factor which introduces the major error into the hydrometer brix readings is the insoluble matter present in the juice. The effect was at a maximum with the dirty first expressed juices at Mount Edgecombe and of much less importance for the relatively clean mixed juices at Illovo. This is in agreement with the findings reported by Clayton. It is therefore apparent that no standard correction can be applied to correct for the effect of insoluble matter on the brix reading.

Filtration of sufficient juice for measuring the brix with a hydrometer presents difficulties which would render such a procedure impracticable in routine factory control laboratories. On the other hand it is relatively easy to filter products such as mixed juice under gravity when only a few cc are required for a refractometer brix reading especially if some filter aid is added. It is imperative to reject sufficient of the first runnings from the filter before collecting the sample. Because of the possibility of introducing errors by filtering juice, high speed centrifuging in closed tubes should be preferred.

There seems to be no doubt that more correct data for factory control would be obtained by using refractometer brix measurements of filtered solutions instead of the hydrometer brix of unfiltered samples. Data obtained previously⁶ showed that the benefit of using the refractometer for brix measurements on final molasses would be much greater than was found with the higher purity products reported above. It is hoped to continue this work to include intermediate products in sugar manufacture as well as final molasses.

While the introduction of refractometers for measuring brix would be expected to give a more accurate picture of what occurs in practice in a sugar factory it is pertinent to draw attention to one of the phenomena which became apparent when all the Hulett's group raw sugar factories changed to refractometer brix control. In this instance it should be noted that the refractometer brix readings were made on unfiltered liquors.

When hydrometers were used for all brix analyses the percentage of non-sucrose recovered in sugar and final molasses was approximately 82% of that obtaining in the mixed juice but after the changeover to the use of refractometers only about 72% of the non-sucrose in mixed juice was accounted for in the sugar and final molasses. The reason for these apparently worse factory balance figures lies in the way in which the percentage of non-sucrose in the mixed juice was calculated in factories using the refractometer. While the brix of the molasses was determined with the refractometer, the mixed juice brix was obtained with the spindle. Hence the non-sucrose recovery ratio expressed by these factories was in reality based on the following expression:

$$\text{Non-sucrose Recovery Ratio} = \frac{\text{Non-sucrose in molasses} + \text{Non-sucrose in sugar (based on refractometer brix)}}{\text{Non-sucrose in mixed juice (based on spindle brix)}}$$

It is clear that the numerator of this fraction will be depressed relative to the figure obtained using spindle brix, resulting in an apparently lower non-sucrose recovery.

In two worked examples from factory data by taking the refractometer brix of the mixed juice to be equal to the spindle brix minus 0.17, the non-sucrose recovery ratios based on the refractometer brix throughout were found to be in close agreement with the non-sucrose recovery ratios based on the spindle brix figures. It is of interest to note that with the same two sets of factory data, a non-sucrose recovery ratio based on the non-sucrose in the clarified juice was calculated, and a difference between the spindle and refractometer brixes resulted in a difference in the percentage non-sucrose recovered of 3% in both cases. Attention is drawn to these aspects in order to emphasize the dangers which are involved in making comparisons between results based on refractometer and spindle brix figures. Until factories are in a position to work entirely according to refractometer brix figures various discrepancies will exist, and it is essential to be aware of them.

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APPENDIX

Analytical techniques

Centrifuging of samples for refractometer brix measurement was carried out at 30,000 g for twenty minutes in closed centrifuge tubes. The samples were cooled in the tubes to ambient temperature before being analysed.

Filtration of samples for refractometer brix measurement was through No. 42 Whatman filter papers supported in stemless funnels covered with watch-glasses to minimise evaporation. The funnels were seated in the necks of small beakers. The first runnings were rejected in order to eliminate the effect of concentration changes on filtering as well as the possible passage of fine insoluble matter initially.

Refractometer brix was determined using a Bausch & Lomb precision refractometer. All measurements were made at 28°C.

The per cent. solids by drying was obtained by drying a known weight of sample at 65°C for 18 hours under ca. 27 in. vacuum in a stream of pre-dried air. Acid washed sand was used as extender for the samples.

Hydrometer brix was measured using hydrometers

which were calibrated beforehand using solutions of known sp gr.

Vacuum filtration of samples for hydrometer brix measurement was through a cotton wool plug which supported a layer of kieselguhr. Kieselguhr was also added and mixed with the sample prior to filtration at the rate of about 50 grams per litre. The filtrate was cooled through a copper coil immersed in ice in order to minimise evaporation losses. A correction for evaporation loss was made from the difference in pol analyses of the filtered and unfiltered samples.

Discussion

Mr. Carter: On page 3, in connection with non-sucrose recoveries, it is recorded that the percentages were 82 for the hydrometer and 72 for the refractometer. But surely this difference is to be expected?

Dr. Graham: We mentioned this to draw attention to the mistake of making comparisons when the measurements have been made in different ways.

Mr. Jennings: Dr. Graham has said it is difficult to correct either of these brix determinations according to the amount of undissolved solids in mixed juice. Has the amount of undissolved solids been determined?

Dr. Graham: This has not been done but I do not think the quantity is significant. I think the nature of the matter is more important, particularly its state of subdivision.

Mr. King: At Mount Edgecombe filter aid was used on all samples and I think this would be essential on a routine basis to speed up the operation.

Mr. Oosthuizen: What are the theoretical causes of these refractive index differences? Do the solids cause a change in the wavelength of the light?

Dr. Graham: I do not know. We used to think that suspended matter did not affect the brix reading.

Rhodes in Hawaii carried out tests and found that the nature of the impurities did not seem important but that particle size did. He used powdered silica for his tests.