

Enquiry on the Presence and Control of Sulphur Dioxide in Natal Raw Sugars for Export.

The following paper was read by Mr. G. C. DYMOND:—

A sub-committee of the Technologists' Association, consisting of Messrs. G. C. Dymond, L. F. de Froberville, and J. Pougnet, made an exhaustive enquiry on the presence and control of sulphur dioxide in Natal raw sugar, and drafted a report which has been submitted to the Committee on Standardization of Chemical Control of the Technologists' Association for the consideration of a larger and more representative body of chemists and sugar manufacturers.

This committee in the main confirms the views expressed by the sub-committee, but has augmented the information contained in the original document.

Control of Sulphur Dioxide in Export Sugar by Selection:

The committee is well aware of the seriousness of allowing sugar of high sulphur content to be exported. It has been shown to them that unless drastic steps are taken to control this impurity, it may later even constitute a menace to the whole sugar industry.

The methods previously employed in the despatching of sugar from the factories to the shipside for exporting have not permitted any care being taken as to the suitability of the sugar for the British market. Instructions have been issued to the factories from time to time to send their total output for a certain period to the Point, and this had to be done irrespective of the quality of the sugar. The committee feel it is their duty to repeat the view expressed on them that not only must the average sugar be well within the limits set by the British refiners, but that every single lot should have such a guarantee.

Although this committee is confident that the production of sugar along the lines recommended in this report will have the desired effect, it is felt that much can be done by proper selection of the sugars exported. It is realised that to carry out this selection with any degree of efficiency, it will be necessary for the industry to provide large storage for sugar at the wharfside. This will, of course,

entail heavy expense, but the opinion of this committee is that before a complete solution of the problem of sulphur dioxide in export sugars is arrived at, some such arrangement will be necessary. If we are to compete with other countries, who do not require to use sulphur in their manufacture, and if the presence of sulphur dioxide in our sugars is a serious obstacle in its saleability, then drastic measures are necessary to keep the sulphur dioxide content at a minimum.

With storage at the wharfside available, the following schemes may be carried out: All sugar leaving the factories and intended for export could be tested to determine the sulphur dioxide content. Any sugar found to contain more than a specified limit will then be diverted to the local refinery. Under these circumstances, it will be possible and may be necessary for some factories to select sugars from first massecuites for export. After the standardized methods of manufacture recommended herein have obtained a decreased sulphur dioxide content in the sugar produced, and rendered it fairly uniform throughout the industry, this method of selection will complete the process and allow only the very best to go overseas. Under this scheme the existing arrangements for the production of their quota of export sugar by each factory or company will not be interfered with.

As an alternative, the production of sugar for export could be confined to several factories where the conditions enable them to produce sugars with the necessary low content of sulphur dioxide. This procedure would be necessary in the event of certain factories (such as white sugar factories) finding that they are unable to apply the recommendations contained in this report, or to reduce the sulphur dioxide content to below the specified minimum.

Control of Sulphur Dioxide Sugar during Manufacture:

From data obtained at the Refinery, the sulphur dioxide contents of raw sugars varied between 27 parts per million recorded in sugars received from Tinley Manor, to 336 parts per million in sugars from Chaka's Kraal.

Very little practical work appears to have been done on investigating the causes of this impurity last crop, while the lack of uniformity in the methods of analysis used, together with the haphazard nature of the samples taken, make a full investigation of the past 1928 crop extremely difficult.

As a result of the enquiry, it is the considered opinion of this committee that the sulphur dioxide content of our raw sugars can be controlled within the limits of the figure (70 parts per million) laid down by the Food Regulation Act of Great Britain. This can, however, only be effected by the complete understanding of the factors influencing the sulphur dioxide content of our sugars and by the efficient carrying out of the recommendations enumerated in this report.

THE PRIMARY CAUSE OF HIGH SULPHUR DIOXIDE CONTENT IN RAW SUGAR IS IMPERFECT CLARIFICATION.

The sulphite in raw sugar may exist in two forms, soluble and insoluble. At the Refinery, it was clearly shown that the sulphur dioxide in raw sugar exists mainly in an insoluble form. Raw sugar first washed to remove gross impurities showed 58 parts of sulphur dioxide per million, but after passing the Vallez filters this quantity was reduced to 20 parts per million.

Further, it was also shown that a grey mill-white sugar from Gledhow contained 200 parts of sulphur dioxide per million, and at Tinley Manor some years ago a white sugar was complained of by Cape sweet manufacturers, which on investigation was proved to contain a high percentage of lime salts including calcium sulphite. Both these conditions were traced back to faulty clarification.

From the above and other experiences, it is beyond doubt that the quantity of sulphur dioxide in the raw sugar manufactured is largely dependent on the clarity of the syrup used in its production.

It was at first thought that the sulphite content of raw sugar would depend on the polarization, but the following figures supplied by the Refinery show that there is no relation between the polarization and the sulphur dioxide content of raw sugars.

Mill	SO ₂ in parts per million			SO ₂ in parts per million		
	Pol.	Ash.	Nett.	Pol.	Solids.	Pol.
Tinley Manor	27	98.17	.32	96.45	36	97.40
Darnall	97	97.57	.42	95.22		
Amatikulu	57	97.65	.40	95.48	96	97.23
Felixton	57	97.55	.50	94.84	38	96.72
Delville	56	97.55	.41	95.34		
Empangeni	62	97.73	.42	95.46	40	97.71
Tongaat	101	98.04	.37	95.80	105	97.75
Verulam	62	97.45	.37	95.29	59	97.19
New Guelderland	113	97.60	.47	95.14	65	97.13
C. Platt	59	97.14	.58	94.02		
Umfolozi	31	97.79	.46	95.48		98.20
Melville	50	95.74	.91	91.26		
Chaka's Kraal	336	95.80	.65	92.18		

The committee nevertheless are of the opinion that a high polarization of raw sugars for export should be maintained.

The Causes of Imperfect Clarification:

The causes of imperfect clarification were then investigated and can be tabulated as follows:—

- (1) The quality of the juice, especially as regards its
 - (a) Phosphoric content.
 - (b) Colloid content.
- (2) A standard method of clarification known to keep soluble and insoluble sulphites in the syrup at a minimum is not in general use.
- (3) In some factories there is an insufficiency of equipment and settling capacities.
- (4) Chemical control in some factories is entirely absent and in others insufficient.

All these factors are partly or wholly controllable.

(1) (a) The question of phosphate content of juice in the process of clarification has occasioned a great amount of study in other countries, especially Hawaii, where it is the general opinion that a content of 0.035 per cent. P₂O₅ is required for efficient clarification and production of a raw sugar free from suspended matter. This theory has to some extent been substantiated in this investigation. Tests carried out at the Experiment Station show great variation in natural phosphate content of cane juices from different localities in this country. Cane juices from the Umfolozi Flats, for instance, contain on the average about 0.07 per cent. P₂O₅ while in other localities the test showed values as low as 0.003 per cent. P₂O₅. This may be a possible explanation why the Umfolozi Factory using the smallest quantity of chemicals should have the sulphur dioxide content of their sugar only slightly higher than Tinley Manor Factory which used the largest amount of chemicals in its production. At Tinley Manor Factory, however, the naturally low phosphate content of the juice was augmented by the addition of a large quantity of phosphoric acid during clarification. There is a possibility that the presence of phosphates in the juice also reduces the solubility of calcium sulphite as does the presence of calcium sulphate (Farnell), but this has still to be investigated.

(1) (b) The influence of colloids is one of importance as this factor governs many local conditions. To some extent this question is bound up with the phosphate content as it is undisputed that the presence of phosphates is influential in the removal of colloids.

The presence of colloids alters the form of the precipitated calcium sulphite from crossed needles to spherical granules, thereby retarding the precipitation and consequent elimination of the calcium sulphite (Farnell). The presence of the phosphates neutralises this effect to some extent.

(2) Large variations were found in the amounts of the various chemicals used in the process of clarification. The following table shows these large differences:—

	Maximum	Minimum
Lime: lbs. used per ton of Sugar	96.18 (Melville)	37.7 (Esperanza)
Sulphur: lbs. used per ton of sugar	40 (Gledhow)	4.29 (Umfolozi)
Phosphoric acid: lbs. used per ton of sugar	14 (Tinley Mnr.)	0.09 (Chaka's Kraal)
Sulphur dioxide: parts per million in mixed juice	3350 (Tinley Mnr.)	700 (Umfolozi)

(3) The enquiry further revealed great variations in the clarification practice and equipment at the various factories, especially in regard to the settling of clear juice, syrup and molasses and filtering area. It is a notable fact that in the 23 factories visited by the sub-committee, not one sugar factory works in the same way as any other, whilst the quality of the sugar produced is largely influenced by lack of suitable capacities and equipment. For instance, the settling capacity for clear juice varied from 1,058 gallons per ton of cane per hour (Melville) to 222 gallons per ton per hour (Prospecton). The minimum time allowed for the settling of the clarified juice was 45 minutes (New Guelderland), with a maximum time of 4—6 hours (Melville), while in the case of syrup, the settling time varied from 36 hours (Beneva) to nil (Maidstone and Tinley Manor).

(4) In the course of the enquiry, the sub-committee found that in at least three factories, no chemical control exists at all, while many others are seriously handicapped by inadequate laboratory equipment and staff. And it is noteworthy that although the question of sulphur dioxide in raw sugar arose during the 1927 season, the enquiry revealed that very little practical work had been done on the investigation of this problem since that time. Efficient chemical control is absolutely necessary to maintain the sulphur dioxide content of raw sugar at a constant minimum.

It is the opinion of the committee that to keep the sulphur dioxide in the raw sugar at a minimum, it will be necessary to incorporate the following tests in the existing control:—

(1) Sulphur dioxide determinations in the sulphited raw juice, clarified juice, syrup and sugar.

(2) Ash control, using conductivity ash measurements.

(3) Lime determinations, by soap test.

These figures as well as being of direct practical value will have a further statistical value in any further investigation.

Control of Soluble Sulphites:

As shown above, a certain proportion of the sulphites in sugar exist in a soluble form. The working of juices acid would cause an increase in the soluble sulphites by the formation of soluble calcium bisulphite, which hinders the normal elimination of the sulphur dioxide as calcium sulphite. It has been established that the high sulphur dioxide contents recorded at Chaka's Kraal were obtained under such conditions.

Experiments recently conducted by G. C. Dymond confirm the view expressed by Farnell and other authorities that soluble sulphites may be eliminated by increasing the amount of chemicals used in clarification. In these experiments, quantities of juice were treated with increasing amounts of lime, the juice sulphited to a pH of 7.5 with the addition of a little phosphoric acid, heated to boiling and the clear juice settled and tested for sulphur dioxide. The following results were obtained:—

Pounds of Lime added per ton of Cane.	Sulphur Dioxide in parts per million.
9.9	180
23.5	100
42.4	30

Control of Insoluble Sulphites:

A properly controlled clarification will yield a clear juice free from suspended calcium sulphite, which on evaporation should yield a syrup containing very little suspended matter.

It is probable that the evaporation will precipitate more calcium sulphite. To remove this the syrup should be heated and settled, and the clear high density syrup so obtained should give a sugar low in sulphur dioxide content.

In this connection it should be noted that no settlement of the syrup was practised at Tinley Manor last season, and the sulphur dioxide content of their sugar was one of the lowest recorded. It is their contention that with good clarification using large quantities of chemicals, there is no need for such treatment. Nevertheless, the opinion is expressed by the committee that had the syrup been settled at Tinley Manor, still better results would have been obtained.

The following figures demonstrate how the sulphur dioxide content increases in the lower grade sugars, which would make molasses treatment necessary:—

Maidstone.

1st sugars	70 parts per million.
2nd sugars	90 " "
3rd sugars	175 " "
Treacle sugars	200 " "

New Guelderland—

1st sugars	95	parts	per	million.
2nd sugars	120	"	"	
3rd sugars	300	"	"	

It is, however, the opinion of many chemists that the dilution and storage of molasses is not in the interests of the sugar manufacturer, and they further contend that if the clarification up to and including syrup has been efficiently carried out, no further treatment of lower grade products is necessary. Such contentions can only be proved during the ensuing crop. The committee have nevertheless recorded in their recommendations methods of molasses treatment which should be tested out especially in view of the possibility of the manufacture of export sugar being centralized in a few factories.

TENTATIVE DETAILS OF FACTORY PRACTICE TO KEEP SULPHUR DIOXIDE CONTENT OF RAW SUGARS AT A MINIMUM.

Milling Control:

To ensure a minimum of colloidal substances in the mixed juice, care must be taken to exclude frosted and drought-stricken canes and to see that the canes are properly topped. The milling plant should be kept clean and free from fermentation for the same reason. The use of steam jets and antiseptic sprays for cleaning down is strongly advocated. Fine cush-cush should be removed from the mixed juice as completely as possible by efficient screening, for the action of lime and heat on cush-cush causes the production of colloidal substances.

Treatment of Mixed Juice:

The mixed juice is first heated to a temperature of 140–160° F. and treated with a quantity of lime such that after sulphiting to 2,500 to 3,500 parts per million, the resultant juice is not acid. About 90% of the total lime used for clarification should be used at the first. The final tempering of the juice is conducted in the tempering tanks where lime is added to bring the pH to 8.3 (slight pink phenolphthalein paper) and a final adjustment made with phosphoric acid to a pH 7.4–7.8. The correct degree of phosphate content required by our juices has not yet been properly investigated. In Hawaii, experiments have shown 0.035 per cent. P_2O_5 to be the optimum, but on the other hand Farnell recommends 0.05–0.06 as being necessary for Uba juices. The committee is of opinion that 0.05 per cent. P_2O_5 would be a good figure to work from until more research data is available.

The large quantity of precipitate formed improves the elimination of sulphite as shown by Farnell and confirmed at Tinley Manor last season, and by Dymond as above, Farnell in his work on the pre-

cipitation of calcium sulphite in sugar solutions (Journal of the Society of Chemical Industry, December, 1925), states that:—

“The factors concerned in the precipitation of calcium sulphite are extent of neutralization of sulphurous acid, the time and temperature of precipitation, the initial concentration of the sulphurous acid and calcium hydroxide and the presence or absence of colloids.”

Furthermore, “The higher a cane juice is sulphited the more complete is the precipitation of the calcium sulphite on liming back to a given pH in the hot juice. It is suggested to precipitate calcium sulphite more completely the pH of the hot juice should in many cases be increased from 6.8 to 7.6–7.8.”

The phosphoric acid assists in giving better settlement and better elimination of colloids.

The tempered juice should be heated to about 208° F. (98° C.) and subsided for 1¼–1½ hours. The clarified juice obtained should be clear and bright and the pH will have dropped to about 7.2. This is considered to be the optimum pH for the elimination of calcium sulphite.

Treatment of Scums:

These should be diluted as much as evaporator capacity will permit and may be decanted if the filter press capacity is deficient. The decanted juice may then join the raw juice immediately after the final tempering. On no account should scums be boiled; this enables the colloidal matter to pass through the filter cloth and so return to process.

The bottoms from the decanted scums may be further diluted and treated with lime, mixed, but no heat applied. Phosphoric acid or kieselguhr may also be used with advantage and the material filter-pressed.

The filter press juice should be returned to the raw juice before sulphitation.

Only clear juice from the subsiders should ever be allowed to enter the evaporating system. The practice of mixing limed dilute scum juice with the clear juice entering the evaporator is bad. The mixing of these two different juices will produce a cloudy liquor.

Treatment of Syrup:

The density of the syrup should be kept over 55° Brix in order to precipitate salts as completely as possible.

Any suspended matter in the syrup will form the nucleus for crystals of sugar, and so all suspended calcium sulphite must be removed from the syrup. The temperature should be raised to 80° C. or

higher, but the syrup must not be acid at this point or considerable inversion would take place at this temperature. A suitable degree of alkalinity is pH 7.2—7.3; if the syrup is more alkaline than this, phosphoric acid may be used with advantage to bring it to the optimum of 7.2. This heating of syrup is a common practice in Mauritius, and was done at Umfolozi with excellent results some years ago. Chaka's Kraal also experimented on these lines during the latter part of last crop with favourable results.

The heating of the syrup not only hastens the process of settling, but should also produce greater precipitation of calcium sulphite by virtue of the fact that the solubility of calcium sulphite decreases with increasing temperature.

The bottoms from the syrup are now returned to the raw juice immediately after the final tempering. The reason for this recommendation is that the thin juice being already saturated with calcium sulphite, will not dissolve out any of the sulphites in the syrup bottoms, these therefore being eliminated in the filter presses.

Treatment of Molasses:

As stated above, it is the opinion of many chemists that the dilution and settling of molasses will be unnecessary if good clarification of the juice is effected. If the lower grades of sugar are still high in sulphur dioxide, the following procedure is suggested:—

The molasses is diluted to 50—55° Brix, limed if considered necessary, boiled in blow-ups and subsided. For this practice tanks sufficient for 12 to 18 hours settling are required for first and second molasses.

Treatment of Treacle Sugar:

Although the percentage of this low grade product is usually small, yet its high sulphur dioxide content (190 - 210 parts per million and possibly higher) may raise the proportion of the sulphur dioxide in the raw sugars above the limit if simply mixed away with it.

One sugar factory mixed away an accumulated stock of such sugars during a period of four weeks. The sulphur dioxide content of their raw sugars thereupon rose from the normal 62 parts per million to 110 - 142 parts per million for this period. On the discontinuance of this practice the sugar became normal.

The procedure last crop was either to mix it away with the first magma with the results already noted, or to remelt and subside, or dispose of it in the raw juice or use it as grain in the pans. The committee recommend that the method of remelting, adding a little lime and phosphoric acid, heating to 90° C and subsiding should be tried out. The method of mixing small quantities with the tempered juice before heating is simple and there appears no urgent reason for its discontinuance.

FACTORIES MAKING MILL WHITES AND A PERCENTAGE OF RAW SUGARS FOR EXPORT.

Out of the eight factories which make mill whites at least five made their quota of raw sugars continuously from their lower grade massecuites, the remainder at one period. Certain mill white factories (e.g., Sezela) claimed an advantage in sulphiting the cold raw juice before liming, and made a special point of cooling the gases from the sulphur stove.

Owing to the good clarification generally necessary for white sugar manufacture, more especially in the treatment and subsiding of syrup and molasses, these sugar factories generally showed a fairly low sulphur dioxide content for their raw sugars. Very few figures were, however, available. If those sugar factories making their quota of raw sugar continuously find that they are unable to bring their sulphur dioxide content within the permissible limit, it is recommended that they make their quota during one period.

Methods of Determining Sulphur Dioxide in Raw Sugar:

The methods have not as yet been standardised, the matter being left over until the arrival of Mr. L. Blacklock, who, it is believed, has special knowledge of the methods adopted in Great Britain.

The Chemical Control Committee will circulate all mills with the official methods before the commencement of the 1929 crop.

SUMMARY OF RECOMMENDATIONS TO KEEP THE SULPHUR DIOXIDE CONTENT OF SUGAR FOR EXPORT AT A MINIMUM.

Control of Sulphur Dioxide in Export Sugar by Selection:

Every individual consignment of export sugar must have a guarantee that its sulphur dioxide content is below a specified maximum. To conduct this selection effectively, it will be necessary to provide large storage for sugar at the wharfside.

The existing arrangements for the production of their quota of export sugar by each factory or company may be continued, or the manufacture may be confined to several factories where the conditions enable them to produce sugars with the necessary low content of sulphur dioxide.

Control of Sulphur Dioxide in Export Sugar during Manufacture:

The primary cause of high sulphur dioxide content of raw sugars is poor clarification, the proportion of sulphur dioxide present in sugars being mainly dependent on the clarity of the syrup.

Chemical Control:

It is necessary to incorporate the following tests in the existing control:—

- (1) Sulphur dioxide determinations in the sulphited raw juice, clarified juice, syrup and sugar.
- (2) Ash control, using conductivity ash measurements.
- (3) Lime determinations by soap test.

Milling Control:

Care is necessary to exclude drought stricken canes from the milling plant and to see that canes are properly topped. Efficient screening of cuscusc from the mixed juice is important. The milling plant must be kept clean and free from fermentation. The use of steam jets and antiseptic sprays for cleaning down is strongly advocated.

Treatment of Mixed Juice:

The mixed juice is heated to a temperature of 140-160° F treated with lime so that after sulphiting to 2,500 to 3,500 parts per million, the resultant juice is not acid. About 90 per cent. of the total lime used for clarification should be used at this point. Correct to slight phenolphthalein alkalinity (pH 8.3) and make the final adjustment with phosphoric acid to pH 7.4 to 7.8. Heat up to 208° F and subside for 1¼ to 1½ hours. The clarified juice should be clear and bright and have a pH 7.2. Only clear juice from the subsidors should ever be allowed to enter the evaporating system.

Treatment of Scums:

These should be diluted as much as the evaporator capacity will permit and may be decanted if filter press capacity is deficient. On no account should scums be boiled. The decanted juice may then join the juice immediately after the final tempering.

The bottoms from the decanted scums may be further diluted and treated with lime, but no heat applied. Phosphoric acid or kieselguhr may also be used with advantage and the material then filter pressed.

The filter press juice should be returned to the heated raw juice before sulphitation.

Treatment of the Syrup:

The density of the syrup should be kept over 55° Brix. The temperature should be raised to 80° C, but the syrup must not be acid at this point. A suitable degree of alkalinity is 7.2 - 7.3. pH; if the syrup is more alkaline than this, phosphoric acid may be used with advantage to bring it to the optimum of 7.2.

The syrup bottoms after settling may be returned to the raw juice after final tempering.

Treatment of Molasses:

If the lower grades of sugar are still high in sulphur dioxide, the following procedure is prescribed.

All molasses for reboiling are diluted to 50 - 55° Brix, limed if considered necessary, boiled in blow-ups, skimmed and subsided. For this practice tanks sufficient for 12 to 18 hours settling are required for first and second molasses.

The molasses bottoms should be retreated, settled and the final bottoms discarded.

Treatment of Treacle Sugars:

Treacle sugar may be remelted, treated with a little lime and phosphoric acid, heated to 90° C, subsided and mixed with the clear juice entering the evaporators. Small quantities of the sugar may also be mixed with the tempered juice before heating.

The method of determining sulphur dioxide in raw sugars has not been definitely selected, and further recommendations will be shortly made in this matter.



Mr. BEHRENS: This is a very important matter. I think this could be remedied if we had our mills on the principle of carbonatation; do away with all settling tanks and filter the juice straight away as it comes out of the pre-heaters. We want about 2 filtrations and 2 sulphitations, and in carbonatation you have 3 filters and practically 3 carbonatations. For raw sugar 2 sulphitations and 2 filtrations would be all right, and that would do away with all our trouble. Of course the mills would have to be remodelled. We could work the mills on the principle of carbonatation by sulphitation. This principle I have been preaching about for some years.

Mr. BECHARD: With regard to the matter of selection, it seems to me that to send all our best sugars to Europe and keep our refineries supplied with all our bad sugar is rather a dangerous way to get out of a difficulty. What does Mr. Blacklock think about that? Would not the sugar of high sulphite content sent to our refinery militate against disposal of the refinery treacle?

Mr. BLACKLOCK: The main objection raised by the Home refiners is the accumulation of SO₂ in the run-offs and residual syrups. In most refineries one or more forms of table syrups are produced. If these are made from melts of Natal sugars it is found that the SO₂ content is above the legal limit of 70 parts per million, and they become unsaleable according to law. Another point brought forward is that when the SO₂ content rises above 100 parts per million the taste and flavour of edible syrups is adversely affected. At the local

refineries here we have for years been in the unfortunate position of having to accept and refine anything and everything that the Natal mills choose to send us. English and American refiners hesitate to accept small parcels of Natal sugars on account of quality, filtrability, sulphur dioxide content, etc. As Mr. Bechard points out—our position with regard to SO₂ is not going to improve much if the lower sulphur sugars are selectively exported. We have at times ascribed certain difficulties in our char-working to the presence of S.O.₂, and sulphides and sulphur compounds are finally in great measure the reason of the objectionable nature of sugar factory effluents here. A determined effort should be made in future to minimise and control the presence of this troublesome constituent in Natal sugars.

SECRETARY: Might I just mention that as from the 1st of this month the Food and Drugs Act is in force in South Africa and is exactly on all fours in this respect with the English Act, and the limit of sulphur dioxide is 70 parts to the million. They have adopted the same figure.

CHAIRMAN: It seems that limit is hardly fair when applied to sugar. The reason for the very strict limitation of the SO₂ in foodstuffs is because in many cases it is used as a preservative, and therefore its presence except in very small quantities must naturally be regarded with extreme suspicion. But that is not the case in sugar where, as we know, it is merely the residue of manufacture and has nothing to do with the preservation of the substance after it has been formed. So the law as it exists is rather unfair on sugar. I suppose it is hardly possible to get a variation of that now.

Mr. MOBERLY: In that connection a series of experiments was carried out in Louisiana to see the effect of sulphur in syrup and treacles for human consumption. An experiment was tried by selecting a number of negro convicts and feeding them for several months on a diet which consisted largely of sulphured syrups, and the reports were that these convicts all thrived on the diet, and, like Oliver Twist, "asked for more."

Mr. H. M. JACOBS: It would be as well to let Mr. Bechard know that the idea of isolating the better quality sugars for sending overseas was not suggested by the Committee. It was suggested by the Refinery Management; with 40 per cent. of the crop to be exported there was a danger of us losing the overseas markets, and the Refinery authorities thought that was the best way out of it.

Mr. DYMOND: You have had this report for a whole year; surely some of you have had time to try out some of our contentions, especially with regard to the treatment of syrups which we so strongly recommended. Has anybody tried out the method of heating and subsiding syrup, and can

Mr. Blacklock tell us if there has been any general improvement in the SO₂ content of our raw sugars this year compared with previous years?

Mr. BLACKLOCK: I can report very favourably on that. The standard has been improved, and up to now there has been no complaint from Home to my knowledge.

SECRETARY: There have been no sensational complaints yet.

CHAIRMAN: I would like to add to that. As you would note from Mr. Coghill's paper there are a number of factories which have been sending samples regularly to the Experiment Station, and on the whole they have been very low in SO₂. There are one or two conspicuous exceptions, and I have sometimes wondered what would have happened to those sugars if they had been exported. One the whole the percentage has been well below the limit permitted.

Dr. HEDLEY: I think the Committee responsible for drawing up this report should be congratulated, as it is a very important report. It took them some time to go and see these factories, and the way the data has been collected and set forth reflects great credit on the Committee, and they certainly did very good service to the Sugar Industry in the work which they carried out last year. (Hear, hear, and loud applause.) With regard to the cooling of the gases from the sulphur furnace so that solution may be facilitated, I am sure that such cooling is useless, and for two reasons. Firstly, the specific heat of sulphur dioxide is very low as compared with that of the juices, i.e., a ratio of 0.1 to 1. Secondly, the volume of the SO₂ gases, which absorb the gas, one only gains 3 mg. per litre, that is 3 parts per million. Is it to be expected that such a volume will affect the temperature of the juices, even granted that the actual volume of gases passing through the juices is ten times that of the sulphur dioxide? More than a 10 per cent. by volume of SO₂ in burner gases is very difficult to obtain.

CHAIRMAN: I would like to endorse Mr. Dymond's appeal for comments, particularly in regard to this last season's work. All the factories have been working with the benefit of this report in front of them, and it would be interesting to have their comments in the light of that.

Mr. JACOBS: I would like to draw attention to that part of the report which says that the percentage of SO₂ in sugar depends on the clarity of the syrup. I think that gives a wrong impression. I maintain that with bad clarification in the first place no amount of trying to work the syrup resulting from that juice will ever help the position to any appreciable extent. I maintain the main place

where we can eliminate SO₂ from our raw sugars is in the juice preparing tanks, not by any treatment of the syrup later on. At Tinley Manor, as Mr. Dymond points out, in the season before last no settling was practised, and yet about the lowest SO₂ in the country was recorded at this factory. I followed out exactly the same method of clarification last year at Felixton and find from Mr. Coghill's report that Felixton has also a comparatively low SO₂. The point where we tried to eliminate SO₂ is right at the very beginning in the preparation of the mixed juice. After that we don't worry about it. We do not treat the syrup; it is not settled before going to the pans, nor is the molasses settled, and the SO₂ is low. It is not only the clarity of the syrup, but more the efficiency of the clarification before evaporation that is required.

Mr. BEHRENS: I think the SO₂ would be greatly reduced if the mills were to work through with a more pure juice. The crystallization would be more perfect, and the SO₂ more liable to be washed away. The SO₂ sticks mostly to the impurities. If we don't get the impurities out we don't get the SO₂ out.

SECRETARY: Might I just say that Mr. Dymond's request for some comments on the work that has been doing during the past year in view of this report being in the hands of the millers during the season, is very much worth while. After all this is a commercial question of very great importance. You see that with export sugar the limit is 70 parts per million, and you sell your sugar on that basis. If the SO₂ content is substantially over that you don't know what you are going to get for your sugar. You pretty well get what the buyer likes to give you, and very serious losses can be made, so serious that the Association took such notice of it that they agreed to the formation of this Committee and to the issue of this report. It is on that account—the commercial aspect—that this becomes not merely a matter of technical interest but of much broader and wider interests, and it ought to be impressed in the strongest way on mill managements that this particular phase of manufacture ought to get the greatest possible attention. I thought it worth while making this remark more or less to emphasise the importance of the request by the Chairman and Mr. Dymond for remarks from managers and chemists as to what happened during last year since the report has been in the hands of the managements.

Mr. JACOBS: I think Mr. Dymond himself has made a good many tests this year.

Mr. DYMOND: Mr. Jacobs remarked on the non-settling of syrups. You will notice in this report that the Committee stress good clarification, and we also stated that the clarity of the syrup was a predominating factor, and further expressed the

opinion that had the syrup been settled at Tinley Manor the results would have been better still. I am still firmly of that opinion. Certain experiments were carried out last year and various tanks of syrup were kept and tested for SO₂ at different periods. Thus from a number of investigations the syrup on sampling contained 350 parts per million. On settling for eight hours it contained 208; after further eight hours it contained 260. The top contained 200 and the bottom 768, showing that the longer the syrup is settled the lower the SO₂ in the supernatant liquid. If the syrup is filtered a very low SO₂ content is obtained, but at present this is not a practical solution of the problem. Further it was not practical to allow syrup to settle in the factory for 20 to 30 hours in order to get the same result. The Mauritian practice of heating syrups is still recommended for trial so as to cut down the time factor within reasonable limits and so lower the SO₂ content. This point has been definitely proved by work carried out last year. Unfortunately there is nobody from Chakas Kraal or Umfolosi here who could tell us more about the heating of syrup. It is also a pity that nobody else has tried this method out during the last year. Laboratory experiments carried out involved the same principle, such as addition of a filtering medium, and it was definitely established that by adding a filtering medium to the syrup, whether it be lime, phosphoric, or kieselguhr, or filtering by mechanical means on every occasion the SO₂ content dropped very considerably. In fact after application of a very large quantity of lime and phosphoric a syrup of an original 464 parts of SO₂ was reduced to 20 parts, which is a very low figure for syrup.

Mr. BIJOUX stated that he thought inquiry should be instituted to ascertain whether a lesser quantity of chemical could be used in the manufacturing process to attain the same results.

Mr. DYMOND: During the whole of the last crop at Empangeni we carried out regular P₂O₅ tests in our mixed juices, and found the amount varied from .001 to .003%. On the flats, as stated in this report, it goes up as high as .07%. There is no doubt that the phosphoric content of juices is a big factor in the SO₂ content in the resulting sugars, because the quality of the clarification in defecation is largely dependent on the original P₂O₅ content of the juice, or the amount added to bring it to a certain standard. In Hawaii they contend that .03 is a sufficient amount to produce an efficient clarification. Out here Farnell reckoned it should be between .05 and .06%, while the Committee agreed that it should be at least .05%. It is my personal opinion that the extraordinarily low SO₂ contents recorded at Umfolosi, despite the small amount of their chemical bill, is due to the naturally high P₂O₅ content of their juices. Mr. Rault, of Mount Edgecombe, kindly gave us a large number of P₂O₅ results which he had obtained from Um-

folosi canes. If there is no natural high P_2O_5 content in juices from a localised area, then the amount must be artificially increased to .05%.

Mr. BEHRENS: It would be greatly improved if a little more care were taken in the alkinity of the juice all through. It should never be worked below .03 or .04. The concentrated juice might go up higher, but in my opinion we should never work with sour juice. Keep the lime up in the juice and you diminish your SO_2 .

Mr. DYMOND: That is a point that we have emphasised in this report—the alkinity should be maintained in the clarified juice at from 7.2 to 7.3 pH. The high SO_2 content at Chakas Kraal the previous year was undoubtedly due to acid juice, and I would stress the point that all juices should be kept at a pH of between 7.2 and 7.3.

Mr. COGHILL: What method was adopted?

Mr. DYMOND: The ordinary colorimetric or spot plate method.

At 5.25 p.m. the Congress adjourned to next day.

SECOND DAY.

At 9 a.m., on Wednesday, April 16th, the Chemist Members of the Association met to discuss the question of the registration of Chemists. The proceedings of this meeting were not recorded, but the following resolution was passed:

“That this Association prepare a Register of Chemists in the Industry, and that a Committee be appointed to meet representatives of the S.A. Chemical Institute with a view to obtaining advice from that body in drawing up a standard of qualification.”

The following Committee was appointed for the purpose:—The Chairman, Mr. Dodds and Mr. Bechard.

At 10 a.m. the Conference resumed the adjourned discussion on the report of the Sulphur Dioxide Commission.

Mr. BECHARD: Yesterday the question of economy in chemicals was raised. We have got to the point where we have to fight for the pennies in the Sugar Industry. This increased quantity of chemicals goes on every year; although it seems to have been very useful in increasing the recovery, no doubt it costs a lot of money. We must remember that settling of the syrup has also a lot to do with it. The Chairman of the Chemical Section tells us that everything must be done at the beginning. The mixed juice is certainly the most con-

venient place to do it, but still the question remains, and some study ought to be given to it—is it the most economic place to dispose of the SO_2 ?

Mr. JACOBS: I don't think I expressed myself very well in the remarks Mr. Bechard has referred to. I don't say settling of syrup is not of benefit, but I do say that unless a drastic attempt is made to treat the raw juice in such manner as will result in low SO_2 in clarified juice, no amount of settling the syrup or later products will ever materially help.

Mr. BIJOUX: By increasing the amount of phosphoric acid in the juice, it would be better.

CHAIRMAN: Phosphoric acid is an expensive ingredient.

Mr. BECHARD: I can tell you that when I was making white sugar I found it very beneficial to add phosphoric acid to the syrup. I know we got brilliancy, and brilliancy of syrup to my mind is a very good guide to the quality of sugar we will eventually obtain.

Dr. HEDLEY: I would like to endorse what Mr. Jacobs says. I have seen bad clarification give a hundred parts of sulphur dioxide per million, and then I have seen that clarification so improved and nothing else done—no settling or anything like that—that it has brought it completely down to between 35 and 55 parts per million, which is exactly what he says. Bad treating gives you bad results, and good treatment gives you perfectly satisfactory results which remain right through the whole factory. While I am on my feet I would like to propose that we extend to those who took the trouble to do this report the most hearty vote of thanks, because I think it is one of the most masterly reports which we have had since this Association began. We certainly ought to vote to these people a very hearty vote of thanks for the trouble and energy they took and put into it. I did not know yesterday that the Committee had also revised this report, but the way it has been put forward, the clarity of its expression, and the assistance which the whole industry gave to it, have lent to the making of an extremely masterful report. (Loud applause.)

CHAIRMAN: I certainly would like to endorse Dr. Hedley's proposal. The report, as he says, is an excellent one of its kind, and it makes one almost wish that similar special difficulties would occur in other branches of manufacture so that we might have such a flood of light thrown on those other difficult aspects also.

Mr. JACOBS: I would also like to endorse the proposal. At the same time I would like to express disappointment that we have contented ourselves with but slight criticism of the report, and there has been no further light thrown on the subject,

showing that the report, although written a year ago, has not had the support of the Chemists in this Industry that it should have had. When you think that it is a year ago that a very large proportion of the sugar made in this country was in a grave condition—so much so that the sale of our export sugar was endangered, and the Millers instituted this Committee to investigate the position—I think it was up to the Chemists of this country during the last season to have studied that report and got more light on the subject. I would like to suggest that the Association institute another Committee to report on the same subject at the next Conference.

Dr. HEDLEY: May I add that I think Mr. Jacobs has not much cause for disappointment. There has been a vast improvement as Mr. Blacklock has said in the SO₂ of sugars. There has been no trouble yet as Mr. Eadie says. At any rate we had not the trouble we had last year. It is now in the hands of the Association Chemists; it went to the Managers but not to all the Chemists, that was one reason why we have not had the practical opportunity of examining it.

Mr. JACOBS: What I might say is that although we have not had complaints about SO₂ during this season, that does not mean that we are unlikely to be faced with the same difficulty again. Mr. Blacklock will bear me out when I say that the Sugar Refiners overseas even propose reducing the standard to somewhere about 30 parts per million, and if we are faced with that problem again, then the position will be grave. I further say it is no use waiting until this happens before we get busy.

Mr. BIJOUX: I agree, but you must not think we are lazy about this question. We have all been trying to do something and we are experimenting to get the amount down. We think that in a year or two's time we will be able to see something done in the way of reducing it.

SECRETARY: On the question of circulation, I think that ought to be put right. Now that the paper is an official paper of the Association every member will get a copy of it.

Mr. DYMOND: While endorsing the remarks passed by Dr. Hedley and Mr. Jacobs, I don't think it is entirely the Chemists' fault that more work has not been done on the subject, especially in a practical way. Frequently the Chemist is handicapped by not being able to carry out practical experiments involving possible extra machinery in the factory, and I think the Managers of our mills are more to blame than the Chemists in this respect, especially as regards certain points we brought up such as the heating of syrup which the Chemists would like to try out, but have not the facilities to do it. If the heads were to take a more serious view and instruct the Chemists to carry out these

experiments the Chemists would be only too pleased to do so. (Applause.) I would further like to make the proposal that a resolution be brought to the Millers' notice on the lines of which I have spoken, that they take more interest in this subject and institute experiments in these directions.

Dr. HEDLEY: I would like to second that. I am quite certain if it came from this body it would be listened to, particularly if attention were drawn to the likelihood of still more rigid regulations, as Mr. Jacobs points out, being laid down. I think the Millers will allow it is in their own interests to institute further experiments.

Proposed by Mr. Dymond, seconded by Dr. Hedley, that the matter be brought to the notice of the Millers' Association.

Mr. BECHARD: On the question of cooling sulphur gases I asked Mr. Dymond if he could throw any light on the reason for stressing the necessity of cooling sulphur gases before admission of the juice. He said perhaps Mr. Pougnet would be able to do that.

Mr. J. POUGNET: We tried it at Sezela, working it both ways, but we always found it much better with cooling.

Mr. BECHARD: Would Mr. Pougnet put that effect down to better absorption of sulphide dioxide gas or to possible inversion where the point of contact is, or again to sublimation of the sulphur in the juice forming colloidal sulphur which would be difficult to eliminate?

Mr. J. POUGNET: I reckoned that cooling of the gases condenses or deposits on the cold surfaces of coolers, fine particles of sulphur. Therefore a purer gas free of solid particles, of lower temperature, is put in contact with the juice, and a better absorption takes place. The sulphur which reaches the juice is due to several causes: (1) Ovens working too hot; (2) Sulphur used being more or less in a powdered state. This is either (a) drawn into the gas by vacuum created by ejectors, or (b) forced in by compressors. Whatever is the cause of its presence, the result is bad, whether one sulphites hot or cold juice. With coolers running short of water we have seen steam ejectors at the top of tall towers (a good way from the ovens) choked with sulphur. If juice comes out of the towers containing that very fine sulphur dust, on being tempered (i.e., lime and P₂O₅ added) and boiled, it affects the clarity of the juice, to say nothing of the increase in scales. In my opinion using a limestone full of magnesia (MgO) is not worse than using hot SO₂ gas, both containing impurities which are detrimental. At Sezela where we cool the SO₂ gas to a great extent we have noticed that 70 per cent. at least of our trouble with the SO₂ in juice can be traced back to the coolers when short of cold water.

Mr. JACOBS: There is a very practical advantage in cooling the sulphur gases. In the process of manufacture in this country the heated raw juice is limed before it is sulphited. When the hot sulphurous acid gas meets the juice there is immediately a precipitation of calcium sulphite which takes place in the sulphur tower. This usually results in serious scaling in the lower trays of the tower, and by the end of the week, unless the gas is cooled, the trays are scaled up in such a way that there is a danger of the juice passages being choked.

Mr. J. POUCKET: I suggest that we should endeavour to ascertain which is the best sulphur plant to use. Having had the opportunity of visiting all the sugar factories of Natal and Zululand in 1929, I paid special attention to the sulphur plants in use, which are different in each factory. Just as a crushing plant consists of three main parts, i.e., engine, gearings, and mills, a sulphur plant consists of (1) ovens, (2) SO₂ pipes, and (3) towers or boxes. If the towers were more or less alike everywhere, the ovens were of all shapes and

sizes. No doubt some are better than others. As regards cooling the gases, very little of it was seen apart from Sezela. Is it generally believed to be useless? or for what reason we could not say. There is no doubt that a lot of improvements can be made on the sulphur plants (ovens, SO₂ pipes and towers). As far as the mixing of the juice and gas are concerned without loss of gas into the air, the Quarez is the best I know, having had personal experience with it. There was one thing we noticed on the South Coast; if we put the lime in the cold juice before the sulphur it made the work worse owing to the presence of leuconostoc. I do not think all places are affected to the same extent. Last year we did not have very much, but the year before we had a lot of trouble. Another source of trouble is cane gums, of which very little are known. The farther South you go the more the conditions are changed.

A hearty vote of thanks was passed to the Committee.