

# COMMITTEE ON STANDARDIZATION OF CHEMICAL CONTROL

## ANNUAL REPORT FOR SEASON 1933-34.

Three meetings of the Committee have been held during the year on December 19th, 1933, January 9th and February 22nd. More work than usual has been carried on by correspondence.

### **Routine Testing of Mixed Juice.**

After an adequate trial over the past two or three years of the four-hourly system of tests, it has been unanimously decided by the committee to recommend the substitution of hourly tests. It was found that the time saved by doing fewer tests was not worth the trouble of the extra precautions necessary in preserving and sub-sampling, and in doing frequent check tests found to be necessary.

### **Filtrability Tests of Raw Sugars.**

On the advice of the International Society of Sugar Cane Technologists, who now favoured the Elliott vacuum-filter test for general comparative tests of sugars, this method had been used during the year at the Hulett's Refinery and at Darnall factory laboratory and had been found consistent and of value provided conditions and equipment were kept as uniform as possible. Much depended on using the right standard filter cloth and maintaining a constant temperature of the test liquid. Very useful work in this connection was done by Messrs. Blacklock, Dymond and Viger. [See in this matter F. W. Zerban "Filtrability Tests on Cane Sugars." **Facts About Sugar**, January, 1934, 7.]

### **General Examination of Raw Sugars.**

It was decided at the beginning of the season to ask all factories exporting raw sugar to send a representative sample of each lot manufactured to the experiment station, stating the polarization, moisture and sulphur dioxide content.

These samples were stored at the experiment station primarily for purposes of reference in case of any question arising with refiners overseas about any particular batch of sugar.

Later it was decided to divert part of these samples to a general examination of the sugars, compositing a sufficient quantity from each factory represented. For further comparison typical specimens of sugars from various other countries including Mauritius, Java, Trinidad, Puerto Rico, Queensland, British Guiana, Great Britain (beet), Cuba, and Reunion, were obtained and included in the series.

Owing to lack of available chemical staff and deficient water supply at the experiment station it was impossible at the time to carry out the work there. Consequently Sir J. L. Hulett & Sons, Ltd.,

kindly undertook the work at their Darnall factory research laboratory, under the supervision of Mr. G. C. Dymond.

The results throw a good deal of light on the factors affecting the keeping and the refining qualities of Natal raw sugars.

In general the change in polarization on storage varies very greatly with different sugars ranging from a loss of 2.2° to a gain of 0.4°.

On the whole a high loss in polarization appeared to be associated with a high safety factor and vice versa, though there were many exceptions; and generally speaking the necessity of thorough drying of raw sugars was indicated.

A substantial fall in polarization of a large consignment of sugar, of course represents a great loss in value, to an extent that is, perhaps, not generally realised.

Sulphur dioxide was determined by the iodine titration method and ranged from a minimum of 18 to a maximum of 75 parts per million. All overseas sugars, except British beet, indicated appreciable amounts of sulphur dioxide by the iodine method. Since iodine of course reacts with many substances besides sulphur dioxide it would be advisable in future work to apply a more specific test.

At all events, from this and other indications, it is evident that much less sulphur dioxide is now allowed to remain in Natal raw sugars than was the case formerly.

The hydrogen ion concentration of Natal sugars ranged from a pH of 6.0 to 6.9; that of overseas cane sugars from 4.4 to 6.3, while British beet sugar was 7.5.

In total ash content Natal sugars ranged from 0.12 to 0.40 per cent and did not differ appreciably from overseas sugars in this respect. A certain correlation was observed between rate of filtration and content of silica, the higher the silica the lower the filtration rate. No such correlation was observed with lime content.

Reducing sugar content in Natal sugars varied from 0.20 to 2.22 per cent in Natal sugars and from 0.14 to 1.45 per cent in overseas cane sugars (British beet, nil).

An increase in reducing sugar content of sugars from the same factory was in several instances associated with a fall in filtration rate and general deterioration in quality of sugar.

Starch, or rather the group of substances forming the starch complex in sugar, was tested for by

the blue to red colour formed with solutions of iodine. There appeared to be no relation between the depth or nature of the colour and the filtering rate of the sugars, but it is of interest to note that while a few Natal sugars showed an appreciable quantity of starch as determined by treatment with diastase and volumetric estimation of the extra reducing sugars thereby formed, there was no measurable quantity found in any of the overseas sugars tested.

In tests for so-called "gums," that is the measurement of the precipitate produced by alcohol, the weight of the precipitate varied from 0.10 to 0.39 per cent of weight of sample in Natal sugars, and from 0.11 to 0.26 in sugars from overseas. In some cases, an increase in gum content between sugars from the same factory was associated with a fall in filtration rate, and also with a drop in polarization.

The rate of filtration has already been mentioned in relation to various other factors. It was measured by the standard Elliott method restandardised by the International Society of Sugar Cane Technologists as referred to above. The results varied greatly between samples from different factories and also in samples from the same factory taken at different parts of the season, showing how sensitive this property is to various influences as yet not understood.

A tabulation of the different factories in order of filtration rates of sugar at various periods of the season show that in general there is a fall in rate of filtration towards the end of the season when the quality of the cane and juice deteriorate and when climatic conditions are more favourable to the rapid deterioration of products. Any rate of filtration seems to be liable to this fall, whether initially high or low. Most factories seemed to be subject to this fall, though it was noted that the Zululand factories, in the main, were not. Some factories maintained a higher filtration than others throughout the season while some were uniformly relatively low. It was noted that the carbonation factory had a relatively high filtration rate. Of the overseas sugars British beet had the highest rate of filtration, 113.59, followed by sugars from Mauritius and Java, that from Reunion being comparatively low.

There was indication that the method of disposal of treacle sugar had an important effect on filtrability. Where treacle sugar was mixed with raws, there was a marked effect on rate of filtration.

This work is of the greatest importance to the industry and should be carried on next season.

While fully appreciating Messrs. Hulett's services to the industry in this matter, and without detracting from their work in the least, for various reasons it is considered advisable that this sort of work, of benefit to the industry as a whole, should be done at a central independent research laboratory, as

at the experiment station, which was established for this kind of purpose.

Copies of Mr. Dymond's final report will be supplied to members on request. It is not proposed to publish it in the proceedings of this conference because of the large amount of tabular matter and the fact that individual factories are mentioned by name.

#### **Collection of Raw Sugar Samples, 1934/35 Season.**

In continuation of the above work it is recommended that monthly composite samples of raw sugar be sent to the experiment station from each factory during the coming season, such samples to be distinct from the lot samples of export sugar sent to the experiment station for reference purposes, which latter samples would then be preserved intact.

#### **Baryta Method of Sucrose Determination.**

Work on this has been continued at the experiment station and Dr. E. P. Hedley reports having found that baryta does not remove amino-acid amides such as asparagine and glutamine, but hydrolyses them into the corresponding amino acids, aspartic and glutamic acids.

All the common salts of the dibasic amino acids such as the former, e.g., the calcium and lead salts, are soluble in water, and there appears to be no convenient way yet known of eliminating this group of optically active impurities that are very apt to occur in sugar house products, especially in molasses.

This is a serious objection to the baryta method, an objection which is not peculiar to that method, but certainly invalidates one of the special claims for its adoption.

#### **Clerget Method of Sucrose Determination.**

If the baryta method cannot be adopted, some alternative, at all events, will have to be found for the present method of acid inversion specified in our official methods.

Any acid inversion method is subject to error through the hydrolysis of reversion products but as pointed out by Dr. F. W. Zerban in the "Report of the Special Committee on Uniformity in Reporting Factory Data" at the Puerto Rico Congress of the International Society of Sugar Cane Technologists, methods Nos. 2 and 4 of Jackson & Gillis as published in Scientific Paper No. 375 of the U. S. Bureau of Standards are to be preferred as least affected by these errors.

Copies of these methods are obtainable by members from the experiment station on request, and subject to the approval of this conference, it is proposed to adopt No. 4 method in the forthcoming revision of our official methods.

The invertase method is the best in principle, but it cannot be said that it is yet suitably adapted for factory routine use.

### Soil Analysis.

In view of the growing interest taken in the study of the soil, and the importance of a knowledge of the composition of the soil when allied to a knowledge of its agricultural qualities as shown by field experiment or experience, it is tentatively suggested that methods of sampling and of chemical and physical analysis of soil be embodied in our official methods. To that end a sub-committee consisting of Messrs. B. E. Beater (convener), H. H. Dodds, G. C. Dymond, J. Rault, and M. Viger was appointed to discuss alternative methods and select a scheme for adoption. This work is now in progress.

### Normal Weight Method of Pol Determination.

This method has been tried at several factories and is strongly recommended by those who have tried it. It has been resolved therefore to recommend it for adoption as an alternative or tentative method in our official methods for use with products to be diluted.

### Laboratory Equipment.

Certain discrepancies in the analysis of juice occurred at a certain factory and formed the subject of an inquiry by an experiment station chemist. The fault was found to lie in the saccharimeter tubes and their cover glasses and as a result of this it was resolved that saccharimeter tubes and fittings should be specified and standardised in our official methods.

**Immersion Refractometer:** The advantages of the refractometric method of determination of total solids in solution have long been recognised but the method has never come into general use, mainly because existing refractometers were not particularly well adapted for sugar products.

A new immersion refractometer described by Bachler in "Facts About Sugar," November, 1933, is said to overcome the objections to the older types of refractometer. One of this model is on order for the experiment station, so that we shall soon have an opportunity of testing its usefulness for local conditions.

**Hand Refractometer:** The Zeiss hand refractometer has been tried for field use and is capable of giving very clear readings with very simple and expeditious manipulation. It is of comparatively little use under our conditions for its original purpose of testing cane in the field for solids in solu-

tion in juice, however, because of the difficulty in getting two concordant readings from the same stool, or even from different parts of the same cane. This is particularly the case in immature cane.

The hand refractometer may serve to indicate in a very general way which of two fields is the more ripe by comparing the average of a number of tests of each at about the same part of the cane stalk, or by comparing the relative differences between different ends of a number of sticks from each field. In general, the wider the difference in total solids of juice between two ends of the same cane, the less ripe it is.

### Methods recommended by the Special Committee on Uniformity in Reporting Factory Data of the International Society of Sugar Cane Technologists.

Having realised the many advantages gained within recent years by the adoption of uniform methods in this country, we are naturally specially desirous to come into line, as far as possible, also with any schedule of methods recommended and adopted for international use. It is satisfactory to note that progress is being made, if somewhat slowly.

We would like to see, besides an internationally adopted scheme of definitions and methods of sampling, analysing, interpreting, and recording, some plan for a methodical interchange of results with perhaps an annual summary issued by the International Society, of factory results from the leading sugar countries of the world.

We are of opinion that the term "boiling house recovery" which is now commonly held in most countries to mean "sucrose (pol) in sugar per cent of sucrose (pol) in mixed juice" should be applied to "sucrose (pol) in sugar per cent of sucrose (pol) in syrup" so as to confine it, as the name implies, to actual boiling house work. The term "Recovery on mixed juice" as used in South Africa, or some other suitable phrase could then be used to define "sucrose (pol) in sugar per cent of sucrose (pol) in mixed juice" which of course takes into account all sources of loss consequent on treatment of mixed juice (filtering and the like) as well as actual boiling so should not be described merely as "boiling house recovery."

This is perhaps the only matter of any consequence in which our practice in this country differs from that of most other countries, except in our use of the "short" ton of 2,000 lbs. The latter can only be defended on the grounds of its fact that it is firmly entrenched in our commercial practice. The metric system is undoubtedly the only logical one to use with a decimal system of numbers, but the difficulties in its general adoption are very great.

For those who do not possess copies of the Proceedings of the Puerto Rico Conference, a detailed abstract of the excellent report on uniformity in factory data prepared for the International Society by Dr. Zerban, will be found in the "International Sugar Journal" for October, November and December last.

Experiment Station,  
South African Sugar Association,  
Mount Edgecombe.

March, 1934.

### Committee on Standardization of Chemical Control:

R. M. Bechard.  
L. Blacklock.  
W. O. Christianson.  
P. L. Draeger.  
G. C. Dymond.  
W. H. Foster.  
E. Haddon.

F. W. Hayes.  
E. P. Hedley.  
G. S. Moberly.  
J. Rault.  
M. Viger.  
H. H. Dodds (*Convener*).

CHAIRMAN: In this paper there are three recommendations which the Committee wants this meeting to adopt, but before I put those to the meeting I think we ought to have the report under discussion. The first recommendation is the hourly test against the four-hourly test; the second is the Clerget method of sucrose determination, and the third is the normal weight method of determination of sucrose. The report is now open for discussion.

Mr. BECHARD: Regarding the normal weight method I see it is suggested that an alternative method should be used. Does that mean it is only allowed for products to be diluted? We have used it for all of our products of the factory, that is everything apart from the mill juices, and we found it very convenient and on the whole more reliable than the dilution method of 100/110.

Mr. DODDS: The Committee intended to countenance the use of this method for everything except mixed juice. The only reason why it should not be applied to mixed juice is the fact that it is not recognised in the Fahey Conference Agreement, but if on further experience of this method we find we wish to use it for mixed juice also, I have no doubt arrangements could be made at the next revision of the Fahey Agreement. That was the only objection to its use that we had in mind.

Mr. BECHARD: So that it would be perfectly in order to use it for everything except mixed juice.

Mr. MOBERLY: And crusher juice.

Mr. BECHARD: Of course crusher juice does not enter into the agreement so far as the laboratory is concerned.

Mr. MOBERLY: Except that the checks are carried out on the crusher juice analysis, and the check test should be done by the same method.

CHAIRMAN: There are one or two points I have noticed in this report. The first is sulphur dioxide in sugar; that has come down with a big bump and it is very fortunate for us that it has done so. That is one of the achievements of this Technologists' Association. A Committee was sent around to investigate the reason for high sulphur dioxide content and from the recommendations put forward it seems this has been rectified. The next point is the mixing of treacle sugar with raw sugar. It seems to me that is very bad practice. I have had correspondence on sugar manufacture with an engineer in America and one of the chief faults he points out is the mixing of treacle sugar and raw sugar, especially where you make raw sugar for refining yourselves. If they have that trouble with soft canes in other countries it must be very much worse with Uba here. The next point is the collection of raw sugar samples. Mr. Dodds suggests here that two lots of samples should be sent, one for filtration tests and the other for reference. Does he mean exactly that separate samples per month have to be sent to the Experiment Station?

Mr. DODDS: It means that as well as the lot samples we have been getting during the past season we would have a monthly composite sample distinct from that, representing the total output from the factory. At present we are getting lot samples from each separate lot consigned for export.

Mr. BECHARD: The lot sample is not a monthly sample but is made daily, and sometimes three times

daily. I understand some factories sent as much as 450 samples to the Experiment Station.

Mr. DODDS: If not quite as many as that, there have been a great number in some cases. At all events I may say we have had great difficulty in finding storage room for these samples.

Mr. BECHARD: If we were notified at the factories of the composition of various cargoes shipped we could make a composite sample representing that particular cargo. In that case it would amount to only about a dozen samples in the year. I do not know whether it is feasible. The Refinery have the actual composition of each cargo by trucks, and it would be possible for us to composite a sample from our own samples kept beforehand. It would reduce the quantity of samples sent to the Experiment Station considerably.

SECRETARY: This is probably a matter for inquiry but at first blush it seems to me to be not very easy. You might quite readily get a situation in which it could not possibly be done and your whole year's work might be more or less vitiated. However, it could be inquired into. I think it must be kept in mind that as far as possible the Experiment Station should be kept out of the position of arbiter in regard to quality of sugar shipped overseas. For information of our own factories the work can be carried on without reference at all to what the outcome of the sugars may be in the hands of the buyers overseas. I think these samples must not be looked upon as sent in for the purpose of forming standards on which the output overseas is judged. That is a very different matter altogether from getting samples for the purpose of investigating the sulphur dioxide conditions of the factories in their ordinary working. It must be kept definitely in mind that those sugars can only be in a very general way regarded as official representative samples of export sugars, although of course they are definitely samples of the sugar turned out in the factories. You will get in all sorts of difficulties if you look upon them as representative samples.

Dr. HEDLEY: I might say that the object of having those samples was that we should have representative samples of what comes from the factories. We discussed the idea of getting samples of the batches as they went overseas, and found it would mean having a man employed solely to do this and even then it would be exceedingly difficult to get correct samples of the batches as they went overseas. To get a sample representative of what actually goes to any buyer overseas is an impossibility here and we decided the best way to do this was to have samples of every 100 tons sent to the Experiment Station and when an occasion to check up analyses arose the samples were there for the purpose. Having an extra composite sample does

not mean very much more sugar; we do not require large samples.

Mr. DODDS: As Mr. Eadie says, the samples taken and sent to us cannot be regarded in any way as standard or official samples in case of dispute, if only for the fact that they are not sealed and are not taken with the precautions required for official samples of a cargo. They are merely taken for our own information.

Mr. BECHARD: The next point, on the first page of the report, bottom of the second column: "An increase in reducing sugar content of sugars from the same factory was in several instances associated with a fall in filtration rate and general deterioration in quality of sugar."

I have in mind the report of the Atlantic Refining Co., in which one of the shipments of sugar sent to Canada was fairly highly commended. They said it was probably one of the best shipments they had. When it came to detailed analysis of that particular sugar it is rather disappointing to see that this sugar undoubtedly inverted considerably during shipment. The general polarisation dropped 1.2 or 1.3 and yet that sugar was reckoned as being one of the best shipments received. That convinced me that it was possible for any sugar to be accidentally inverted. I was not present at the last meeting of the Chemical Control Committee, but if I remember rightly at the previous meeting I suggested that investigation might be made into the effect of deterioration of sugar on filtration.

Mr. DODDS: Evidently the matter is a complex one affected by a good many factors and much work is necessary before we can come to any definite conclusion on the matter.

Mr. BLACKLOCK: I remember the cargo that Mr. Bechard is talking about. It evidently suffered inversion and deterioration and this cargo is specially mentioned as showing a decided improvement in filtrability. Our own results with the Elliott tests in respect of deteriorating sugars are somewhat conflicting. Some were improved, some were adversely affected, and others did not vary materially. The Research Department at Darnall did not agree with that. We did not agree with the conclusions drawn from the analyses of the Atlantic Refining staff. Apparently it is possible for deterioration to take the form of a gummy fermentation and then loss of filtrability will occur. This should be confirmed by an increase in the colloidal content or gummy content as shown by the alcohol gel test. Practical factory experience of deteriorating sugars from storage has always created the impression that their filtrability has been improved rather than otherwise. In this connection the earlier tests as carried out during the season afforded some useful information in spite of a certain amount of trouble and uncertainty

caused by difficulty in obtaining concordant results, which difficulties were finally traced to the tightening up in use or filling of the standard filter cloth when repeatedly used. However, when the procedure and apparatus become more strictly standardised the tests should be more informative and useful.

Mr. DODDS: With regard to the procedure by the Elliott test, a report recently received from Dr. Zerban states that the standard filter cloth, which is the one Mr. Blacklock is using, should only be used once and should be very securely fixed, which I suppose implies being wired in position.

Mr. BLACKLOCK: That clears up the point, because I also think the cloth should only be used once. We find even on the second time of using a very much decreased filtering from that same cloth.

CHAIRMAN: I would like to ask Mr. Blacklock does it make any difference which side of the cloth you put at the top?

Mr. DYMOND: I would like to emphasise the point that Mr. Dodds made that this investigation so far has been purely a tentative one, but it has drawn attention to many factors which may influence the filtration rate of our sugars which may be colloidal, may be due to gummy products, fermentation, and so on. When I was overseas the tendency for testing out filtrability of sugars seemed to be rather on the basis of hot filtration tests, and it was quite a surprise to me that they seemed to have come back to cold filtration tests now, that is the Elliott, and we have used that and not tried out the hot filtration tests at all. I think both these tests should be used because it is possible that the effects of gummy fermentation might show up in one method and not in the other, and it is possible that there may be no correlation between the Elliott results and those shown by the Refinery. As regards the collection of samples, we think that it is most important that the mills should co-operate more fully this year in giving us more samples than we have had. We split the numbers of samples we obtained last year for export sugars into three periods only—the first part of the season, the middle, and the last. And it was only with the last lot of samples that we obtained remarkable results such as big drops in filtration rate, big drops in polarisation, and so on, and we definitely recommend that all the mills should give a monthly sample of all their products so that this investigation can be carried out in a more thorough manner this year. There is one point that has been brought to light in this investigation and that is that the loss in polarisation amounts to a considerable amount of money in many factories. The drop in polarisation in one factory was over 2, and that was for a bottled sample. What it was under bagged

conditions I do not know but the loss must have been rather heavy.

CHAIRMAN: I can give our experience at Illovo. We do not keep our sugars I suppose more than half an hour, so they do not have time to dry out or invert. As soon as the raw sugar is made it is washed up to 99° and put into the Refinery, and we have tried everything that may affect filtration, and are no further now than when we started. We know different methods of clarification affect filtrability. We know that various methods of treatment at the presses affect it and we know that different methods of pan boiling do so. From what I can see the whole problem is that there is some substance which goes right through and is absorbed by the grain of the sugar on pan boiling. You will get a 98.5° sugar which will filter beautifully but a 99.5° will not. Now it is reasonable to expect that a 99.5° sugar is a fairly good grade, as it must be if washed up properly. But if you melt that down it will not filter at all well, and even bringing the density of the melt down within reasonable limits does not seem to make much difference. So I think the investigation should go on in regard to what the substance is that is occluded in the crystal during pan boiling.

If there is no further discussion I will put the three recommendations to the meeting. We had better take them one by one. The first is the routine tests of mixed juice; it is recommended that hourly samples should be done rather than four-hourly.

On being put to the meeting, on a show of hands the recommendation was agreed to unanimously.

The second recommendation, Clerget method of sucrose determination, method No. 4 of Jackson & Gillis as published in Scientific Paper No. 375 of the U.S. Bureau of Standards was agreed to with two dissentients.

The third recommendation, Normal weight method of Pol. Determination as an alternative or tentative method in the official methods for use with products to be diluted, was then put to the meeting.

Mr. BECHARD proposed that the words "except mixed juice and crusher juice" be added after "products to be diluted."

Mr. DODDS stated that the phrase "products to be diluted" was used in its broadest sense, and they intended to include clarified juice. All they had in mind to exclude was the crusher juice and mixed juice. If the meeting wished to accept the amendment and have it recorded more explicitly he would concur.

On being put to the meeting the recommendation as amended was agreed to unanimously.

CHAIRMAN: I wish to thank Mr. Dymond for all the work he has done on raw sugar investigation and also Mr. Dodds for this report. (Loud applause.)

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The President called upon Mr. Dymond to read the Report of the Clarification and Filtration Committee.

Mr. DYMOND: Before reading this Report, I would like to explain the difficulties the Committee were faced with in writing such a report. We found we had quite a mass of information, and we had either to write a book, or write a somewhat restricted report, such as we have here. There are a number of points in it which, we felt, would form the basis of a discussion, and the papers that are referred to in the report can be had by those interested from the Technical Secretary.

Read by Mr. Dymond.