SOME NOTES ON THE DETERMINATION OF GUMS IN SUGAR PRODUCTS WITH SPECIAL REFERENCE TO THEIR DISTRIBUTION IN HULSAR PROCESS

By R. P. JENNINGS

Introduction

When it is considered that raw sugars received at Hulett's Refinery during the 1962/63 season contained over 500 tons of gums, it can be appreciated that a study of these tenacious sugar impurities is of more than academic interest.

Carbohydrates of high molecular weight precipitated from sugar solution by acidified alcohol have been classed as gums or alcogels. These gums can be sub-divided into three major groups.

The first group, consisting principally of pectins, hemicelluloses and dextrins, are present in solution in the original cane juice.

The second group, of which the best known member is starch, comprise those insoluble gums present in the original cane juice which are rendered soluble by the manufacturing process.

The last group contains substances produced by bacteria before and during the milling process. Important members of this group are dextrans and levans.

Previous Investigations

Referring to gums, Meade in the revised ninth edition of the Sugar Cane Handbook [4] states that "the concentrations of these substances in the syrups were not determined for want of suitable quantitive analytical methods."

Ruff and Withrow [5] extensively examined methods for the determination of gums in sugar products and proposed an analytical procedure which has been recommended by Browne and Zerban [3].

Ruff and Withrow suggest precipitation of the gums from 50° Brix solutions of sugar products using acid alcohol in the ratio of 1 ml. concentrated hydrochloric acid with 100 mls. of ethyl alcohol to 20 mls. of the sugar solution. The precipitating solution is added slowly with constant and thorough agitation. The precipitate is allowed to settle for 15 minutes and filtered through a Gooch crucible containing at least 0.2 gms. of dry asbestos as the mat. The precipitate is well washed with slightly acidified alcohol, dried at 105° C. for one hour, cooled, weighed, ignited for 15 minutes, cooled and re-weighed. The difference in weight is recorded as gums.

Ruff and Withrow conclude that the concentration of hydrochloric acid may be varied quite considerably but that results are very sensitive to variations in alcohol concentrations. They also found that denatured alcohol can be substituted for ethyl alcohol with suitable precautions.

Gum Determination at Hulett's Refinery

The Hulsar laboratory used a method of gum determination based on a procedure developed in Java. In this method 20 gms. of 50° Brix solution of the sugar product were treated with 150 mls. of a solution of alcohol containing 10 per cent of 50 per cent hydrochloric acid. Following precipitation the gums were allowed to stand overnight before filtering, drying, weighing, igniting and re-weighing as before.

It is of interest here to compare the relative quantities of the solutions used in the two methods:

	Ruff and Withrow	*Hulsar*
mls. 50° Brix solution . .	20	±16.25 (20 gms.)
mls. concentrated HCl. .	1	7.50
mls. water	0	7.50
mls. alcohol	100	135

It is evident that the Hulsar method employs a much higher ratio of both acid and alcohol to sugar solution.

Reasons for Present Investigations

During the course of a series of gum determinations on mill molasses it was found that the Ruff and Withrow method often led to the formation of a sticky black precipitate which was impossible to wash satisfactorily and did not yield reproducible results. When attention was turned to raw sugars it was found that the results obtained using the Ruff and Withrow method were generally higher than with the Hulsar method. For this reason it was decided to investigate the effects of variations in alcohol/acid/solution ratio, and in time of standing between precipitation and filtration.

The Effects of Acid Concentration on the Gum Precipitate

It was found that, as the acid to alcohol ratio of the precipitating solution was raised, the quantity of gums precipitated diminished. The effect was apparent even with raw sugars, where the quantity of gums was small, but more noticeable with molasses.

Twenty mls. aliquots of a 50° Brix solution of raw sugar were precipitated with 100 mls. portions of acid-alcohol solution using different acid concentrations. The results of gums precipitated are tabulated below:

mls. HCl. in 100 mls. acid alcohol	–	0.5	1.0	2.0	3.0	4.0	5.0
mgms. gums . . .	23.1	22.9	22.0	21.5	20.7	20.7	

In the case of molasses, aliquots of a 50° Brix solution of mill molasses were precipitated in duplicate, one set standing half an hour before filtering and the other twenty-four hours.

mls. acid in 150 mls. precipitating solution	0	1.0	2.5	5.0	7.5
mgms. gums half hour	*	209.4	178.7	165.6	144.8
mgms. gums 24 hours	493.4	186.9	159.1	143.5	132.8

* unworkable precipitate.

From the results of the molasses experiment it was obvious that an increase in standing time from the quarter hour recommended by Ruff and Withrow to the vague "overnight" of the Hulsar method, could materially affect the quantity of determined gums. It seemed likely that some form of hydrolysis was responsible for the drop in gums. This hydrolysis was further illustrated by the following investigations.

(*a*) Gums were precipitated from 20 gms. aliquots of a 50° Brix solution of raw sugar using 100 mls. portions of alcohol containing various amounts of acid. Standing time was varied between 1, 2 and 24 hours.

		mgs. of gums precipitated		
Standing time (hours)		1	2	24
Acid added in 100 mls. alcohol	0.5 mls.	23.6	23.3	20.4
	1.0 mls.	24.0	22.0	20.7
	5.0 mls.	22.6	22.1	20.2

(*b*) 20 gm. aliquots of a 50° Brix solution of a low pol raw sugar were precipitated using Hulsar solution (135 mls. alcohol plus 15 mls. 50 per cent HCl.). Standing time before filtration was varied.

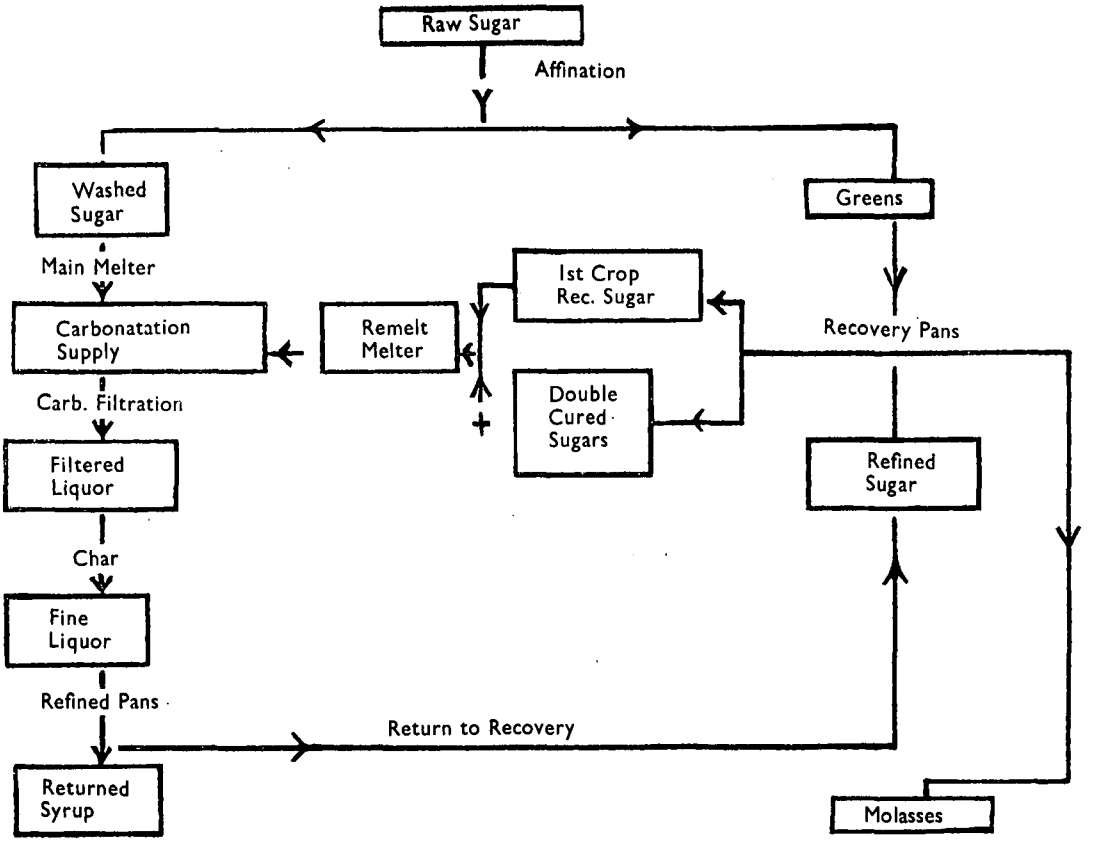
Standing time (hours)	½	1	2	3	4	21
mgms. gums	44.0	43.6	43.2	41.5	39.4	40.2

(*c*) To investigate the effect of acid on gums before precipitation, a 50° Brix solution of low pol raw sugar was treated with a volume of hydrochloric acid calculated to correspond with the Hulsar solution. The mixture was maintained at 30° C. throughout the test, 20 ml. aliquots withdrawn at intervals, and the gums precipitated with 130 mls. of alcohol. The standing time between precipitation and filtration was maintained at one hour, and the gums determined in the usual way.

Time before alcohol addition (hour)	½	1	2	3	4	5	6	24
mgms. gums	84.7	83.2	81.8	81.6	80.7	80.4	80.6	75.4

The Effects of Alcohol Concentration on the Gum Precipitate

Ruff and Withrow found that the quantity of gums precipitated from sugar solutions is very sensitive to changes in alcohol concentration.



A series of 144 gum determinations was carried out on raw sugar using a range of alcohol concentrations from 50 to 100 mls. per 20 gms. of 50° Brix sugar solutions combined with acid concentrations between 0.5 and 5.0 mls. per determination. It was found that the quantity of gums precipitated by the high alcohol concentrations was about 10 per cent higher than for the low concentrations while low acid concentrations generally gave higher results than their more acid equivalents. In some cases with low acidity and higher alcohol concentrations sugar crystals were precipitated on the sides of the beaker.

The following table shows a portion of the results of these tests:—

Alcohol concentration (mls. per determination)		50	60	70	80	90	100
		milligrams gums precipitated					
Mls. acid per determination	0.5	24	26	Sugar precipitated			
	1.0	25	26	27	27	27	28
	2.0	24	25	26	26	26	28
	3.0	22	25	24	26	27	26
	4.0	22	22	23	24	24	24
	5.0	21	22	23	23	24	24

A similar experiment, between wider limits, yielded the following results:—

mls alcohol per determination		100	150	200
		milligrams gums precipitated		
Mls. Acid per determination	0	56.0	56.1	47.6
	0.5	29.6	33.0	34.2
	1.0	29.4	30.0	32.5
	5.0	27.3	28.6	30.1

Again it is seen that, apart from the anomalous case where acid is absent, gum precipitation is highest with most alcohol, least acid.

Further support for the dependance of gum precitation upon alcohol concentration was found when dealing with a raw sugar solution before and after filtration through the Colonial Sugar Refining filtration apparatus. With these determinations the concentration of acid to alcohol was fixed on a volume/volume basis:—

	mgms. gums precipitated from 20 gms. 50° Brix solution	
	Before Filtration	*After Filtration*
50 mls. acidified alcohol	21.7	20.6
100 mls. acidified alcohol	23.7	21.7
150 mls. acidified alcohol	31.0	24.8

The Dependance of Gum Precipitation upon pH

In order to obtain a satisfactory method for determining gums throughout the refining process, the critical relationship between gum precipitation and pH was investigated.

Ruff and Withrow concluded that calcium salts of the various organic acids can be precipitated by alcohol at low acid concentrations while the free acids cannot. At higher mineral acid concentrations the organic acids are liberated and the calcium salt precipitation does not take place.

A potentiometric titration between concentrated hydrochloric acid and 10 gms. of 50° Brix solution of refinery final molasses diluted with 50 mls. distilled water yielded the following results:—

Mls. Conc. HCl.	*pH*
0	6.02
0.2	4.00
0.4	3.13
0.6	2.05
0.8	1.60

Using the results of this titration, gums were precipitated from 10 gm. aliquots of the molasses solution at calculated pH values using 50 mls. of alcohol in each case. Precipitates were allowed to settle for 16 hours and then filtered through previously tared crucibles, in order to obtain a weight of ash as well as a weight of gums in each case.

pH	*gms. Gums*	*gms. Ash*	*Gums % Solids*
6.0	1.400	0.289	28.0
4.7	1.277	0.279	25.5
4.0	1.115	0.262	22.3
3.6	0.897	0.234	17.9
3.1	0.732	0.210	14.6
2.6	0.394	0.148	7.9
2.1	0.378	0.144	7.6

With pH higher than 3.0 the precipitates obtained were black and extremely sticky. It was very difficult to transfer the precipitate from the beaker to the crucible, whilst washing the precipitate properly was impossible.

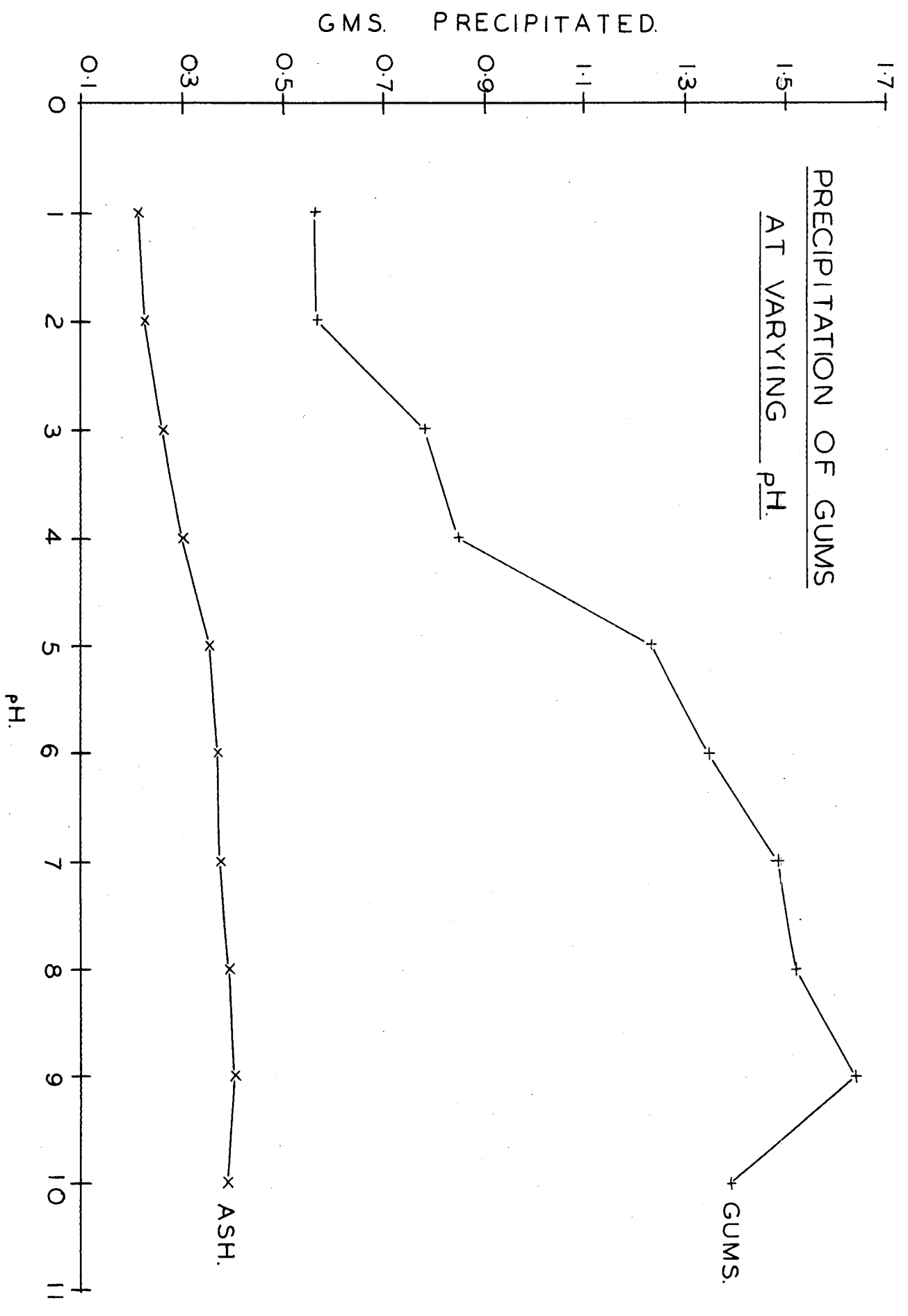
A similar experiment was carried out using 20 gms. of a 50° Brix solution of tail end syrup from refined boilings.

mls. Acid	*pH*	*mgms. Gums*	*mgms. Ash*	*Gums % Solids*
0	6.7	613.2	53.8	6.13
0.2	3.7	241.5	45.1	2.42
0.3	3.0	201.0	36.8	2.01
0.4	2.3	173.9	32.6	1.74
0.5	1.9	161.7	31.4	1.62

As with the final molasses, sticky precipitates, difficult to wash, were obtained at higher pH values.

Extending the pH range further by the addition of concentrated ammonia, gums were determined on 10 gms. of 50° Brix mill final molasses from 1.0 pH to 10.0 pH. Results of both gums and ash are shown in the following table and attached graph:—

mls. conc. HCl.	*mls. conc. NH_4OH*	*pH*	*gms. Gums*	*gms. Ash*	*Gums % Solids*
0.70	—	1.0	0.562	0.215	11.2
0.35	—	2.0	0.569	0.225	11.4
0.25	—	3.0	0.783	0.267	15.7
0.15	—	4.0	0.848	0.303	17.0
0.05	—	5.0	1.257	0.356	25.1
—	—	6.0	1.354	0.372	27.1
—	0.05	7.0	1.484	0.378	29.7
—	0.10	8.0	1.521	0.397	30.4
—	0.20	9.0	1.650	0.407	33.0
—	1.50	10.0	1.333	0.395	26.7


GMS. PRECIPITATED.
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pH.
PRECIPITATION OF GUMS
AT VARYING pH.
ASH.
GUMS.

As with the previous investigations the determinations at high pH were most difficult to handle. It was noticed that precipitates from alkaline solutions were lighter in colour than those from acid solutions.

Gums in Refinery Liquors

To obtain a meaningful gum balance throughout the refinery process it was first necessary to settle on an arbitrary method for gum determination.

The extreme sensitivity of gum precipitates to changes in alcohol concentration can be overcome to some extent by using the same concentration throughout, though it must be realised that results so obtained are purely comparative and not absolute values of the gums present in the liquors.

The pH at which precipitation takes place must be of the same order in each case and should be low enough to obtain a workable precipitate without being sufficiently low to cause too much hydrolysis of the precipitate.

In the first set of determinations 20 gms. of 50° Brix solutions were used and precipitation effected at an adjusted pH of ± 1.8 by 50 mls. of ethyl alcohol. The weight of gums precipitated was determined in the usual way.

The second series of tests differed only in that a pH of ± 1.3 was maintained throughout.

The starch contents of the various products were determined concurrently.

The schematic representation of the refining process below indicates the materials sampled for the gum and starch determination.

Materials	First Series			Second Series		
	G, Gums %	S, Starch %	G minus S %	G, Gums %	S, Starch %	G minus S %
Raw Sugar	0.259	0.091	0.168	0.272	0.093	0.179
Washed Sugar	0.179	0.078	0.101	0.222	0.084	0.138
First Crop Recovery Sugars	—	—	—	0.283	0.091	0.192
Double Cured Second and Third Crop Recovery Sugars	—	—	—	0.272	0.088	0.184
Remelt Melter	0.304	0.116	0.188	0.329	0.108	0.221
Carb. Supply	0.215	0.091	0.124	0.252	0.098	0.154
Filtered Liquor	0.173	0.053	0.120	0.195	0.053	0.142
Fine Liquor	0.184	0.052	0.132	0.187	0.054	0.133
Refined Sugar	0.075	0.028	0.047	0.080	0.030	0.050
Greens	1.323	0.243	1.080	1.366	0.276	1.090
Tail end Syrup	2.015	0.378	1.637	1.569	0.379	1.190
Molasses	5.329	1.000	4.329	5.498	1.171	4.327

All percentages are based on solids (Refractometer).

Four stages exist between raw sugar and refined sugar in which gums may be removed:

1. During affination.
2. During filtration.
3. By adsorption on char.
4. During crystallisation.

The respective removals of total gums, starch, and gums other than starch (G-S) are summarised:

Removed During	First Series			Second Series			Average		
	Total Gums	Starch	G-S	Total Gums	Starch	G-S	Total Gums	Starch	G-S
Affination	31	14	40	18	10	23	25	12	32
Filtration	20	42	3	23	46	8	22	44	6
Char	+ 6	2	+10	4	+ 2	6	+ 1	—	+ 2
Crystallisation	38	50	33	40	52	35	39	51	34

During affination only 17 per cent of the total gums removed was starch. This suggests that the "starch free" gums are more concentrated in the molasses surrounding the raw sugar crystals (and are thus more easily removed by affination) than the starch, which is thought to "co-crystallise" with sucrose [1].

85 per cent of the gums removed in the filtration stage is starch, in fact the removal of other gums is negligible. This suggestion that starch is not in as true solution as the other gums, can perhaps be accounted for by the well known tendency of starch solutions to retrograde.

It is surprising that there is no adsorption of gums by bone char during the decolourising process, especially when it is considered that vegetable carbon has been shown to remove both starch and other gums.

51 per cent of the starch in fine liquor finds its way into the refined sugar compared with 34 per cent of the "starch free" gums. This again indicates "co-

crystallisation" of starch with sucrose. The percentage of starch in total gums in refined sugar (37 per cent) is of the same order as the percentage in washed sugar (40 per cent), this despite the removal of 44 per cent of the starch in the filtration stage. Indeed, starch can be considered the major single impurity in refined sugar.

More work is being carried out on the gum balance in the refinery, especially relating to the removal of gums during filtration. Gum and starch determinations are being made on sugar solutions both before and after filtration through the Colonial Sugar Refining apparatus, and it will be interesting to compare the reduction in gums during laboratory filtration with that obtained in the factory.

Analytical Methods used

Precipitation was carried out in beakers, the alcohol being added at a controlled slow rate, with constant mechanical agitation of the solution, except in the case of the molasses determination at high pH when it was found impossible to remove all the "gummy" precipitates from the stirrer. In these cases the beakers were agitated by hand.

The precipitates were filtered through Gooch crucibles containing a mat of not less than 0.2 gms. of asbestos. Crucibles were ignited before use. Careful decantation of supernatant liquid before transferring the precipitate was found to aid filtration considerably.

The precipitates were well washed with alcohol, usually containing a little hydrochloric acid to aid filtration. Generally it was found that filtration was satisfactorily rapid.

The crucibles were dried at 105° C. for not less than 1½ hours, cooled in dessicators and weighed to 0.0001 gms. They were then ignited for not less than 20 minutes, cooled and re-weighed. Checks for constant weight were carried out. In the experiment relating gum precipitation to pH, acid and ammonia were added from a semi-micro burette.

Summary and Conclusions

It is difficult to suggest a method to obtain an absolute value for gums which will be satisfactory for both high and low purity products. With sugar solutions, and high purity liquors with low gum content, the Ruff and Withrow method may prove satisfactory, but lower grade products such as molasses need a different technique based largely on an increased quantity of acid.

It has been shown, however, that an increase in the acid content reduces the quantity of gums precipitated, probably by hydrolysis, the effect of this hydrolysis being accentuated by increasing the standing time before filtration. Doubt is thus cast as to whether the figure obtained for gums at the lower pH is absolute.

Again, the concentration of alcohol used appears to be critical. The amount of gums precipitated increases with the quantity of alcohol used, but two factors control the use of a large excess of alcohol, one being the bulk of material employed and the other the precipitation of sugar crystals from higher purity solutions with a large excess of alcohol. It is probable that further non-sugars will precipitate at higher alcohol concentration, again rendering the absolute determination of gums impossible.

It has been found that pH has a very marked effect on the quantity and nature of the alcoholic precipitate, especially with low purity solutions. Between 2.0 pH and 7.0 pH the quantity of material precipitated from a low grade product can treble. Ruff and Withrow mention that calcium salts of organic acids are precipitated at higher pH values but, although insufficient work has been done on the subject as yet, it is thought that organic calcium salts are not the only non-sugars, apart from gums, precipitated. This view is confirmed by Wells (6) working with Queensland sugars. Examination of the higher pH precipitates is proceeding.

In conclusion, it is suggested that comparative gum determinations should be made on a 50° Brix solution at some definite pH in the range 1.5 to 2.0 pH and using 100 mls. of alcohol for every 10 gms. solids in the test solution. The low pH will ensure that molasses precipitates are workable. Precipitates should be filtered after not more than one hour standing time, but filtration after less than 30 minutes is not recommended in the case of molasses solutions as the solution will not filter well. Precipitates should be very well washed with slightly acidified alcohol.

Acknowledgments

I would like to thank my colleagues the Hulsar laboratory staff, particularly J. Bourne and P. de Froberville, for their assistance in obtaining certain of the data for this report.

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Dr. Douwes Dekker (in the chair): The papers which have been read are all concerned with problems confronting Natal sugar technologists in the production of better quality raw sugar. Starch is present in raw juice in the form of granules and since the juice is normally filtered prior to carrying out a gum analysis starch is removed and not included in the result of the analysis. But, after the juice has been heated starch passes into solution so that after that stage the gums which are precipitated in the analysis include starch. Changes may occur during processing, e.g., the retrogradation of starch and hydrolysis of

polysaccharides but the detailed picture of what actually happens is not yet clear.

Mr. Bruijn: Do the gums present in molasses and sugar include starch, because if they do a balance will not be obtained since gums in mixed juice do not include starch. I do not believe that gum is made during processing. Other organic chemicals co-precipitate with polysaccharides during analysis and the quantity of such materials is usually much greater for molasses than mixed juice. Even after repeated precipitations the protein content of gum precipitated from molasses is higher than that of a similar precipitate from mixed juice. This results in too high values being found for the gum content, especially in molasses.

Mr. Boyes: The mixed juice was not filtered prior to precipitating the gum so that the data for gums in mixed juice include starch as do those for molasses. I agree that Mr. Bruijn's explanation for the apparent increase in gums during process may be correct.

Mr. Bruijn: How do you eliminate clay, sand, etc. from mixed juice without a pre-filtration?

Mr. Boyes: The juice is passed through a fine mesh screen to remove bagacillo but clay and sand are corrected for in the ash determination which is subtracted from the total weight of precipitate.

Dr. Graham: All data given by the S.M.R.I. on gum analysis relates only to soluble gums. Even with raw sugar there is a lot of insoluble material present which, if it is not removed before the gum analysis, will have a phenomenal effect on the final answer. I think that a possible solution to the problem of obtaining a gum balance in the factory might be to carry out separate gum and starch analyses throughout the process and obtain starch-free gum data by difference. This requires further investigation.

Mr. Boyes: In my analysis only the mixed juice and filter press cake samples were screened prior to the gum precipitation. Inorganic insolubles were taken account of in the analysis by ashing the precipitate. Generally, the data are in sufficient agreement for practical conclusions to be drawn.

Dr. Graham: The method for allowing for ash in the gum precipitate is not fool-proof due to the possibility of change in composition of the inorganic material during ashing, e.g., if calcium carbonate is converted to calcium oxide the weight will be approximately halved. This factor together with that of other organic materials co-precipitated with the gums could be the major sources of error in the analysis.

I note that the alcohol/water ratio varies from one product to another in Mr. Boyes' paper, and that the S.M.R.I. and Hulsar use different such ratios for their analyses. It seems desirable to standardise the method between all three laboratories, including the pH at which precipitation should be carried out. In standardising the method consideration should be given to whether the total gum should be precipitated or the fraction which was of real significance in sugar quality considerations.

Mr. Boyes found a lower content of gums in molasses in 1962 than in 1963. Although this is contrary to the general opinion of factory operating staff it is in agreement with the fact that raw sugars produced in 1962 contained less gums than those produced in 1963. The apparent anomaly may be explained by the nature of the gums.

Mr. Boyes: Mr. Jennings says that the major impurity being contended with at the refinery is starch, whereas the data of Table I in Mr. Alexander and Dr. Graham's paper suggests that the major impurity is gums. When the paper on filterability was being read Dr. Graham said, in an aside, that carbonatation was not particularly effective in the refinery for removing gums and starch, but I recall that in a previous paper from the S.M.R.I. data was presented which indicated that carbonatation at Mount Edgecombe was effective in the removal of gums.

It is alarming to see that by adding starch to sugar the filterability of the sugar is improved. Dr. Graham also says that the pH of the clear juice should be kept high in order to reduce the P_2O_5 content. This is contrary to Dr. Douwes Dekker's advice given previously to reduce the pH of clear juice at Tongaat from 7.3.

I hope I have misunderstood Dr. Graham in thinking that the C.S.R. method should be used for filterability determinations in future as Tongaat has just spent a lot of money on the bomb apparatus.

Mr. Jennings: I said that starch is the largest single impurity in refined sugar. Gums are a group of compounds, not a single impurity.

Dr. Graham: The test which is recommended at present is the bomb test, not the C.S.R. test. Regarding the removal of gums and starch by the carbonatation process it was mentioned that carbonatation does not give complete removal. In contrast it would be possible to effect complete removal of starch and gums by precipitation with alcohol.

The data given for the refined sugars show some gums and starch concentrations which would be considered as undesirably high in raw sugar. Hence it was concluded that carbonatation was not the perfect answer for gum removal though presumably it does remove some starch.

Regarding the apparent increase in filterability by addition of starch this was the result of applying the regression equation outside the permissible limits.

Dr. Douwes Dekker: With reference to the matter of carbonatation and gum and starch removal, I recollect that data found for the gum and starch contents of syrup and molasses from Natal Estates when it was employing the carbonatation process were much lower than corresponding data from factories using defecation or sulphitation, so the conclusion could only be that gums and starch had been removed to a very large extent by the carbonatation processing.

Regarding the pH of clarified juice it has been noted that with higher pH the P_2O_5 content decreased but simultaneously the lime salts content increased. A

compromise had to be reached which it was suggested should be pH 6.9.

Mr. Alexander: It is unfair to compare carbonatation of mixed juice such as had been practised at Natal Estates with carbonatation of melt as practised at the refinery. In mixed juice carbonatation the starch is present in granular form so that there is a much better chance of removing it. This is not the case in the melt at the refinery.

Reference has been made to the mechanical removal of starch by heating the juice and also the removal of starch and gums by vegetable carbon. I remember figures of 300 p.p.m. starch in Natal Estates refined sugars which does suggest that even carbonatation of mixed juice is not the perfect answer.

I draw your attention to a C.S.R. Co. patent recently taken out in South Africa entitled "The refining of sugars using aqueous alcohols". Although I can give no information on the economic feasibility of the process it is well known from laboratory work that the use of alcohol removes not only polysaccharides but, under neutral conditions, nearly all the filter-impeding substances. If carried out at an early stage, say the raw sugar syrup, the resultant increase in purity should give a substantial increase in recovery. Furthermore, removal of gums and starches should be of advantage in all subsequent processing so that both raw house and refinery would benefit. This appears to be an important advantage over, e.g., remelting, where only the refineries may reap the benefits.

The quantity of gums received at the refinery last season was over 900 tons. Since gums polarized at 3 times that of sucrose it could be calculated that the refinery had paid nearly a quarter of a million rand for these gums.

Dr. Douwes Dekker: Referring again to carbonatation in mixed juice carbonatation it is possible that starch removal is just a mechanical filtration of the granules while in melt carbonatation the removal may be due only to absorption.

Mr. du Toit: There appears to be sufficient evidence that gums and starch have an effect on the filterability of sugar, but proof is required for some of the statements that have been made.

Mr. Boyes in his paper has drawn attention to what he calls major factors affecting the gum content of the juice, viz. age of cane, whether cane has been through a drought or not and time elapsed between cutting and crushing. Is there any proof available for the statements made? I know that tests made by Mr. Bruijn show that if cane is cut and left in storage the gum content increases. But the increase is really significant only after the cane has been kept for a long time—more than a week. I am particularly interested to know whether three year old cane has a higher gum content than two year old and whether two year old has a higher gum content than one year.

Secondly, I consider it tremendously important to know what is eliminated during processing. It is necessary to compare the percentage elimination achieved with other processes, e.g., sulphitation process.

The carbonatation process when applied at Natal Estates yielded sugar with a very high filterability, so is there not a possibility of subsidising some such process. What is important in the present discussion is whether the gums and starch in the sugar can be equated with the gums and starch in juice. Clarification and boiling processes may determine to an appreciable extent what ends up in the sugar.

We must move fast in this matter of sugar quality, and I think all processes, including the Dymond preclarification process should be examined to see how far it may be possible to get towards producing the ideal raw sugar.

Dr. Douwes Dekker: Firstly, regarding carbonatation — as far as I know one factory is seriously considering applying carbonatation, and the matter is under consideration by many responsible people in the industry.

Regarding gums in juice and sugar there is some data available which leads to the conclusion that the sulphitation process does not differ substantially from the defecation process in respect of the elimination of gums and starch in process. Carbonatation is much better, not only in respect of the removal of starch but also other non-sugars. P_2O_5 content is much lower and this can be seen from the regression formula to be an important factor.

Concerning Dymonds process, the problem is the removal of the precipitate after acidification. Tests have been started by the S.M.R.I. on this problem and although there have been some hold-ups there should be a fairly conclusive answer on the applicabitity of Dymonds process by August, 1964.

Mr. Boyes: It must be pointed out that the industry is heavily capitalised in defecation equipment and there is a growing feeling among technologists that something additional should be envisaged to take care of the gums. Maybe the syrup can be given a light carbonatation, just enough to remove sufficient gums to effect an improvement in filterability. Regarding Dr. Douwes Dekker's comments that starch removal in carbonatation of mixed juice is effective because it is not gelatinised, it is gelatinised at 160° F. and I though that at Mount Edgecombe the juice had been carbonated at a temperature higher than 160° F.

With reference to the three conditions under which cane deteriorates, the last one has been proved by Mr. Bruijn, the second is supported by data in the literature and the first should be proved or disproved by the Experiment Station. The data given in my paper for gums in molasses in 1962 tend to disprove the statement.

Mr. du Toit: There appears to be no difference from experience of drought and irrigation conditions but I wonder if there is proof of this. The starch content of

old cane does not show significantly higher values than that of younger cane nor is the gum content higher.

Mr. Rault: Sugar produced by the carbonatation process at Natal Estates compared to raw sugars produced by other processes in Natal had had 80 per cent. less colour, 70 per cent. less starch and the filterability of even the second sugars of Natal Estates had been better than that of the first sugars of all other mills. Economics is an important consideration, however, since the cost of production by the carbonatation process was about R1.50 more per ton of sugar.

Mr. Descroizilles: Using the C.S.R. apparatus on unaffinated samples, B sugar had a filterability of 10 while that of the C sugar was 3. After remelting and boiling a mixture of these sugars the filterability was in the range 55 to 63. This sugar was a dirty grey colour.

Mr. Dedekind: One sample of B sugar produced at Sezela would not filter through the C.S.R. apparatus but gave a filterability of 39 per cent. in the bomb apparatus while another sample gave identical values of 44 per cent. in both apparatuses.

Tests at Sezela have shown a correlation between pan boiling and filterability of the sugar. A badly boiled massecuite gives rise to a poor filtering sugar. During the past season Sezela did not have difficulties from filterability of sugars but had many other difficulties. Refined liquors showed an aconitic acid content of 1,100 p.p.m. on brix. This may have been responsible for observed corrosion in the milling plant and piping. It is very necessary also to check lime quality.

Mr. Gunn: Is the clarification process patented by the C.S.R. Co. so new that no thought has been given it, particularly in South Africa?

Mr. Alexander: Reference is made to the use of alcohol in an old edition of Spencer & Meade's book. This is probably not a new process but the C.S.R. Co. have possibly introduced some new features.

Mr. Dedekind: Did Mr. Rabe of Umzimkulu find an improvement in the filterability of the sugar after introduction of the Eimco belt filtration process?

Mr. Rabe: There was no difference.

Mr. Alexander: There were violent fluctuations observed in gums in final molasses in 1962 as reported in Boyes and Wilson's paper. It does not seem likely that such variations can be attributed to variation in the cane received.

Dr. Douwes Dekker: Can Mr. Boyes tell us first, has he any new data on the amount of starch removed in his starch removal trials, and secondly what happened to the starch on hydrolysis, e.g., were dextrins formed or was the hydrolysis to glucose?

Mr. Boyes: I regret I cannot answer either of those questions. In 1960 there was 50 per cent. removal of starch but in 1963 only 40 per cent. removal. This may have been due to the fact that the juice was heated to 180° F. in 1963 but to only 160° F. in 1960.

Mr. Bentley: When the starch removal process was stopped for a period of two months the refinery drew attention to a sudden increase in the starch content of the Tongaat sugar, without being aware of the changed operating conditions at Tongaat.

Dr. Douwes Dekker: This is a very simple process and could easily be employed by all sugar factories.

Mr. Boyes: There is a higher efficiency of this process for starch removal at Amatikulu than at Tongaat, which may be due to the longer retention time employed.

Mr. Fourmond: At Amatikulu the retention time is half an hour.

Mr. Boyes: At Tongaat it was reduced to 8 minutes to minimize sucrose losses, which were estimated to be 0.1 per cent.

Mr. Fourmond: According to the tables given by Stadtler for rates of inversion of sucrose at different pH's, since the reducing sugar ratio from mixed juice to syrup had decreased there could be no inversion.

Dr. Douwes Dekker: The present discussion relates to enzymatic action in mixed juice, using this particular process. This is quite different from any chemical action which might take place. Destruction of reducing sugars will take place at a later stage when the pH of the juice has increased.

Mr. Fourmond: Mr. Bruijn, when does enzymatic action cease?

Mr. Bruijn: This has not been completely investigated.

Mr. Boyes: We have determined that enzyme activity ceases at 180° F.

Mr. Kramer: It might be worthwhile to conduct a thorough investigation at Amatikulu and Tongaat into this starch removal process.

Dr. Douwes Dekker: The S.M.R.I. will be happy to co-operate.

Mr. Gunn: It should be mentioned that operation of the starch removal tanks often leads to difficulties in juice clarification.

Mr. Fourmond: We have had similar trouble at Amatikulu. Measurements carried out there confirm that operation of the starch removal tanks results in faster filtering sugars. What is the currently recommended procedure for improvement of raw sugar quality?

Dr. Douwes Dekker: The advice at the moment is to remelt. In the first place, remelt the double-cured C sugar, then, if possible the B sugar and then remelt the A sugar or double-cured A sugar. Whether there are simpler and cheaper methods is not certain but various possibilities are currently being investigated.

The remelting process has the support of the Natal Sugar Millers' Association.

Mr. Alexander: I would like to remark on the large number of questions which have gone unanswered. The establishment of a pilot factory seems the best

method for comparison or evaluation of different processes. In evaluating a process the overall picture must be ascertained, e.g., if say 0.3 per cent. sucrose were lost in the starch removal tanks might not this possibly be recovered later in process? Such data could be obtained more satisfactorily in a pilot plant than by carrying out tests at one mill and having to try and complete the work later at another mill.

Mr. Hayne: Is research going on to find new cane varieties which, like Cuban canes, will yield sugar with a high filterability?

Mr. du Toit: I do not know what wonderful varieties in Cuba are being referred to. The Experiment Station is continually carrying out variety testing agriculturally but not in a manufacturing sense. During the past year sufficiently large samples of cane have been supplied to the S.M.R.I. to permit the production of sugar on a laboratory scale. No single variety appeared to be responsible for the particularly bad filterability of Natal sugars. This is also confirmed by the fact that specified varieties give trouble in some countries whereas in other countries they do not. The trouble may be climatic. Research into the problem should be carried out and detailed factory balances made of undesirable impurities entering the factory. The tests made at the S.M.R.I. on the cane supplied from the Experiment Station may yield valuable results but having a full size mill may help further.

Mr. Fourmond: Mr. Rabe attributes the high Boiling House Performance of his factory to fresh cane. What is the filterability of the sugar?

Dr. Graham: Umzimkulu makes largely Golden Brown sugar, using a double magma system. As this sugar is for direct consumption the filterability is not normally determined as it is of no significance.

Mr. Fourmond: Amatikulu uses the same system and produces sugar with a high filterability although the cane is not very fresh.

Mr. Ashe: Mr. Kramer at a previous meeting had attributed the high filterability to the large quantity of wash water used in the centrifugals. This causes the removal of the outer layer of the crystal.

Dr. Douwes Dekker: A lot of remelting is done at Amatikulu. I wish to thank the authors for the papers dealing with a subject which is of such great importance to the Natal sugar industry. Much progress has been made in the last five years but there is still a lot to be learned.

There is evidence to indicate that there are non-sugars in our juices which are not present to the same extent in the juices of other countries. Much more information is required on the subject in order to be able to decide upon the best procedures to follow to produce a good quality raw sugar under Natal conditions.