A MODIFIED METHOD FOR DETERMINING FILTERABILITY

by R. P. JENNINGS

Introduction

During the 1963-64 season two different tests were used within the Natal Sugar Industry to determine the filterability of raw sugar. These tests were:

1. That using the bomb filtration apparatus of the Johns Manville Corporation,

   and

2. That developed by the Colonial Sugar Refining Company of Sydney.

Comparison of the results obtained by these two methods on affined Natal raw sugars indicated appreciable differences on occasions, while correlation of results with the actual behaviour of the sugars in a carbonatation refinery proved difficult.

The Modified C.S.R. Test

It was decided to develop a filterability test incorporating the best features of the two existing tests, and to compare results obtained by the new method with those obtained by the bomb method and the C.S.R. test at 20° C. To overcome sampling problems it was decided that results should be grouped on a monthly basis. Details of the modified C.S.R. method appear in the appendix.

The main advantages of the new method appear to be:—

(*a*) The test is conducted at approximately factory working temperature.

(*b*) The time taken is shorter than for the bomb test, about the same as for the C.S.R. method at 20° C.

(*c*) The quantity of sample used is less than for the bomb test.

(*d*) The apparatus lends itself to the direct determination of the filtration rate of refinery liquors.

The chief disadvantage, compared with the bomb method, is the absence of stirring while the test is in progress. Since the duration of the test is far shorter than for the bomb method this shortcoming is proportionately less serious.

The necessity for maintaining the apparatus at 80° C, which might seem a disadvantage when compared with the standard C.S.R. method, is easily overcome by the use of an efficient constant temperature circulating water bath and by ensuring that the connecting pipes and the apparatus itself are well lagged. It has been our experience that it is easier to maintain temperature with the modified apparatus than with the C.S.R. apparatus at 20° C.

Results obtained with the new Method:

Table I and the attached graph illustrate the monthly variations in the filterability of affined melt sugar during the period August 1964 to December 1965. Results are given for analyses by each of the three methods.

The usefulness of a filterability test for raw sugar depends upon the accuracy with which the test may be used to predict the actual behaviour in the refinery of liquor produced from that sugar.

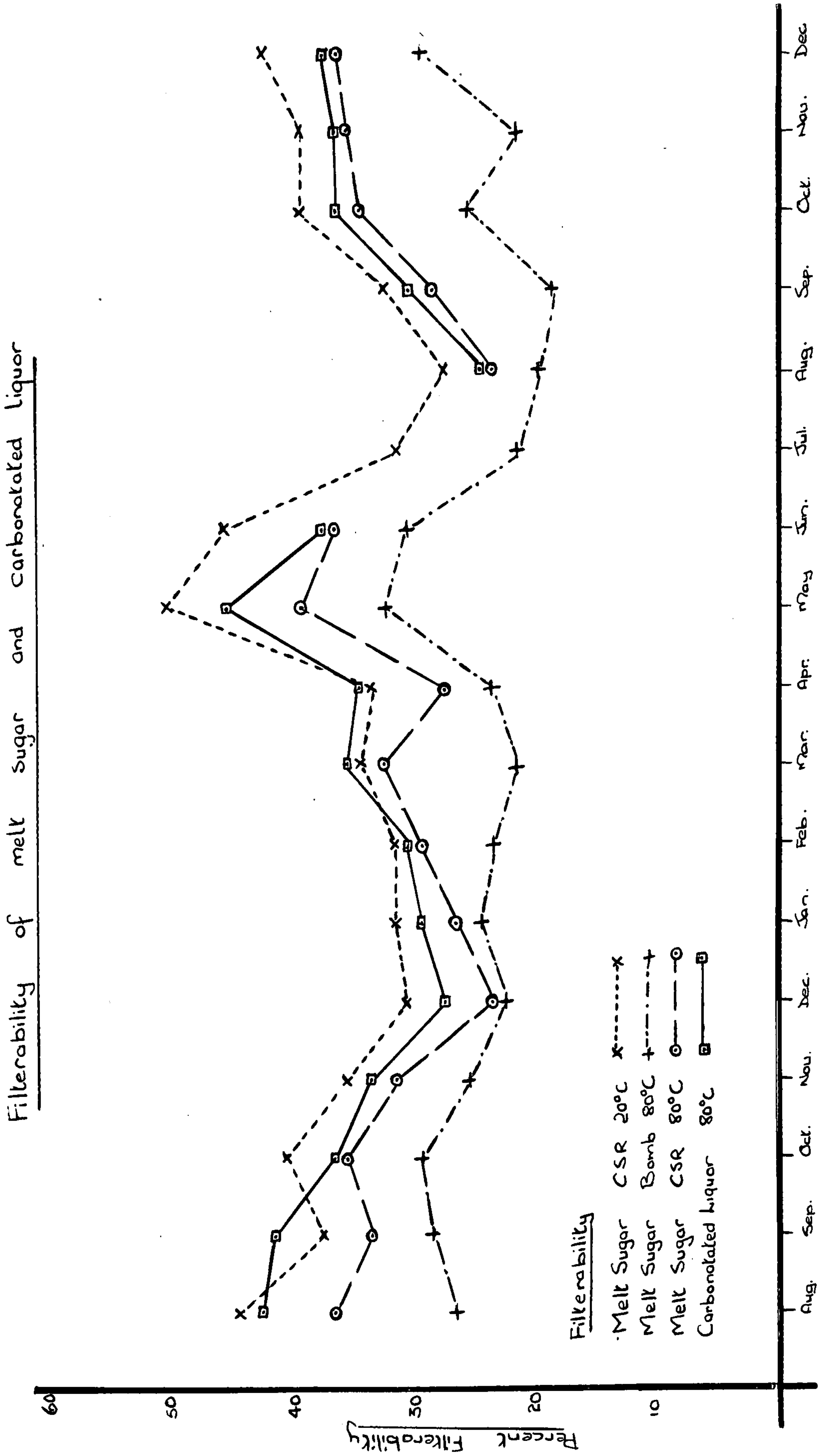
Attempts were made during the 1963-64 season to find an expression for the ease of filtration of sugar liquors under actual factory conditions in a carbonation refinery. Factors considered were, inter alia, tons of solids filtered in unit time, number of filter cycles per unit time, quantity of filter aid used and the length of life of the filter cloth. When all these factors were taken into account it was found impossible to derive a usable expression for "Refinery Filter Performance".

Approaching the problem from a different angle in the 1964-65 season it was decided to determine daily the rate of filtration of factory carbonated liquors through the modified C.S.R. apparatus. No filter aid was used for the determination. Research showed that a comparison of the flow rate of the liquor at natural brix with the flow rate of sucrose at the same brix yielded a figure which differed very little from the "filterability" figure obtained by comparison at 60° brix. It was thus decided that there should be no dilution of the carbonated liquor before testing. The figure for the filterability of carbonated liquor in Table I and in the graph is thus the comparison of flow rates of the liquor and of a sucrose solution at the natural brix of the liquor.

Table I

Filterability of Melt Sugar and Carbonated Liquor

	Melt Sugar			Carb. Liquor
Month	C.S.R. 20° C.	Bomb 80° C.	Modified C.S.R. 80° C.	Modified C.S.R. 80° C.
Aug., 1964	45	27	37	43
Sept., 1964	38	29	34	42
Oct., 1964	41	30	36	37
Nov., 1964	36	26	32	34
Dec., 1964	31	23	24	28
Jan., 1965	32	25	27	30
Feb., 1965	32	24	30	31
Mar., 1965	35	22	33	36
April, 1965	34	24	28	35
May, 1965	51	33	40	46
June, 1965	46	31	37	38
July, 1965	32	22	—	—
Aug., 1965	28	20	24	25
Sept., 1965	33	19	29	31
Oct., 1965	40	26	35	37
Nov., 1965	40	22	36	37
Dec., 1965	43	30	37	38



Filterability of melt sugar and Carbonatated Liquor
Filterability
Melt Sugar CSR 20°C
Melt Sugar Bomb 80°C
Melt Sugar CSR 80°C
Carbonatated Liquor 80°C
Percent Filterability
0
10
20
30
40
50
60
Aug.
Sep.
Oct.
Nov.
Dec.
Jan.
Feb.
Mar.
Apr.
May
Jun.
Jul.
Aug.
Sep.
Oct.
Nov.
Dec.
1964
1965

Note: Due to a fault in the pressure system of the modified CSR apparatus, filterability figures for melt sugar and carbonated liquor for July 1965 were not determined.

Certain factors have to be taken into account when attempting a correlation betweeen the filterability of melt sugar and the corresponding carbonated liquor:

1. The melt sugar tested was a composited sample made up of 90 per cent affined raw sugar and 10 per cent recovery sugars. This represents an approximation of the actual ratio of recovery sugars to total melt in practice but may vary considerably, especially when low pol sugars are being processed.
2. Melt sugars were composited on a daily basis sub-sampled, and composited as a monthly sample on which the filterability was determined.
3. The carbonated liquor tested was a composite of catch samples taken hourly over 24 hours. Daily figures were averaged arithmetically to give the monthly filterability of the liquor.
4. The compositions of neither the carbonated liquor nor the melt sugar samples were dependent upon tons of sugar melted. Errors may have occured through accepting arithmetic instead of weighted averages.
5. The comparisons have been made at a time when the refinery has been processing a very "mixed bag" of raw sugars. Table II shows the percentages of Natal and Imported raws for each month of the comprison.

Table II

Month	Natal Raws %	Imported Raws %
August, 1964	54	46
September, 1964	65	35
October, 1964	80	20
November, 1964 to April 1965	100	0
May, 1965	34	66
June, 1965	55	45
July, 1965	64	36
August, 1965	65	35
September, 1965	62	38
October, 1965	89	11
November to December, 1965	100	0

6. During April the refinery reprocessed a considerable quantity of contaminated refined sugar.

7. The factory carbonation process is by no means standard, and the amount of "gassing" is varied as the quality of the incoming sugar dictates.

With these factors considered there appears to be definite correlation between the filterability of melt sugar determined using the C.S.R. apparatus — both at 20° C and 80° C and the filterability of carbonated liquor derived from that sugar. The correlation is closest between October 1964 and March 1965, when the refinery was processing raws obtained almost exclusively from Natal. The anomalous result obtained in September 1964 occurred during a time when the refinery was processing sugars from San Domingo and Indonesia. The latter sugars, though yielding a modest laboratory filterability figure ($\pm$ 50 per cent, C.S.R. 20° C), behaved in practice like sugars of far better filtering characteristics. It would appear that, for this sugar, the filterability tests used for the affined sample were unable to predict factory performances with any degree of accuracy, while tests on the carbonated liquor showed the situation in a truer light.

There is a fair correlation between the filterability results obtained on the melt sugar by the three different test methods. The correlation between the two sets of results obtained with the C.S.R. apparatus is better on the whole than correlation with figures obtained using the bomb method. There is little to choose between the C.S.R. method and modified C.S.R. method as far as correlation with carbonated liquor filterability is concerned. While both appear to predict carbonated liquor filterability more accurately than the bomb test, this may result from the method used for determining carbonated liquor filterability. Had the bomb apparatus been used the situation might well have been reversed.

Summary and Conclusions

The C.S.R. method at 20° C, and the modification of the C.S.R. method at 80° C may be used to indicate trends in the performance of the refinery filter station. The modified apparatus may be used to determine the filtration rate of carbonated liquors. It would appear that correlation of data between laboratory filterability and factory filter performance is closest when sugars of one origin are being refined, though this may well be due to changes in the carbonation process in the factory.

Acknowledgments

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References

Alexander, J. B. and Graham, W. S., 1964, unpublished Report.

Archibald, R. D., Variation of Filterability with Brix, Proc. S.A.S.T.A., Vol. 30, 1965, p. 50-55.

APPENDIX I

DETAILS OF THE MODIFIED COLONIAL SUGAR REFINING METHOD FOR THE DETERMINATION OF RAW SUGAR FILTERABILITY

Preparation of Sample

As with the Bomb and C.S.R. methods, the raw sugar sample is affinated prior to testing, according to the following technique.

1,000 ml saturated aqueous refined sugar solution at room temperature are added to 1,200 g. of the sugar to be analysed in a one gallon Mason jar, the lid of which is provided with a rubber gasket. Thorough mingling is effected by rotating at 30 r.p.m. for 30 minutes after which the magma is centrifuged in a laboratory centrifugal. Washing of the sugar in the centrifuge is carried out using 50 ml. cold water delivered in a fine jet from a specially constructed wash bottle. Centrifuge for six minutes at 3,000 r.p.m. in an eight inch diameter basket. The sugar is then spread out in a thin layer on a sheet of paper to air dry before proceeding with the analysis.

Reagents

Standard Buffer solution which should be prepared as follows:

1. Prepare about one litre of 50 per cent W/W glycerol made from B.P. Glycerol.

2. Take two beakers:
   (*a*) Dissolve 15 g. A.R. calcium acetate in sufficient of the 50 per cent glycerol solution, with heating if necessary.

   (*b*) Dissolve 400g. triethanolamine in sufficient of the 50 per cent glycerol solution. The triethanolamine should be commercial water white or pale yellow grade.

3. Transfer the contents of the two beakers to a clean, dry litre flask, rinsing the beakers thoroughly with the 50 per cent glycerol solution. Cool, if necessary.

4. Make up to one litre with the 50 per cent glycerol solution, mix well, and allow to stand overnight. Add a little supercel, and filter in the test filter or Buchner funnel. Store in a stoppered, clear glass bottle which has been cleaned, and rinsed with some of the filtered solution.

Filter Aid

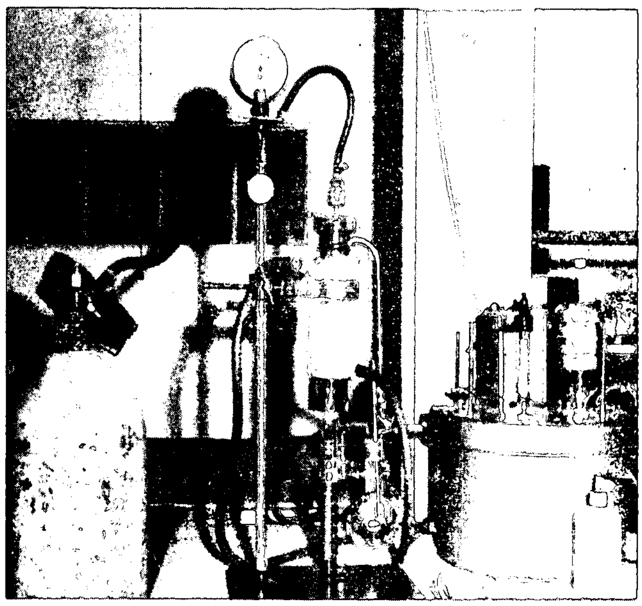
Laboratory Standard Filter Cel supplied by the Johns Manville Corporation, California, U.S.A.

Filter Septa

Filter cloth conforming to the South African Bureau of Standards Specification 512-1956 Type D157, the essentials of which are:

Weave	:	Satin 4/1.
Weight per sq. yd.	:	10 ozs.
No. of Threads,		
Warp	:	99
Weft	:	58
Tensile Strength		
Strip Method		
Warp	:	275
Weft	:	180
Weight of filling %	:	5

The fabric composition is exclusively cotton. The cloth is cut into circles 5.5 cm. in diameter.



Apparatus (See Photograph)

The apparatus is a modification of that developed by the Colonial Sugar Refining Company of Sydney, details of which may be found in I.C.U.M.S.A. Proceedings, 12th Session (Subject 10), Washington 1958.

The filtration apparatus consists essentially of a cylindrical brass tube 1.7/8 inches in diameter by 9.7/8 inches long. The filter cloth is supported by a filter disc which is firmly held in position in the base end of the tube. The top of the tube is closed by a screw on lid. Pressure may be applied to the contents of the tube through an air line passing through the lid.

As much of the length of the tube as possible (not less than 7½ inches) is enclosed by a metal jacket through which water at 80° C is circulated from a constant temperature water bath. The apparatus and the pipes connecting apparatus and water bath are lagged with asbestos. The apparatus is attached to a rigid stand by means of a clamp, designed in such a way that the apparatus may be tipped for cleaning purposes without complete removal from the stand.

To obtain values for the filtration rate of sucrose it is necessary to connect together, end to end, two jacketed tubes, attaching the filter disc to the base of the lower tube. The tubes are easily connected by means of a short brass connecting piece giving a pressure tight seal on both tubes.

Procedure

300 g affinated sugar are dissolved in 200 g water and the pH of the solution adjusted to 9.0 using standard buffer solution ($\pm$ 4 ml.). The beaker is covered with a watch glass and heated to just over 80° C in a water bath. Maintaining the solution at a little over 80° C an amount of Standard Filter Cell equivalent to 0.35 per cent on brix (1.050 g for 300 g sugar) is added to the beaker and dispersed in the solution by stirring for 30 seconds with a mechanical stirrer. The solution is transferred to the previously assembled apparatus where the test is carried out at 80° C. The lid of the apparatus is screwed on after checking the temperature of the solution.

Air pressure of 50 p.s.i.g. is built up as quickly as possible in the apparatus and a stop watch started simultaneously with the application of pressure. Filtrate collected during the first two minutes of the filtration is discarded. The remainder of the filtrate is collected in a graduated cylinder, noting the volumes in the cylinder at the end of the sixth, seventh and eighth minutes from the commencement of filtration. The pressure is maintained at 50 p.s.i.g. for the duration of the test. The following table illustrates the method of calculation:

Time from Start of Test (Min)	ml. Filt.	Vol. pure Sucrose Sln. filt. under same conditions	Filterability %	
6	81	200	$\frac{81 \times 100}{200}$	40.5
7	96	240	$\frac{96 \times 100}{240}$	40.0
8	108	275	$\frac{108 \times 100}{275}$	39.3
			Average	40%

With the fast filtering sugars it was found more practical to use filter discs of $1\frac{1}{2}$ inches to an inch in diameter in place of the $1\frac{3}{4}$ inch I.D. disc used in the C.S.R. method. In each case the volume filtered is compared with volume of a sucrose solution filtered under the same conditions, using the same sizes disc.

Note

1. With fast filtering sugars, especially refined sugar, it is necessary to double the quantities of solution used.
2. Care must be taken to avoid a substantial increase in the brix of the solution due to evaporation. It may prove necessary to reduce the original brix, before heating, to 59° to allow for evaporation.
3. Periodic checks should be made on the apparatus using refined sugar solutions. The filtration rate should be such that the volumes of filtrate collected should differ, on the average, by not more than 10 ml. from those shown in columns 3 of the example given above. (This is assuming that the highest quality refined sugar is used in every check.)

APPENDIX 2

THE FACTORY FILTER PERFORMANCE OF RAW SUGARS

Introduction

Provided that the many variables in the refining process prior to filtration are kept constant, the filter performance of a sugar may be gauged by the number of tons of solids handled by unit filter:

Filter Performance =

$$\frac{\text{Tons solids filtered in unit time}}{\text{Number of filters started in unit time.}}$$

For comparison purposes, allowing for a constant ratio of re-melted recovery sugars to total melt, the equation becomes:

Filter Performance =

$$\frac{\text{Tons sugar melted in unit time}}{\text{Number of filters started in unit time.}}$$

Practical Considerations

During the tests on filter performance of various sugars at the refinery, the fullest co-operation of the Process department was obtained to ensure the following:

1. Sugar from one source only was processed during the period of the test.
2. A constant melt rate of affinated sugar.
3. A constant ratio of recovery sugars to total melt.
4. A constant supply of liquor to the Carbonatation station.
5. No variation in the procedure of carbonatation. (Quantity of lime added, gassing rate, composition of gas, pH of liquor at various stages.)
6. A standard technique in the filter station using cloths of approximately the same age for each test.

Samples of affinated sugar, carbonatation supply liquor and carbonatated liquor were taken at regular intervals and composited to give a sample representative of the period of the test. The filterability of each sample and the gum and starch content of the affinated sugar were determined.

Results

Test Number	1	2	3	4	5
Raw sugar source	DL	EM	FX	UK	UK
Tons processed	138	268	302	480	420
Hours of test	2	4	4	6¾	5½
Tons per hour	69	67	75	71	76
Pounds filter aid added	200	Nil	Nil	Nil	Nil
No. of filters used	14	8	8	4	4
Tons melt per filter	9.9	33.5	37.8	120.0	105.0
Affinated Sugar					
Filt. % C.S.R. 20° C.	30	39	41	36	40
Filt. % C.S.R. 80° C.	22	35	39	35	39
ppm. Gums	1,800	1,500	1,400	1,100	1,000
ppm. Starch	430	580	470	90	40
Carb. supply liquor Filt. % C.S.R. 80° C.	7	30	37	39	39
Carb. liquor Filt. % C.S.R. 80° C.	14	38	36	61	51

Note:

(1) Tons melt per filter is based on tons of test sugar melted and does not take into account re-melt sugar.

(2) The test run with Darnall sugar was curtailed because of the poor filtration of the carbonatated liquor. Despite the use of filter aid, the flow of liquor to the Carbonatation station had to be reduced, upsetting the controls of the test.

Discussion

Though the accumulation of data concerning the performance of raw sugars in the filter station of the refinery is still at a very early stage, some very interesting features have already come to light.

There appears to be a decided absence of correlation between affinated sugar filterability and "filter performance". This is most marked for Umzimkulu sugar.

Though the laboratory filterability of the sugars from Umzimkulu indicated that its performance in the refinery would be about equal to that of the Empangeni and Felixton sugars (test 2 and 3), in practice the performance was far superior. The figures of 120 and 105 tons per unit filter are three times the values for the earlier tests.

At the other end of the scale, laboratory figures were unable to predict the very poor filtration of the Darnall sugar in test one.

It would appear that more significance can be attached to the filterability of carbonatated liquor. The carbonatated liquor filterability figures of tests four and five are both well above average, while Darnall sugar tested produced a carbonatated liquor filtering only 14 per cent.

It seems certain that the excellent filtering characteristics of the sugar from Umzimkulu are connected with the below average gum content and the very low starch content of this sugar. Less easy to explain is the relatively low filterability figure obtained for this sugar in the laboratory. A parallel situation already mentioned in the earlier part of this paper. was noted for sugar from Indonesia. It is interesting to note that these sugars also contained very little starch. These results tend to indicate that the laboratory filtration test cannot be used with any confidence to compare the potential filter performance of sugars of high and low starch content. With such sugars it would be better to follow the lead of at least one overseas refinery and predict filter performance on the strength of starch content alone.

Investigations into the factory filter performance of raw sugars will be continued next season. It is hoped that, when a lot more data has been accumulated, the upper and lower confidence limits of the present laboratory filterability tests may be determined.

Mr. Carter: Mr. Jennings mentioned that "low starch" sugars filtered better in practice than seemed likely from laboratory results. Because of the nature of the Rabe process is it not possible that the explanation lies in the poor removal of other impurities, e.g. phosphate, during clarification?

Mr. Jennings: I think it more likely that the reason for the discrepancy lies in the mechanism of the laboratory filterability test, which employs an added filter aid, compared with factory carbonatation using a produced calcium carbonate as filter aid. It would appear likely that starch has considerable influence on the formation of the calcium carbonate precipitate.

Mr. Carter: We should then direct our research towards a laboratory test incorporating carbonatation.

Dr. Graham: It seems apparent that the filterability tests used at present are misleading, especially for sugars low in starch. I agree that the development of a new test incorporating laboratory carbonatation is important. Work started in this direction at the S.M.R.I. was discontinued even though a carbonatation tank was built. It is especially important to develop this test now since the possibility of the establishment of a bonus system for fast filtering sugars is being considered by the South African Sugar Millers. Using present tests Umzimkulu sugar would be penalised under such a scheme.

Research towards improvement in filterability has centred on establishing what impurities impede filtration and the removal of these impurities. It has been established that starch is a major contributor to poor filtration and various starch removal methods have been tried. The procedure devised at Umzimkulu is cheap to operate, requires little capital equipment and has been operating successfully for almost a complete season. It is my opinion that the sugars produced by this method are of such a high quality that, were the process used throughout Natal, the filterability problem would cease to exist.

Dr. Roux has pointed out to us that a delay in the application of new ideas can cause a major loss in the advantages to be gained. I hope his remarks will not be prophetic of this industry's attitude in respect of the solution to our filterability problem.

Mr. Alexander (in the chair): I agree wholeheartedly with all that Dr. Graham has said and would like to congratulate all associated with the experiment at Umzimkulu, and especially Mr. Rabe, on the success of the project and the excellence of the sugar produced.

Dr. Graham: Why was the figure used for filter performance in the refinery test runs, tons melt per filter and not tons melt per filter per hour?

Mr. Young: The figure which best illustrates factory filter performance is tons melt divided by the number of filters started during the time in question. In the refinery the term "filter starts" is used to indicate the interval between putting filters on stream. If a filter station processes 70 tons per hour using 10 filters each filter handles 7 tons per hour. With ½-hour starts each filter is left on stream for 5 hours and processes 35 tons. If 5 filters are on stream the result is also 35 tons per filter since though the quantity processed per filter per hour is double, the length of the filter cycle is halved.

Dr. Graham: Mr. Jennings mentioned a "below average gum content" for Umzimkulu sugar. It was the experience of the S.M.R.I. that the Rabe process removed no starch-free gums.

Mr. Jennings: It appeared from our analyses that there was some removal of starch-free gums, though nothing like the removal of starch.

Mr. Buchanan: Was any record kept in the refinery of the performance of Taiwan sugar, which has a high starch content?

Mr. Alexander: The refinery did not carry out tests on Taiwan sugar since the sugar was not processed separately but mixed with Natal raws. A recent paper reporting research in Japan disclosed a disappointing correlation between starch and laboratory filterability determined using the CSR apparatus, but a very good correlation, with a correlation coefficient of 0.95, between starch and the filterability of laboratory carbonatated liquors.

Mr. Jennings: It may appear from the paper on filterability that the laboratory tests used at present are not very meaningful. This is not the view of the refinery which considers the laboratory tests very useful for normal Natal sugar, containing average starch content of 350-500 p.p.m. on the affinated sample. For sugars of low starch content the test is not satisfactory but if any Natal mill can send sugar to the refinery consistently containing less than 200 p.p.m. starch they are welcome to throw away their filterability apparatus!

Dr. Matic: I must congratulate Mr. Jennings in particular and the refinery in general on their research and on the applicability of the data presented. I hope that other industrial laboratories will follow Hulsar's example and report results of investigations which are of general interest to the industry.