COMMITTEE ON STANDARDIZATION OF CHEMICAL CONTROL

DRAFT ANNUAL REPORT FOR SEASON 1935-36

Three meetings of the Committee were held during the year, on May 9th, September 27th, and February 20th. The Convener was absent from the country from August 2nd to December 4th, Mr. F. W. Hayes acting as Convener during that period.

METHOD OF INVERSION OF SUCROSE FOR STANDARD CLERGET TEST FOR SUCROSE DETERMINATION.

A more satisfactory and reliable method of inversion for double polarization than the one originally tentatively adopted by us has been under consideration for the past two or three years.

Jackson & Gillis method No. 4, as published in Scientific Pauer No. 375 of the U.S. Bureau of Standards, has been again subjected to exhaustive comparative tests in many laboratories, especially at the Umfolozi, Empangeni, Umhlatuzi, Amatikulu, Darnall, and Maidstone factories, with the result that it has been found entirely satisfactory in use and indicated results of no significant difference from those obtained by the former official method of inversion.

The Natal Sugar Millers' Association has been informed accordingly and urged to sanction the change to this method for all South African factories.

PUBLICATION OF REVISED OFFICIAL METHODS OF SAMPLING, TESTING, AND CALCULATION OF RESULTS.

This work, now considerably overdue, has again been postponed because of possible changes in practice arising out of the revision of the public agreement regulating the terms and conditions of the purchase of sugar cane (Fahey Conference Agreement). In any case there would have been little time for this work during the past year.

NORMAL WEIGHT METHOD.

This was adopted tentatively two seasons ago as an alternative method for use with all products except mixed juice for sucrose test. Since further experience with the method has also been entirely satisfactory, the Committee now recommends the adoption of the method for general use, including the analysis of mixed juice for calculating the sucrose content of cane, replacing the Schmitz dilution (100ml.-110ml.) method.

In the application of the normal weight method it is recommended that 2½ times the normal weight (65 grams) be weighed into a 100 ml. flask, or five normal weights (130 grams) into a 200 ml. flask, according to quantity of sample required for test; then make up volume to the mark, add sufficient lead subacetate (Hornes "dry lead"), filter, de-lead, and polarize in a 400 mm. saccharimeter tube.

TESTING OF CRUSHER JUICE FOR SUCROSE DETERMINATION OF INDIVIDUAL CONSIGNMENTS OF CANE.

For the purpose of analysing samples of individual consignments of cane from crusher juice tests, it is recommended to use the original undiluted juice, clarifying by means of Horne's dry lead, beginning with the 1937 crop and continuing till then the use of the Schmitz dilution method.

The Committee has under consideration the adoption of the Pellet tube, but desires to gain further experience with this apparatus before definitely recommending it.

MUTAROTATION.

The possibility of mutarotation of invert sugar occurring after inversion has been pointed out. There may be a tendency for the attainment of the normal rotation to be a little delayed for various reasons. It is therefore recommended that inverted solutions be allowed to stand for half an hour before the final polarization to ensure a constant reading.

THE INTERNATIONAL SOCIETY OF SUGAR CANE TECHNOLOGISTS.

The Convener attended the Queensland Conference of this society in August last. He reported the presentation of the Report of the Special Committee on Uniformity in Reporting Factory Data by the chairman of that committee, Dr. F. W. Zerban, and a subsequent discussion thereon. Very few changes were made from the draft report presented at the Puerto Rico Conference. The Committee had decided by a majority to adopt Noel Deërr's "reduced extraction" figure, but not his proposal to calculate the relative efficiency of operation of different mills by taking into account the degree of imbibition, size and number of milling units, and the average fibre content of the cane.

Apparently the only matters in which we in South Africa have not come into line with the international committee are (1) that we have not adopted the metric system, for example, in the recording of quantities of massecuites in hectolitres

instead of in cubic feet, and (2) our retention of the phrase "recovery on mixed juice" instead of "boiling house recovery," to describe sucrose in sugar per cent. of sucrose in juice.

STUDY OF RAW SUGARS.

There was a most interesting symposium at the Queensland Conference on the manufacture of raw sugar, and the study of its refining and filtering qualities. In this connection the Convener prepared for presentation and read at the Conference an account of the work done in this matter at the Experiment Station last season by F. W. Hayes, and briefly noted in the last Annual Report of this Committee. This paper will appear in the proceedings of the International Conference, and in the meantime separates are available at the Experiment Station here for those desiring them.

OTHER CONFERENCE PAPERS.

Among other interesting papers at the International Conference on the control of manufacture were those by D. L. McBryde on "Pan Boiling by Conductivity Control in Queensland," and R. E. Simmonds "The Automatic Recording and Controlling of the pH of Thin Juice," and "The Normal Weight Question in the Analysis of Sugar Factory Products," by C. A. Browne.

USE OF "REDUCED EXTRACTION" AND "REDUCED RECOVERY" FORMULAE.

Last season we introduced for the first time at the suggestion of the International Society of Sugar Cane Technologists, the above formulae which are based on an assumed standard fibre content of cane of 12.5 per cent. for calculating relative extraction, and a mixed juice purity of 85 for calculating a boiling house recovery (recovery on mixed juice) from the "virtual" gravity purity of the molasses calculated by the s-j-m formula from the observed purity of the mixed juice and the actual boiling house recovery.

The Reduced Extraction value is of some value in indicating the minimum increased extraction that might be expected from a cane variety of lower fibre content than Uba, though it does not take into consideration the difference in quality of fibre from different varieties of cane. The fibre of Uba cane seems to be more highly lignified than the fibre of so-called soft canes, and is harder and more refractory, though only its lesser quantity is taken into account in the "Reduced Extraction" formula.

In "Reduced Boiling House Recovery" and "Reduced Overall Recovery," the standard mixed juice purity of 85 is one which is often exceeded by Uba juice. These formulae take no account of the special difficulties in recovering sugar from Uba juices and syrups due to the nature of the non-sugars in this type of cane and their greater content of colloids entailing relatively high losses of time and of sucrose, both at the filter station and in the boiling processes.

These difficulties are universally experienced with Uba juice as it exists in this country, notwithstanding the relatively high juice purity, so that no one has yet succeeded in making any grade of commercial sugar without sulphitation, a process seldom, if ever, used for raw sugars in other countries.

Consequently, although the Reduced Extraction does to some extent take into account the special difficulties in crushing Uba cane, as far as its high fibre content is concerned, the Reduced Recovery formulae are of no value in making comparisons between Uba and other varieties, because they take into account only the relative quantities of non-sugars (which are not usually abnormally high in Uba juice) and not their nature.

INTERNATIONAL COMMISSION FOR UNIFORM METHODS OF SUGAR ANALYSIS.

This is a body that was appointed to compile specifications of tests for sugar and sugar products that should become recognised international standard tests. It is therefore important for any sugar producing country to have some say in the framing of these specifications, which may have important consequences for that country.

At the last meeting of this Commission, the Eighth Session, which was held in Amsterdam in September, 1932, the Ninth Session was called for London, where it will be held in August of this year. South Africa was invited to separate membership for the first time (having previously been represented only by the British delegation), and urged to form a National Committee.

Two of our members have been appointed associate referees to the Commission, H. H. Dodds for subject 6, the Testing of Molasses; and E. P. Hedley for subject 15, the Determination of Moisture in Sugars, and Sugar Products by Drying Methods.

This Committee recommends, and the General Committee of our Association, as well as Dr. Bates, President of the International Commission, approves, that in the meantime we act as the National Committee for South Africa for purposes of the International Commission.

Suggestions would be welcomed for any suitable member of our Association, who may happen to be in England next August, to act as our delegate at the Ninth Session.

Voting power on the Commission of any country is determined by the total quantity of produced and imported sugar combined; under the present scale South Africa is entitled to two votes on a quantity of sugar between 300,000 and 600,000 tons produced and imported.

Committee on Standardisation of Chemical Control:

R. M. BECHARD.
L. BLACKLOCK.
B. P. CAMPBELL.
W. O. CHRISTIANSON.
G. C. DYMOND.
P. L. DRAEGER.
W. H. FOSTER.
F. W. HAYES.
E. P. HEDLEY.
G. S. MOBERLY.
B. E. D. PEARCE.
J. RAULT.
M. VIGER.
H. H. DODDS (Convener).

Experiment Station,
South African Sugar Association,
Mount Edgecombe, Natal,
March, 1936.

The PRESIDENT: Thank you, Mr. Dodds. The Report is now open for discussion.

Mr. MOBERLEY: Mr. Chairman, there are just two points I wanted to mention in connection with the changes recommended, and that is Horne's dry lead and the Pellet tube for testing crusher juice samples. That paragraph about the use of the Pellet tube should really come under the following heading "Testing of Crusher Juice for Sucrose Determination of Individual Consignments of Cane," because it is not proposed to use it for any other purpose. Its value lies mainly when a large number of consecutive tests of products of about the same sucrose content are being carried out. I have had some experience with the Pellet tube method, and although a number of people are suspicious of it, I have always found it a very useful method and a great time-saver. Generally the fear is that there will be mixing of two consecutive samples, that you may not get your next sample clearly isolated in the tube. But there is no real danger from that, because you will find in using this method that as long as there is any admixture between the previous sample and the one you are trying to do, you will not be able to read the instrument. The moment it clears and becomes transparent. you have a certain indication that your tube is filled entirely with the new liquid that you are going to polarize. The fitting up and manipulation of the apparatus is quite simple, and in those tests where you very often have a large number of polarizations to do one after the other, the time saved is considerable. However, we have decidd to wait a little before actually putting it into effect. There is a good deal in adjusting the tube, and the inlet and outlet should be so arranged as to get a good circulation, and a sort of swirling motion to drive out the previous solution that was in the tube.

With regard to Horne's dry lead, that was also recommended as a time-saving operation, and I have also found that useful, with one thing to bear in mind. The great difficulty about it is getting the right amount of lead into the juice. As in all polarizations, you should only use as much as is necessary. The tendency of the average laboratory assistant is to make sure of it and put in too much. As I have seen it worked, it has usually been done by taking some on the end of a spatula. When I started the experiment, I got Horne's dry lead made up into tablets, so that a definite counted number of tablets should be put in. I had some of those made up at Lennons, but the first lot which came to hand were made much too hard. They would not dissolve readily but needed quite a lot of breaking up. I hope to go on with this and see if I can get them to make up a fairly soft, friable tablet, which will break up readily in the juice, and in that way we will be able to standardise the amount of lead put in with each test—one, two or three tablets, according to whatever quantity is necessary. I hope during this coming season to be able to run a series of comparative tests with these two—the Pellet tube and Horne's dry lead. Next season we may be able to put them into definite use.

Mr. BECHARD: Mr. Chairman, with regard to the phenomenon of mutarotation, Mr. Buchanan and I drew the attention of the Committee to the state of affairs. On one particular occasion, over a seven days' run, we definitely had mutarotation for some reason or other that afterwards disappeared. One thing that ought to be made clear is that the time that the solution has to stand is half an hour after making up to the mark. For obviating the difficulties of mutarotation I have found myself that half an hour is sufficient, in fact 20 minutes is actually enough but I would not like to recommend it, and half an hour is a safe margin.

The next point I want to bring up is the question of reduced recovery, and it has been in my mind for some time that we may be able to correlate recoveries with purities if we take into consideration the varying quantity of reducing sugar.

Mr. HAYES: Mr. Chairman and gentlemen, this question of mutarotation is a rather puzzling one. As far as I remember Mr. Buchanan did not have much difficulty with mutarotation in routine analysis. There is one point, however, in this paragraph on mutarotation, which will have to be corrected. It is stated that "It is therefore recommended that inverted solutions to which sodium cloride has been added (as in the Jackson & Gillis method) be allowed to stand for half an hour before the final polarization to ensure a constant reading."

Of course there is no sodium chloride added to the inverted solution. It seems to me that the trouble lies in some analytical procedure, and is not entirely due to mutarotation, because the inverted solution is of sufficient acidity to inhibit any such phenomena. The higher the acidity of a solution of invert sugar, the less the extent of possible mutarotation, and the quicker it attains optical stability. But there is still a great deal of work to be done on this subject. We have not had any real working difficulty at the Refinery with the phenomenon of mutarotation, and quite a number of factories state they have never experienced it.

Mr. BECHARD: I might say, in explanation of what I have said before, that I do not associate the Jackson & Gillis method with mutarotation in any way, but it just happened that for two or three days we had difficulty. It simply came and went away again for some undetermined reason.

Mr. BIJOUX: In connection with the point of mutarotation, I should like to point out that last year we did have some considerable difficulty. But I think it is mostly due to the dilution caused by making up to the mark rapidly.

Mr. BIJOUX: With regard to the point that Mr. Moberly brought up about the lead acetate, I think it is rather difficult at times to gauge what is the right amount to put in. If we judged by the original Brix before doing the clarification, it would be helpful. The higher the Brix, the more clarifying agent we should have to put in.

Mr. HAYES: In one respect Mr. Bijoux is correct. Where a solution is made up in a fairly concentrated state and then diluted and polarised immediately, there is more likelihood of an occurence of the phenomenon of mutarotation than in one which has been made very closely to the concentration at which it is to be read. It is not a question of thorough mixing in the flask, but rather of what time has elapsed from the making of the solution in the flask to the final dilution and adjustment to the mark.

Mr. DUCHENNE: With reference to the question of de-leading, it must be evident to any chemist that this is an absolute essential. Were not the solution diluted the sodium chloride would precipitate with the lead that would be left over, and therefore nullifyy the addition of any sodium chloride afterwards. I pointed this out to the Committee, and I was told that the de-leading had been made obligatory.

Mr. DODDS: Mr. Chairman, with reference to the remarks on mutarotation, it is certainly an oversight in the report that I wrote of the inverted solutions to which sodium chloride has been added. It is, of course, not added to the inverted solution at all, but to the other one for comparison, the idea being, of course, to add an approximately equivalent chloridion to that added in the Hydrochloric acid used for inversion.

I certainly understood from Mr. Bechard that this had something to do with the method of inversion, because, as is well known, mutarotation speedily disappears in the case of an alkaline or acid solution, but can be slightly prolonged in the presence of sodium chloride. But apparently that was not the case. I am glad to have Mr. Bechard's assurance that it was not due to any proposed modification of the method of testing, but to some natural causes which are unknown.

One or two matters, such as de-leading of the solution, which I understand is now not optional, but is obligatory, I will ask Mr. Hayes to speak on, as he was convener during the period when this matter was discussed.

Mr. HAYES: Further to Mr. Dodds' remarks in replying to Mr. Duchenne, I must say that the Committee realised right from the start of the work with the Jackson & Gillis method, that, especially in impure solutions, where the quantity of lead added in all likelihood be excessive, the sodium chloride added to the solution for direct Pol. would be very largely eliminated and precipitated as lead chloride, and hence the effective sodium chloride left in the solution would be greatly reduced. The Committee realised this, and it will be found recorded in the minutes. Nevertheless there were objections raised to de-leading with mixed juice. But at no time was the de-leading procedure left as optional with molasses. Mr. Duchene wrote to the Committee from Umfolozi, pointing out the possible error by omitting de-leading after we had discussed it several times, and reports containing these decisions had been circulated. Subsequently we decided to make the de-leading procedure obligatory for molasses, mixed juice and all products, irrespective of the stage in manufacture. We wrote Mr. Duchenne to that effect, explaining the Committee's views, which were entirely in accordance with those he expressed, since it was only logical that any sodium chloride added to the solution would be largely precipitated by any excess of lead allowed to remain. At present there is nothing tentative about the method of de-leading by means of potassium oxalate in all products.

Mr. DODDS: I would like to point out with regard to the adoption of the normal weight method, that any change of this kind could only be effected in a general basis agreement governing the Industry.

Dr. McMARTIN: Mr. Chairman, there is one point which interests me here on page 2, the statement on the fibre of Uba cane: "The fibre of Uba cane is more highly lignified than the fibre of so-called soft canes." Have we any evidence here that of the actual fibre of the cane itself, Uba contains a higher content of lignin than the fibre of any of the other canes? What method was used in the determination of this lignin?

Mr. DODDS: I was relying almost entirely on Dr. McMartin's own demonstration of slides on a microscope in which the lignified fibres had been selectively stained, and there was obviously a much larger proportion of stained material in the Uba than in the P.O.J. 2878, as I think the soft cane was. That was certainly the impression I gained at the time, and I think it was confirmed in a report by Dr. McMartin supplied for the information of the Soft Canes Committee about a year ago. The test is, of course, only qualitative but is very characteristic.

Dr. McMARTIN: It is quite true that I suspect that the fibre of Uba cane is actually harder. but I wondered whether any quantitative work had been done on it.

Mr. DODDS: I don't know of any quantative work in support of the statement.

Dr. McMARTIN: I was only basing the relative lignin content upon the depth of colour produced by the staining reactions. Certainly it is the case that with Uba we get a much deeper staining reaction.

Dr. HEDLEY: Last year Mr. Hayes and I published an analysis of P.O.J. and Co. cane fibre, and the lignin results, which have just been shown to me, were between 20 and 22 per cent. all the way through—and 22.05 for Uba, which is the highest except for P.O.J. 2878. The lowest were the two Co. canes, 281 and 290.

Mr. DODDS: The method of quantitative determination of lignin are generally admitted to be unsatisfactory, and as far as I know, that is still the case. Perhaps Dr. Hedley would remind us of the method he used.

Mr. HAYES: The method used by us for the determination of lignin was by extraction with 72 per cent. sulphuric acid, which is now largely regarded as being fully as accurate, and far less tedious than the Willstatter method. However, in our first analyses results were not consistent. It was found that the proportion of lignin indicated by extraction with 72% sulphuric acid was very largely a function of the temperature. When the extraction temperature was below 20 degrees C.. and kept fairly constant. the results were quite reliable and always bore repetition.

The PRESIDENT. Gentlemen, that concludes the discussion on this Report on Standardization. We are all very much indebted to the members of this Committee for their investigations and the drafting of their Report. and Mr. Dodds for kindly reading it. I wish you to accord him a vote of thanks in the usual way.