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THE INTERNATIONAL SUGAR JOURNAL

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NOTES AND COMMENTS

Commonwealth Sugar Agreement.

This year's talks between the parties to the Commonwealth Sugar Agreement which began on 8th November ended on the 16th.

The agreement has been extended for a further year and will now run until 31st December 1974. Negotiated Price Quotas for 1967 remain the same as for this year, as follows: Australia 335,00 long tons, British Honduras 20,500, East Africa (Kenya, Tanzania, Uganda) 7000, Fiji 140,000, India 25,000, Mauritius 380,000, Swaziland 85,000 and West Indies and Guyana 725,000 long tons.

The Negotiated Price for the three years 1966, 1967, 1968 was determined at the 1965 talks, i.e. £43 10s 0d a long ton f.o.b. and stowed bulk 96°, with, for the less developed Exporting Territories in the Commonwealth Sugar Agreement, a special payment, calculated annually, consisting of a fixed element of £1 10s 0d and a variable element ranging from £2 10s 0d to nil.

The parties to the Commonwealth Sugar Agreement noted that the price of sugar on the world market had fallen to its lowest level since the war. They re-affirmed their belief that the present situation demanded an early and effective International Sugar Agreement requiring undertakings and restraint by both importers and exporters, and their determination to work for such an Agreement.

* * *

International Sugar Council.

The 23rd Session of the International Sugar Council was held at the seat of the Council in London on the 2nd and 3rd November 1966. The Session was presided over by Sir ROBERT KIRKWOOD (Jamaica) and was attended by representatives of 39 countries as well as by governmental observers from Bolivia and Guyana and observers from the F.A.O. and the E.E.C.

The Council noted the favourable response of member Governments to a prolongation beyond 31st December 1966 of the International Sugar Agreement of 1958 as extended under the 1963 and 1965 Protocols and agreed to request the Secretary General of UNCTAD to arrange with the Government of the

United Kingdom of Great Britain and Northern Ireland, the depository power, for a new Protocol to be opened for signature as from 14th November 1966. The 1958 Agreement will be extended under the new Protocol until 31st December 1968 unless a new International Sugar Agreement enters into force before that date.

The Council considered the first estimate by its Statistical Committee of the supply and demand position for 1967. It adopted the Committee's estimate of the minimum net import requirements of the world market of 15.7 million metric tons, raw value (details of which appear on page 383). The Council was aware that this first estimate, as has been the case in the past, was conservative and that the relatively low level of requirements was largely due to a favourable crop in most sugar beet producing countries. In comparing this estimate with the last estimate for 1966 (15.98 million metric tons), the Council was also aware of the important difference between the two estimates, as, because of the lack of information, the estimate for 1967 did not include any estimate for Mainland China (the figure included for that country in the 1966 estimate was 400,000 tons). As regards supplies in 1967, the Council noted that these would be more than sufficient to meet the estimated requirements.

The Council received an oral report on the activities of the Preparatory Working Group on Sugar appointed by the Secretary General of UNCTAD to examine and report on the general content of a comprehensive long-term International Sugar Agreement. The Council noted that the next series of meetings of the Working Group was to begin in Geneva on 21st November 1966. The Council stressed its vital concern in this matter and expressed the hope that the activities of the Working Group would soon establish the foundation on which fruitful and early negotiations could be initiated.

¶ In view of the formal position arising out of the expiration of the present Protocol at the end of this year, the Council agreed to defer the election of its Chairman and Vice-Chairman, as well as the appointment of its Committees, for 1967 to the first Session of the Council under the new Protocol.

International Sugar Agreement prospects.

As mentioned in the report of the 23rd Meeting of the International Sugar Council, the next series of meetings of the UNCTAD Working Party on the possibility of the re-convening of a negotiating conference for a new International Sugar Agreement—a conference which must be sure in advance of having a fair prospect of success—was to start in Geneva on the 21st November. The German sugar economist, Dr. H. AHLFELD, recently discussed the efforts to reach a new Agreement and his views might be quoted in an attempt to show the way in which short-sightedness has brought the world sugar market to its present depressed state.

Dr. AHLFELD writes¹: "Again and again sugar production exceeds consumption requirements which are increasing by about 2—2½ million tons annually. As a result of this absurd production policy, large surplus stocks are arising which show every buyer that he will always get the necessary sugar at ruinous prices". Cuba's production policy is considered as an example of the planning of several countries: "According to the plans of the Cuban Government, 7.5 million tons are to be produced in 1967 and even 10 million tons in 1970. If one hears such production figures one has to ask the question: who is to eat these huge quantities? It seems that the 'planners' do not trouble their heads about this question". . .

"The International Sugar Agreement of 1937 . . . has several times been renewed after World War II and is still in existence today. The main provisions of this Agreement are, however, suspended. International cooperation in the sugar sector has had, up to now, not much success. One of the main reasons for this development was the fact that the export quotas requested and mostly also received by the exporting countries were too high to make the agreement effective. The delegations went home fully convinced that they had worked to the advantage of their country by having received a large export quota. In fact, however, this policy has made the Agreement useless from the very first, all the more as a result of the failure to grant powers to the International Sugar Council to adapt the too-high exporting quotas by corresponding cuts to the actual outlets". And so, of course, by lack of such regulation of production these same exporting countries are now losing vast sums because their return is only a fraction of the cost of producing their sugar.

Dr. AHLFELD continues: "In the agreements of 1927, 1931 and 1937 there was no political antagonism. Even in the 1953 and 1958 agreement the East-West conflict was of no decisive importance. However, since Cuba has turned to the East block, the situation has considerably changed. Now, Cuba as the main sugar exporting country and the United States as the main consuming country, and one of the most important sugar importing countries, are

politically in opposition, which has an influence on the sugar sector and on all international sugar negotiations. In such a situation special questions are often relegated to the background by political aims. Therefore, solutions which should be possible are often rendered difficult".

"Another completely new point appeared for the first time at the Geneva negotiations (in September–October 1964). At that time the developing countries began to make demands for an increased outlet of their products in the developed countries. The views and plans were different in detail . . . but . . . all resulted in the claim that the developed countries should give their aid for the establishment of sugar industries (in the developing countries). Furthermore the undeveloped countries have claimed that the developed countries should renounce parts of their production and should cover the resultant deficits by imports from the undeveloped countries and this at prices as high as possible. Less crass demands from these circles aim at halting production in the developed countries at the present levels and meeting subsequent consumption increases by imports from undeveloped countries . . . It is understandable that the agricultural and sugar industries in the developed countries are no advocates for a reduction of sugar production, which will worsen their economic basis".

"The fixing of a minimum price is a great problem" Dr. AHLFELD considers. "The prices wanted (by the producing or exporting countries) considerably exceed the price of 3.25 cents negotiated in 1953 because production costs have increased meanwhile. If one considers, however, that already at the present ruinous world market prices too much sugar is produced, one has to fear that at a price of 4.00 cents or even 4.50 cents, a further increase in production would be the consequence. . . . One must not forget that all stocks, and particularly surplus stocks, are potential exports; this is only a question of the price . . . An improvement of the situation on the world market will not be possible (only) by fixation of a 'just' minimum price . . . but by reaching a balance between supply and demand . . . This can, however, only be reached by exporting quotas being adapted to outlets and by fixation of maximum stocks. *In this adaptation of production to outlets lies the key to the whole problem*".

In view of the history of previous attempts to return to orderly and profitable sugar manufacture, Dr. AHLFELD is pessimistic about the prospects for an early conclusion of a new International Sugar Agreement. But it can only be hoped that in this he is wrong and that the illusory nature of the benefits of attaining large quotas and increasing production capacity eventually will be realized by sugar producers before long.

¹ F. O. LICHT, *International Sugar Rpt.*, 1966, 98, (30), 1–4.

SUGAR CANE BREEDING AT COIMBATORE

A résumé of some of the main activities in sugar cane breeding at Coimbatore is given in a mimeographed booklet¹ that has recently been issued by that Institute and from which the following information or abstracts have been taken.

Production breeding

Three of the new Co varieties evolved at the Institute are showing considerable promise and likely to make a mark as commercial varieties replacing the existing ones. They are:

(i) **Co 1287.** This variety is a very vigorous grower with profuse tillering capable of giving a very good yield, higher than Co 419, the ruling variety in the Southern States. The sucrose content is very satisfactory and the yield of sugar per acre is more than that of Co 419.

(ii) **Co 1305.** This variety has recorded good yield under North Indian conditions with satisfactory sucrose content, and promises to enter the commercial stage early. It is resistant to red rot.

(iii) **Co 62174.** A very rich variety, Co 62174 has recorded better sucrose than even Co 997, known as the wonder cane for quality. The yield is also good.

Resistance breeding

Since field resistance to red rot disease is a must for any variety to be successfully adopted for commercial cultivation in North India, there has been a continuous attempt at evolving resistant varieties by suitable choice of parents and artificial screening of large populations of seedlings. The latest breeding technique of "mutation breeding" has been successfully utilized for imparting the resistant character into an otherwise commercially acceptable variety.

Vegetative buds of a number of commercial varieties were irradiated at the acute gamma source available at the Indian Agricultural Research Institute, New Delhi, and the population arising out of it, screened for the disease by artificial techniques. In all, over 65,000 stalks were inoculated and examined for resistance to the disease. One clump (out of about 1600) derived from buds of the clone Co 449 exposed to 500r had five stalks each of which showed a high degree of resistance to the strain D of the pathogen which had the widest spectrum. The normal Co 449 is susceptible to the strain. In the resistant stalks, the disease lesion was confined to the inoculated internode and was narrow, without lateral spread. The material has been tested for four years now and has maintained its resistance as compared with the normal Co 449 which it resembles in all other respects.

The variety has now been sent to all the State Research Stations, especially in North India, for trial and it is expected that it might perform well under North Indian conditions with built-in resistance to red rot disease.

Resistant genetic stocks

Work on P.L. 480 Project for evolving genetic stocks resistant to the diseases red rot, smut, mosaic, ratoon stunting disease and rust and cold tolerant has been in progress at the Institute since 1963. The objective of the programme is ultimately to evolve about 100 genetic stocks with built-in resistance to all or many of the characters mentioned above which will be useful in the breeding programme in India and U.S.A. During the year, a large population of seedlings was screened for the various characters by techniques standardized at Coimbatore and 30 genetic stocks selected with resistance to a majority of the diseases and cold. Material from these has been sent to the United States Department of Agriculture as part of the programme.

Sugar cane × Bamboo hybridization

In view of certain cytogenetical peculiarities, sugar cane hybridizes freely with widely distant genera. The crosses with sorghum and maize are now established to be genuine ones. However, doubts have been expressed about the genuineness of the hybrids between sugar cane and bamboo. In view of this, fresh attempts were made during the year to effect the cross between the two genera, including reciprocals. A very large number of spikelets were emasculated and crosses done under absolutely controlled conditions. While no evidence of fertilization could be obtained in the crosses involving sugar cane as the pistil parent, in the reciprocal cross, embryological studies have revealed that fertilization takes place with evidence of the embryo in the multicellular stage and the multi-nucleate endosperm. In most of the cases, the embryo later degenerates and no seed is formed. In two cases, germination was observed but the seedlings died subsequently. It would appear from the investigations the intergeneric hybridization between *Saccharum* and *Bambusa* can be successful and hybrids possible by resorting to embryo culture or ovule culture. This work was to be intensified during 1966.

Mutation studies

Work on irradiation using X-ray and gamma sources has been intensified during the year and that on chemical mutagens started. Two of the interesting results obtained are non-spiny (sheath) mutants in the variety Co 419—one from 3000r and the second through the use of the chemical mutagen at 0.01 M pyrogallol concentration. Production of non-spiny sheath mutants in sugar cane is commercially very important as varieties such as Co 527, with a highly spiny leaf sheath, are not popular in spite of very desirable economic attributes, owing to difficulties in stripping of trash by human labour. A mutant, showing profuse tillering, a desirable economic attribute, has also been isolated.

¹ Salient Research Achievements, 1965 (Sugar Cane Breeding Institute, Coimbatore), 10 pp.

Studies in heritability

Important investigations have been started during the year on heritability, the findings of which are expected to be of use in furthering the efficiency of the breeding and selection work. Any character recording high value of heritability (particularly in

the narrow sense) is less affected by environment and more governed by genetic factors and hence capable of being fixed by proper methods of breeding. A genotype with high heritability values for a number of desirable economic attributes is bound to transmit the same to the progeny.

F.N.H.

DETERIORATION OF SUGAR CANE AFTER HARVESTING

Part II. Investigation of the polysaccharide formed

by J. BRUIJN

(Sugar Milling Research Institute, University of Natal, Durban)

Paper presented at the XIX Convention of the South African Chemical Institute, Stellenbosch, February 1966

INTRODUCTION

IT was shown in Part I of this series¹ that the main product formed in deteriorating sugar cane is a polysaccharide. It is generally assumed that this polysaccharide is identical to dextran². We have now isolated this polysaccharide, determined its structure and compared it with a dextran obtained in a pure culture of *Leuconostoc mesenteroides*.

EXPERIMENTAL

Isolation and properties of the polysaccharide

Sugar cane which had been stored in the open for six weeks was milled in a three-roller unit and the starch and other particles removed from the expressed juice by continuous centrifugation. The juice was subsequently concentrated to approximately 50% dissolved solids. The polysaccharide was precipitated from the resulting syrup by adding 3.5 times its volume of acidified ethanol (150 ml ethanol, 15 ml conc. hydrochloric acid plus 15 ml water). After one day the precipitate was filtered, washed with aqueous ethanol (70% v/v) and re-dissolved in hot water. The polysaccharide was then re-precipitated by adding aqueous ethanol (70% v/v). This procedure was repeated once more and the final product was dried *in vacuo*. In the first isolations the procedure was followed by a dialysis against cold tap water, but later it was found that a nearly ash-free product could be obtained without dialysis. Subsequent fractional precipitation in ethanol did not indicate the presence of more than one polysaccharide. The typical analysis of the product is given in Table I.

Table I

Protein	1.17%
Starch	0.13%
Ash	0.14%
Polysaccharide, other than starch..	94.17%
Specific Rotation	160°

The protein content was determined by a micro-Kjeldahl technique³ and starch colorimetrically with iodine according to the method of ALEXANDER⁴. The polysaccharides, other than starch, were determined by acid hydrolysis of the product, followed by the

titration of the glucose formed, using the method of HAGEDORN & JENSEN⁵. The obtained value was corrected for the amount of glucose resulting from the starch present.

Preparation of pure dextran

A 10% sucrose solution, containing a small amount of peptone and the usual inorganic salts, was inoculated with *Leuconostoc mesenteroides* isolated from sugar cane. After three weeks the dextran formed was isolated from the culture by repeated precipitation with ethanol. A product free from ash and protein, and having a glucose content of 98.6% after acid hydrolysis, was obtained.

Acid hydrolysis

The polysaccharide was hydrolysed for five hours in 1N sulphuric acid at 100°C and the hydrolysate was neutralized with barium carbonate and filtered. Paper chromatographic analysis of the filtrate with various eluants⁶ resulted in only one spot with an *R_f* value identical to that of glucose. Reaction with phenylhydrazine produced an osazone (m.p. 204°C) identical to glucosazone by microscopical examination. The melting point was not depressed on admixture with authentic glucosazone.

Molecular weight

The molecular weight was determined by viscosity measurements with a Hoeppler viscometer and calculated from a formula suggested by WALES & MARSHALL⁷ for degraded dextran. Various batches of polysaccharide showed molecular weights between 8000 and 34,000.

¹ BRUIJN: *I.S.J.*, 1966, **68**, 331-334.² MEADE: "Cane Sugar Handbook" (Wiley, New York), 1963, p. 305.³ LANGE: *Anal. Chem.*, 1958, **30**, 1692.⁴ ALEXANDER: *Proc. 28th Congr. S. African Sugar Tech. Assoc.*, 1954, 100.⁵ BATES: "Polarimetry, Saccharimetry and the Sugars" (U.S. Government Printing Office, Washington), 1942, p. 198.⁶ WHISTLER: "Methods in Carbohydrate Chemistry", Vol. 1. (Academic Press, New York), 1962, p. 25.⁷ *J. Polymer Sci.*, 1953, **10**, 229.

DETERIORATION OF SUGAR CANE AFTER HARVESTING

The molecular weight of the pure dextran was found to be 600,000 by the method referred to above. The WALES & MARSHALL formula is not entirely valid, however, for higher molecular weight dextrans.

Periodate oxidation

The periodate oxidation was carried out at 4°C in the dark for seven days using 100 mg of polysaccharide and 400 mg of NaIO₄, made up to a total volume of 100 ml with water. At intervals the periodate uptake was determined by the method of FLEURY & LANGE⁸ and the formic acid produced was titrated with 0.01N barium hydroxide to pH 7.5. Potato starch and dextran were treated in a similar way. After seven days the daily increment in periodate uptake was only 0.5% of the total amount of periodate used. The results are shown in Table II.

Table II

Polysaccharide	Moles of IO ₄ reduced per glucose unit	Moles of HCOOH formed per glucose unit
Dextran	1.50	0.78
Starch	0.83	0.05
Cane polysaccharide	1.08	0.26

Exhaustive methylation of dextran and cane polysaccharide

The first Haworth methylation was carried out at 5°C. Ten grams of polysaccharide was dissolved in 100 ml 30% sodium hydroxide, and 180 ml dimethyl sulphate and 250 ml sodium hydroxide were added over ten hours in thirty equal portions. In the case of dextran 200 ml of acetone was added in 50-ml portions at intervals. The mixture was continuously stirred. On completion of the reaction the mixture was neutralized while cooling in ice and in the case of dextran the acetone was distilled from the mixture. The methylated product was subsequently recovered by dialysis against cold tap water, concentrated and dried *in vacuo*. In the second Haworth methylation the temperature was gradually increased from room temperature to 50°C. The methoxyl content at this stage, determined according to the method of VIEBÖCK & BRECKER⁹, was 35% for the cane polysaccharide and 38.4% for the dextran. Further methylations were carried out according to the KUHN procedure¹⁰. Ten grams of the partly methylated polysaccharide was dissolved in 120 ml dry dimethylformamide and, while being stirred, the solution was treated with 45 ml of dry methyl iodide, followed by 45 g of silver oxide¹¹, which was added gradually. The reaction was carried out at room temperature for twenty-four hours, the solids removed by centrifuging and washed with dimethylformamide and chloroform, which was collected and combined with the reaction mixture after removal of the solids.

This combined solution was washed with a 1% aqueous potassium cyanide solution, followed by water, and the washings discarded. The solvent was evaporated *in vacuo* and the product dried. After the fifth KUHN methylation the methoxyl content was

found to be 41.6% for the cane polysaccharide and 41.0% for the dextran. Subsequent methylation did not raise these values any further. Both products were brittle and light yellow.

Gas chromatographic analysis of the methylated polysaccharides

The analyses of both methylated polysaccharide and dextran were carried out in two stages using a Beckman CG-2A gas chromatograph fitted with a hydrogen-flame ionization detector. After methanolysis the tetra- and trimethyl derivatives were determined using a 3 ft copper column, of 1/4-inch outside diameter, containing 6% of ethylene glycol succinate polyester on "Chromosorb P" (80-100 mesh) at 155°C and using nitrogen as carrier gas. To determine the dimethyl derivatives 14% ethylene glycol succinate polyester on "Chromosorb W" (80-100 mesh) was used at 160°C with helium as the carrier gas.

For the tri- and tetramethyl derivatives retention times were obtained in agreement with published results¹². For the dimethyl derivative no authentic samples nor literature data were available. The quantitative results calculated from the relevant peak areas and related to 2,3,4,6- tetra-O-methyl-D-glucose are shown in Table III.

Table III

	2,3,4,6- tetramethyl glucose	2,3,4- trimethyl glucose	2,3,6- trimethyl glucose	Dimethyl glucose
Cane polysaccharide	1.00	31.2	93.0	Nil
Dextran	1.00	12.6	Nil	5.9

Infra-red spectra

Approximately 10 mg of the dried and powdered cane polysaccharide, starch, dextran and cellulose respectively were mixed with 200 mg potassium bromide and formed into pellets under a pressure of 62,000 p.s.i. The mixing was carried out under an infra-light to prevent absorption of moisture. The spectra were recorded using a Perkin-Elmer model 521 spectrophotometer.

In the range 700-960 cm⁻¹ the following absorption peaks were obtained:

Table IV

	Frequency of absorption peaks (cm ⁻¹)		
Starch (potato)	702	760	849 917
Dextran		754	841 900
Cane polysaccharide	705	752	840 920
Cellulose			893

DISCUSSION

The polysaccharide from deteriorated cane was isolated in a fairly pure state as indicated by the analytical data in Table I. The only product obtained on acid hydrolysis of the polysaccharide was glucose.

⁸ WHISTLER: "Methods in Carbohydrate Chemistry", Vol. 1. (Academic Press, New York), 1962, p. 463.

⁹ *idem* *ibid.*, 454.

¹⁰ *Angew. Chem.*, 1955, 67, 32.

¹¹ WHISTLER: "Methods in Carbohydrate Chemistry", Vol. 2. (Academic Press, New York), 1963, p. 146.

¹² ASPINALL: *J. Chem. Soc.*, 1963, 1676.

In order to determine the structure of the polymer, periodate oxidation experiments were performed. It is known that periodate differentiates between 1-6 linkages on the one hand and 1-4 or 1-2 linkages on the other. If end groups in a glucose polymer are ignored, 2 glucose units linked in 1-6 position reduce 2 moles of periodate and form 1 mole of formic acid per glucose unit. A 1-4 or 1-2 bond reduces 1 mole of periodate and no formic acid is produced^{13,14}. Although it is apparent from the data in Table II that even after seven days the oxidation was not complete, valid conclusions could still be drawn from the ratio of iodate reduced to formic acid formed.

For dextran, for example, this ratio is nearly 2 (see Table II), which indicates that only 1-6 bonds are present. Starch, which is linked in the 1-4 position except for the branch points, produced on oxidation only a small amount of formic acid, which originates from its end groups. The cane polysaccharide shows a ratio of iodate reduced:formic acid produced of nearly 4, which indicates 33% of the glucose units linked in 1-6 position and 66% of the glucose units linked in either 1-4 or 1-2 position.

These results were confirmed by the G.L.C. analysis of the methylated polysaccharides. The presence of dimethyl derivatives in the methanolysate of fully methylated dextran (Table III) indicates a branched structure in accordance with published data¹⁵. Although insufficient methylation or demethylation during methanolysis may have occurred, the branched structure is confirmed by the high ratio of 2,3,4,6, tetramethyl derivatives to trimethyl derivatives. The absence of 2,3,6 trimethyl-glucopyranoside shows that the backbone of the dextran is entirely linked in 1-6 position.

The absence of dimethyl glucose in the case of the cane polysaccharide indicates a straight chain. From the ratio of 2,3,4-trimethyl glucose to 2,3,6-trimethyl glucose it is concluded that about 25% of the units are linked in 1-6 and approximately 75% in 1-4 position, which is in reasonable agreement with the results obtained from periodate oxidation.

Assuming there is no degradation during methylation of the cane polysaccharide there is one non-reducing end group per 125 units. As the molecule was shown to be a straight chain one, the M.W. = 20,000. This is in good agreement with the values found by viscosity measurements.

The infra-red spectra (Table IV) show an absorption peak of 840 cm^{-1} for the cane polysaccharide. The value for the α -glucosidic bond, is given in the literature as $844 \pm 8 \text{ cm}^{-1}$ ^{16,17}. The value for the β -glucosidic bond is given in the literature as $891 \pm 7 \text{ cm}^{-1}$, and was confirmed by the presence of a peak at 893 cm^{-1} in a recorded spectrum of cellulose, but was absent in the case of the cane polysaccharide. The two absorption peaks are sufficiently separate to enable differentiation of α - and β -glucosidic bonds.

From the above evidence the polysaccharide isolated from deteriorated cane is a relatively low molecular weight straight chain α -glucoside, linked in 1-4 (75%) and 1-6 (25%) positions.

SUMMARY

The polysaccharide formed in deteriorating cane was isolated and its structure established by periodate oxidation, G.L.C. analysis of fully methylated products and the infra-red spectrum. The compound was shown to be a straight chain glucose polymer having 25% 1-6 and 75% 1-4 α -glucosidic bonds.

ACKNOWLEDGEMENTS

Thanks are due to Mrs. L. DONKIN, Mrs. J. A. POYNTON and Mrs. E. M. J. SWART for their assistance, to Dr. J. McD. BLAIR, University of Capetown, for the G.L.C. analysis and to Dr. K. H. PEGEL, University of Natal, for the I.R. analysis.

¹³ HEHRE: *J. Biol. Chem.*, 1951, **192**, 161.

¹⁴ JEANES & WILHAM: *J. Amer. Chem. Soc.*, 1950, **72**, 2655.

¹⁵ BARKER *et al.*: *J. Chem. Soc.*, 1954, 2395.

¹⁶ *idem ibid.*, 171.

¹⁷ BROCK NEELY: "Advances in Carbohydrate Chemistry", Vol. 12. (Academic Press, New York), 1957, p. 13.

LATEST DEVELOPMENTS IN THE G.P. FILTER

by P. DUPONT

(Raffinerie Tirlmontoise, S.A., Tirlmont, Belgium)

Paper presented to the 18th Technical Conference, British Sugar Corporation Ltd., 1966.

PART II

Methodical sweetening-off by repeated filtrations and dilutions

It is a well-known fact that the sweetening-off of muds on a vacuum drum filter may lead sometimes to unsatisfactory results, particularly when juices of low quality are processed.

Experience has shown that, while in such conditions the G.P. I filter goes on working properly

as a thickening filter, efficient sweetening-off of the thickened muds on a drum filter still remains a problem.

This has led to consideration of a way of sweetening-off based on another principle, i.e. a counter-current washing of the dry matter by successively thickening and diluting the muds in a cascade of G.P. filters.

In such a process, the ability to reach a sufficiently high mud density is as important as the necessity of

LATEST DEVELOPMENTS IN THE G.P. FILTER

efficient draining of the cossettes in an RT diffuser. Therefore, the principle of the G.P. I filter, particularly with regard to its recent development, is very well suited to such operation.

In Fig. 6 appears a schematic outline of the installation.

In the first filter F1, 1st carbonatation juice is filtered. The clear juice is sent to 2nd carbonatation, while the thickened mud from the total purge is dropped into tank D₁, where it is mixed with the partial purge from filter F2 and diluted by the clear sweet-water collected from filter F3.

This diluted mud is then fed to filter F2. Its clear effluent constitutes the sweet-water from the methodical sweetening-off, while the thickened mud from the total purge is again diluted with water or the filtrate from a further filter F4.

In this way a series of successive thickenings and dilutions is obtained. Supposing that at the end of each filtration an identical mud density is reached, it follows that the whole of the mud goes through all filters, while also identical volumes of clear juice are collected at filter F2 and each of the following filters. This volume is actually given by the amount of fresh water added in the last step.

Assuming that, at each dilution, sugar is entirely dissolved into the liquid phase, a sugar balance can be considered. The amount of sugar in the diluted mixture is given as the sum of sugar in the thickened mud plus the sugar in the diluting liquid.

$$AX_1 + BX_3 = CX_2 \dots\dots\dots(1)$$

$$AX_2 + BX_4 = CX_3 \dots\dots\dots(2)$$

$$AX_3 + BX_5 = CX_4 \dots\dots\dots(3)$$

$$AX_4 + BX_6 = CX_5 \dots\dots\dots(4)$$

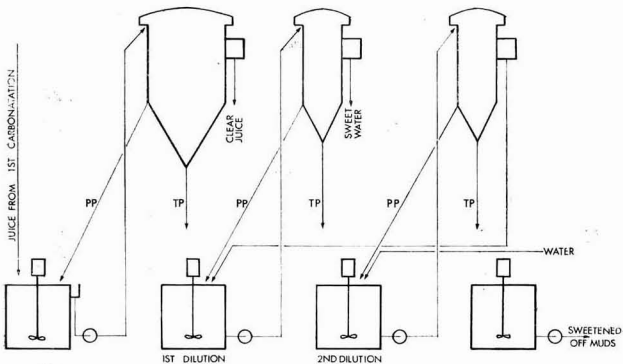


Fig. 6

where A = the volume of liquid in the muds, which is constant and is given by the mud density; B = the volume of fresh water added in the last step; $C = (A + B)$ = the total volume after mixing; X_N = sugar content of filtered juice and juice occluded in the muds from step N ; it also equals the sugar content of the mixture after dilution at step $N-1$.

$$\text{Then } X_2 = \frac{AX_1 + BX_3}{C} \dots\dots\dots(5)$$

$$X_3 = \frac{A^2X_1 + CBX_4}{C^2 - AB} \dots\dots\dots(6)$$

$$X_4 = \frac{A^3X_1 + (BC^2 - AB^2)X_5}{C^3 - 2ABC} \dots\dots\dots(7)$$

$$\text{and } X_5 = \frac{A^4X_1 + (BC^3 - 2AB^2C)X_6}{C^4 - 3ABC^2 + A^2B^2} \dots\dots\dots(8)$$

Example 1:—Supposing filtration and sweetening-off of the 1st carbonatation muds by means of a cascade of 3 filters, i.e. 2 successive dilutions.

We put $X_1 = 14$, while, of course, $X_4 = 0$.

The mud density is 600 g/litre; $A = 0.75$

At each dilution 2 litres of clear liquid are mixed with 1 litre of mud. So $B = 2$

$$C = 2.75$$

$$X_3 = \frac{(0.75)^2 \times 14}{(2.75)^2 - (2 \times 0.75)} = 1.31.$$

Muds of 600 g/litre density correspond to a dry solids content of 45% w/w.

The sugar loss % sludge at 50% dry solids content will be: $\frac{55 \times 1.31 \times 50}{100 \times 45} = 0.8$ g.

The volume of wash water required is 2 litres for 0.6 kg of dry solids, or $\frac{2 \times 0.5}{0.6} = 1.6$ per kg of sludge at 50% dry substance, which is considered excessive.

Therefore we will consider:

Example 2:—Supposing filtration and sweetening-off of the 1st carbonatation muds is by means of a cascade of 4 filters, i.e. 3 successive dilutions.

We again put $X_1 = 14$ and $X_6 = 0$.

With a mud density of 600 g/litre, $A = 0.75$.

We will assume a dilution of the mud in a ratio of 1.2 litre water added to 1 litre of mud at a density of 600 g/litre.

So $B = 1.2$

$$C = 1.95$$

$$X_4 = \frac{(0.75)^3 \times 14}{(1.95)^3 - (1.5 \times 1.2 \times 1.95)} = 1.4$$

which corresponds to the following sugar loss, expressed as % sludge at 50% dry solids content:

$$\text{Sugar loss} = \frac{55 \times 1.4 \times 50}{100 \times 45} = 0.85.$$

On the basis of 7 kg of sludge (at 50% dry solids) for 100 kg beet, the sugar loss % beet in the sludges will amount to 0.06.

The volume of wash water required equals 1.2 litre for 0.6 kg of dry substance or 1.0 litre per kg sludge at 50% dry solids.

Both values, i.e. sugar left in the sludge and water consumption, look normal.

A pilot installation including 3 filters and 2 successive dilutions operated on this scheme in Tirlemont for 3 weeks during the 1965 campaign.

Table I shows the analytical results which were obtained for sugar content in the sludges and in the clear effluents. The data are shown for 2 successive days.

The different samples relate to the following:

Mud No. 1: sampled ahead of filter 2, after the 1st dilution.

2: sampled ahead of filter 3, after dilution with fresh water.

3: sampled after third filtration and dilution with river water to permit its evacuation to the lagoons.

The different samples of the filtrate correspond to the clear juices sampled respectively from filter 1, 2 and 3.

For mud sample No. 3, which represents the actual sweetened-off slurry, we noted the following sugar contents expressed as % w/w at 50% dry solids:

1-15; 1-12; 0-76; 0-63; 0-57; 0-67; 0-51; 0-49; i.e. an average value of 0-73.

Water consumption was very near to 2 litres per litre of sludge. The figures look better than in the numerical example cited above since the mud density exceeded 600 g/litre.

The above-outlined process of sweetening-off presents from a theoretical point of view important advantages over vacuum filtration on a drum filter:

(1) vacuum filtration is replaced by pressure filtration. This allows working at higher temperature which is most important with viscous juices;

(2) it calls for equipment which is simpler, less expensive and less fragile;

(3) it avoids the difficulty of applying a homogeneous spray of wash water to the cake;

(4) and it avoids the possibility of irregularities in the mud deposit on the filter cloth or in the mud structure as well as porosity differences in the cake, which can lead to preferential paths for the wash water and to low sweetening-off efficiency.

The experimental results we have obtained demonstrate the validity of the principle of this method. However, we think that more tests are necessary before we can consider an actual installation in the factory.

We return now to the typical filtration station for 1st carbonatation juice in a 4000 ton sugar factory which was under consideration before the detailed discussion of the sweetening-off process.

As shown in Fig. 5, three continuous vacuum cell-divided (2 vacuum lines) drum filters each of about 40 sq.m. filtering area are included. Two would be permanently working, the third being a spare unit. They would operate only as sweetening-off filters.

Table I
Test runs on sweetening-off by means of G.P. filter
Muds

Date	Hour	Sample	Weight 500 ml	Dry solids % w/w	Dry solids % w/w	Total sugar	Filtrate	
							Brix	Pol
10.11.65	8	1					16-09	14-90
		2					6-87	6-10
		3					1-51	1-275
10.11.65	10	1	580	25-9	30-1	7-2	—	—
		2	560	20-1	22-5	2-75	5-34	4-60
		3	629	38-6	48-4	1-15	1-59	1-33
10.11.65	12	1	560	20-1	22-5	7-75	—	—
		2	556	18-9	21-06	2-40	3-92	3-38
		3	618	35-9	44-34	1-12	1-27	1-10
10.11.65	14	1	569	22-8	25-84	9-48	15-26	14-13
		2	556	18-9	21-06	2-16	6-12	5-45
		3	649	43-08	56-10	0-76	1-00	0-83
10.11.65	16	1	570	23-1	26-2	8-74	—	—
		2	547	16-3	17-9	2-59	5-65	4-95
		3	669	47-6	63-5	0-63	1-11	0-95
11.11.65	8	1	570	23-1	26-2	8-78	15-62	14-47
		2	564	21-3	24-02	1-68	5-72	5-08
		3	624	27-36	46-62	0-57	1-46	0-93
11.11.65	10	1	578	25-34	29-34	6-93	—	—
		2	567	22-20	25-12	1-91	5-13	4-40
		3	624	37-36	46-62	0-67	1-38	1-15
11.11.65	14	1	562	20-70	23-26	8-32	16-23	15-02
		2	556	18-90	21-06	1-59	4-67	4-05
		3	588	28-08	33-04	0-51	0-89	0-70
11.11.65	16	1	559	19-80	22-14	7-73	—	—
		2	562	20-70	23-26	1-59	4-10	3-50
		3	579	25-62	29-72	0-49	0-96	0-77

Mud density in the total purge of the G.P. thickening filters should be kept as high as possible. This density will, of course, be too high for sending these muds directly to a drum filter. If they are diluted by adding a liquid of lower sugar content than the juice in the muds, this brings about the first stage of the sweetening-off process described above, i.e. successive dilutions and filtrations. Then to the drum filter is pumped a slurry which is already partly sweetened-off.

If two vacuum valves are provided on the drum filter, dilution will be effected with the most dilute effluent. Its sugar content will be lower than in the classical process. One may indeed expect a lower polarization for the initial juice soaking the cake on the cloth of the drum filter.

Therefore, compared with the classical method, identical sugar contents in the unsweetened cake should be obtained by using smaller volumes of fresh water, i.e. 0.7 to 0.8 litres of water per kg of mud at 50% dry solids.

Should the drum filter have only one vacuum line, and given a total water consumption of 1.1 litre per kg mud, the water could be divided thus: 0.4 litre to be used as diluting agent in the mixer for the total purge; 0.7 litre for spraying on the cake on the drum filter.

Actually, experience has shown that by this preliminary dilution of the mud (before sending it to the rotary filter) the performance of the latter is improved. With identical values of vacuum and speed of rotation, the cake deposit grows thicker and is more porous as a result of decrease in the viscosity of the liquid phase.

In conclusion, we would add that we feel the G.P. filter design to be optimal. Any research work still to be carried out will only relate to details. Most of it actually concerns other applications, while we are also examining the possibility of continuous extraction of mud from the filter.

REDUCTION OF RETENTION TIME OF JUICE AT HIGH TEMPERATURES

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INTRODUCTION

A DEMAND for increased vapour pressure and vapour bleeding may be occasioned by a need for more power for irrigation or factory extensions, e.g. addition of a refinery, or by a need for heat economy owing to a shortage of bagasse.

An increase in vapour pressure is synonymous with a higher boiling temperature of juice in the vapour cells or in the first vessels. More vapour bleeding requires vessels of bigger capacity with a longer retention time of juice. The combination of these two factors, i.e. higher juice temperature and longer retention time, increases the danger of juice discoloration and sucrose inversion. In this respect the cane sugar industry is handicapped in comparison with the beet sugar industry. Owing to the absence of reducing sugars in beet juices, the clarified juice can be sent to the evaporator with a pH well above 8.0, thus materially reducing the danger of inversion. Owing to the presence of reducing sugars in cane juices, the pH of the clarified juice may be taken to only slightly above 7.0 in cane sugar mills.

Here we draw attention to the fact that with increasing temperature the dissociation constant of water increases, which implies that the concentration of H^+ ions as well as of OH^- ions increases, as the following table for (pure) water illustrates:

Temp. (°C)	0	22	50	75	100	125	150
pH + pOH	14.92	14.00	13.26	12.70	12.29	11.85	11.63
pH = pOH	7.46	7.00	6.63	6.35	6.14	5.92	5.82

In the case of factory juices a similar phenomenon occurs, but, fortunately, owing to the presence of buffering substances the increase in H^+ ion and OH^- ion concentration is smaller than that of (pure) water.

In a beet sugar factory, during a period of several months, SCHLEGEL determined the pH of samples of clarified juice extracted after the first vessels of a pressure evaporator¹. The measurements were made both at 20°C and 128°C, a recording potentiometer being used for the higher temperature. Based on the collected data SCHLEGEL calculated an average pH drop of 0.013 per °C for the temperature range 20° to 128°C. Clarified juice with a pH of 8.6 at 20°C appeared to have a pH slightly above 7.0 at a temperature of 128°C.

In the cane sugar industry we are less fortunate as we have to operate at much lower pH values. This is one of the main reasons why pressure evaporation with juice boiling temperatures of 125°C and higher has not been introduced in cane sugar mills.

For instance, if we assume, for argument's sake, that the same temperature coefficient as determined by SCHLEGEL for beet juice holds good for cane juice, then clarified cane juice with a pH of 7.4 at 20°C will have a pH of 6.2 and a pOH of 5.9 at the relatively low temperature of 110°C.

¹ *Zeitsch. Zuckerind.*, 1963, **88**, 14-23.

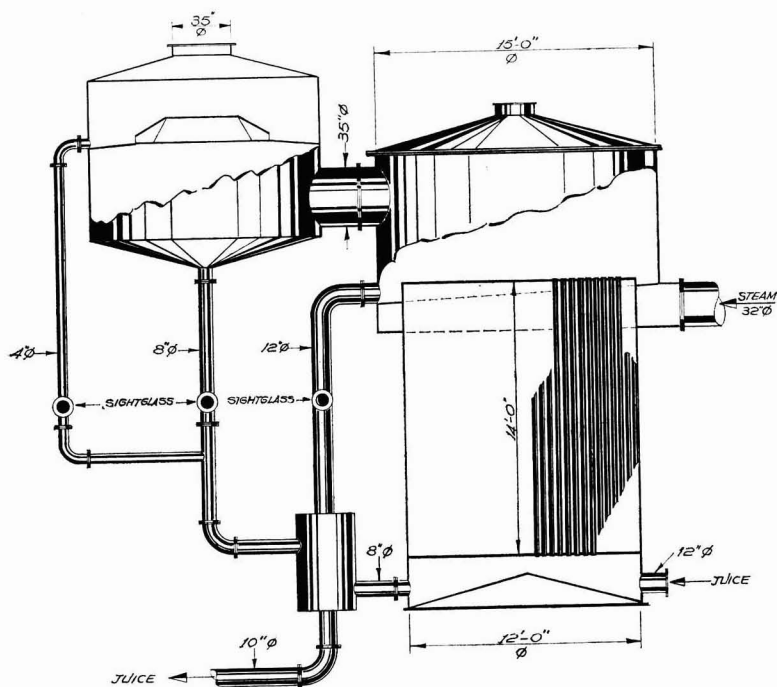


Fig. 1

This example shows that even at the relatively low vapour pressure of 5 p.s.i.g. (corresponding to a juice temperature of 230°F or 110°C), there is imminent danger of inversion as well as of destruction of reducing sugars, owing to a material increase in H^+ ion as well as in OH^- ion concentration.

Since both inversion and decomposition of reducing sugars are governed by similar parameters (pH or pOH, temperature and time), reduction of the retention time of the juice to the barest minimum is the only measure available.

RETENTION TIME

The following examples illustrate the increase in retention time in first vessels when vapour bleeding is introduced:

(i) A factory crushing 200 tons cane per hour was originally equipped with a straight quadruple effect of four vessels, each of 14,000 sq.ft. heating surface.

(ii) When vapour bleeding was introduced the straight quad was replaced by two first vessels of 15,000 sq.ft. each, followed by three vessels of 9,000 sq.ft.

(iii) Instead of two first vessels with a combined heating surface of 30,000 sq.ft. one semi-Kestner evaporator of 20,000 sq.ft. was installed.

Note: The Robert type vessels had 6 ft-long tubes of 1 3/8 inches o.d. and the semi-Kestner tubes of similar diameter but with a length of 14 ft.

Initially the retention time of the juice in the first effect was 2 min 41 sec, in system (ii) it increased to 10 min 25 sec and in system (iii), where the semi-Kestner was used, it fell again to 2 min 25 sec. This illustrates that the retention time with the two ordinary vessels is 4 1/2-times as long as that of a specially designed vessel².

SEMI-KESTNER APPARATUS

Fig. 1 depicts a semi-Kestner apparatus with a heating surface of 20,000 sq.ft. composed of 4300 tubes of 1 3/8 in. o.d. \times 14 ft 0 in. Two sets of apparatus of this size were recently installed in factories in Southern Africa, in addition to a number of smaller sets.

Owing to the high juice velocity through the evaporator tubes the vessel keeps clean longer and the heat transfer rate is also higher than that of ordinary

(Robert) vessels operating under similar conditions. The approximately 50% higher heat transmission coefficient reduces the required heating surface to two-thirds that of Robert vessels. This, combined with the reduction in the number of tubes because of their greater length, results in a calandria with a greatly reduced diameter. In addition, the dishing of the bottom of the semi-Kestner as depicted in Fig. 1 reduces the juice volume below the calandria still further compared with the inverse conical bottoms of the Robert vessels, the total result being that the juice capacity of the latter is about 5 1/2-times that of the semi-Kestner of equal evaporation capacity. Since the retention time is proportional to juice capacity, it follows that the juice stays 5 1/2-times longer in the Roberts vessels than in the semi-Kestner.

The dishing of the bottom of the vessel not only reduces the juice capacity of the vessel, but also helps to distribute the juice uniformly over all tubes.

Installation of the save-all in a separate chamber has two advantages. First, it leads to easier cleaning, as the space above the calandria is free from obstructions, and, second, it reduces materially the danger of entrainment.

Trying to clean more than 4000 tubes in one day would require the operation of many motor-driven brushes in a confined space. However, as the

² PERK: *Quarterly Bull. Sugar Milling Research Inst.*, (3), (4) & (6); *S. African Sugar J.*, 1957, 41, 549-552; 815-818; 1958, 42, 317-321.

tubes stay clean longer (notwithstanding the higher evaporation rate per sq. ft.), the cleaning can be spread out by brushing a certain number of tubes each weekend.

The small vessel to the left of the calandria of the semi-Kestner vessel serves as a manifold for the downcomers and as a float chamber for the automatic juice level control. The sight-glasses in the downcomer pipes make it possible to observe the

operation of the evaporator and its save-all.

In the pipe connecting the juice collecting vat and the space below the calandria of the semi-Kestner a gate valve, and sometimes also a non-return valve, is usually fitted. The gate valve is used to control the circulation rate, while the non-return valve is fitted to ensure that no short-circuiting of juice can occur when the juice level in the collecting vat accidentally becomes too low.

CONSTITUENTS OF STANDARD LIQUOR FILTER CAKE

by A. CARRUTHERS, J. F. T. OLDFIELD and M. SHORE

Paper presented to the 18th Technical Conference of the British Sugar Corporation Ltd., 1966

Introduction

At all white sugar factories of the British Sugar Corporation, high raw and after-product sugar is dissolved in the thick juice to form standard liquor, which is filtered after sulphitation before crystallization. Leaf, candle or plate-and-frame filters are employed for the standard liquor filtration and kieselguhr or perlite is used both as precoat and for concurrent addition during filtration. The filter aid usage is normally equivalent to some 20 to 60 lb per 100 tons of beet.

In some countries, white sugar is produced without any filtration after the evaporation station, and so the value of standard liquor filtration was assessed by examination of the composition of standard liquor filter cake.

Procedure

At intervals during the 1965/66 campaign, samples of filter cake weighing about 2 kg were collected during discharge of the standard liquor presses at Nottingham factory. This cake is normally returned to 1st carbonation and so the cake had not been sweetened-off before sampling. Each sample was thoroughly mixed and analysed by the following procedure:

Samples of the fresh cake were triturated with N hydrochloric acid and filtered. Calcium was determined in the filtrate by EDTA titration and oxalate was determined by permanganate titration after reprecipitation of the calcium salt.

Samples of the fresh cake weighing 30 g were extracted with 500 ml of 90% methanol, filtered and dried. 0.5 g portions of the dried residue were hydrolysed with 3 ml of 50% hydrochloric acid in sealed tubes at 110°C for 16 hours. The hydrolysate was made alkaline with 2N sodium hydroxide and heated at 100°C to remove ammonia. After neutralization the residual amino nitrogen in the hydrolysate was determined with the MOORE and STEIN ninhydrin:hydrindantin reagent¹. This value was multiplied by 6.25 and expressed as the protein content.

Fresh cake was also washed with water to remove sugar as completely as possible; filtration after washing proved intractable and so the solids were recovered by centrifugation. The washed material was dried at 110° for determination of dry substance in the original cake. The dry material was extracted with ether and the ether extract washed with N sulphuric acid and with N sodium carbonate and then dried with sodium sulphate. The ether solution was filtered and, after removal of the solvent, the residue was weighed and recorded as the oil content.

The amount of filter aid in the cake was deduced by subtracting the weight of accountable constituents from the weight of dry substance. For this purpose the calcium in excess of that present in calcium oxalate was multiplied by 2.5 to convert to weight of calcium salts.

Results

The analytical values are recorded in Table I.

Table I. Components of standard liquor filter cake (values expressed % dry substance in washed cake)

Date	Filter aid (%)	Oxalic acid anhydrous (%)	Calcium %	Protein %	Oil %
14.10.65	92.8	1.27	1.92	0.78	0.95
8.11.65	91.9	1.08	2.28	1.37	0.47
11.11.65	92.6	1.43	1.44	2.28	0.95
4.12.65	92.9	1.35	2.17	0.58	0.35
5.12.65	92.4	0.93	2.23	0.71	0.89

The analytical values are not proportional to the concentrations in standard liquor because the filter aid usage is varied during the campaign. From the actual filter aid usage, it is possible to calculate the concentrations relative to the amount of beet sliced, and relative to the white sugar produced. These values are recorded in Tables II and III.

During the 1965/66 campaign, Peterborough raw beet sugar for concurrent refining was also dissolved in the standard liquor at Nottingham factory. Juice

¹ J. Biol. Chem., 1954, 211, 907.

is not filtered after the evaporator stage in the raw sugar process and any insoluble material would remain in the raw sugar and contribute to the Nottingham standard liquor filter cake. The concurrent refining would therefore slightly inflate the values per 100 tons beet in Table II, but would not be expected significantly to affect the values relative to sugar production recorded in Table III.

Table II

Components of standard liquor filter cake relative to beet sliced
(values expressed as lb/100 tons beet)

Date	Filter aid	Calcium oxalate mono-hydrate	Other calcium salts	Protein	Oil
14.10.65	43	0.95	0.63	0.36	0.44
8.11.65	68	1.30	1.33	1.02	0.35
11.11.65	84	2.10	0.72	2.06	0.86
4.12.65	76	1.80	1.29	0.48	0.29
5.12.65	76	1.26	1.50	0.59	0.73

The cost of filter aid alone rises to about 2s 6d per ton of sugar but the removal of the components shown in Table III is clearly an essential feature in the production of high quality white sugar. In the absence of a standard liquor filtration stage, the white sugar would contain from 0.007% to 0.016% impurity from this source. The origins and effects of the individual components are considered below.

Table III. Components of standard liquor filter cake relative to white sugar produced
(values expressed as mg/100 sugar)

Date	Filter aid	Calcium oxalate mono-hydrate	Other calcium salts	Protein	Oil
14.10.65	125	2.6	1.8	1.0	1.3
8.11.65	202	3.9	3.9	3.1	1.1
11.11.65	234	5.9	2.0	5.8	2.4
4.12.65	245	5.8	4.2	1.5	0.9
5.12.65	245	4.0	4.8	1.9	2.4

Calcium Oxalate

On average, rather more than half of the calcium salts removed on the standard liquor filters consisted of calcium oxalate. Although the solubility product of calcium oxalate is extremely low, it has been shown both from theoretical considerations² and by radiochemical assay³ that the oxalate in raw juice is incompletely removed in the liming and carbonatation procedure. The residual oxalate level of some 10–15 mg/litre in 2nd carbonatation juice⁴ is about 100 times greater than would remain after liming an aqueous solution of potassium oxalate in 10% sucrose.

If only a solubility relationship was involved, the residual oxalate after juice clarification would be independent of the amount of lime used and of the oxalate concentration in raw juice. The effects of variations in lime usage, and of raw juice oxalate concentration at a constant lime addition of 15 g/litre, are recorded in Tables IV and V. The variations in Table V were obtained by adding potassium oxalate to the raw juice in samples B to E.

The residual oxalate levels demonstrate that the oxalate elimination is determined predominantly by solubility relationships.

Table IV. Relationship between lime usage and residual oxalate concentration

Juice	Lime usage g/litre	Residual oxalate, mg anhydrous acid per litre
A	7.5	19.1
B	15.0	17.4
C	22.5	15.5

Table V. Relationship between oxalate in raw juice and residual oxalate concentration

Juice	Oxalate in raw juice, mg anhydrous acid per litre	Residual oxalate, mg anhydrous acid per litre
A	300	14.3
B	600	13.0
C	900	11.2
D	1200	14.1
E	1800	12.3

During clarification, the bulk of the oxalate is precipitated in liming and, when the calcium content is lowered by carbonatation, the 1st carbonatation filtrate is no longer saturated with respect to calcium oxalate; consequently no more oxalate is removed in the normal 2nd carbonatation stage.

As the water is removed in the evaporators, the residual calcium and oxalate become more concentrated so that the juice is again saturated with respect to calcium oxalate. Calcium oxalate precipitates in the evaporator juice, and some remains suspended in the juice while some is deposited to form scale on the evaporator tubes. Analysis of an evaporator scale from Spalding factory showed the scale to contain more than 75% of calcium oxalate monohydrate. The suspended calcium oxalate contributes to that removed in standard liquor filtration.

About half of the oxalate remaining after clarification is precipitated during evaporation, as demonstrated by the oxalate concentrations in 2nd carbonatation and in unfiltered thick juice recorded in Table VI.

Table VI. Precipitation of calcium oxalate during evaporation

	Soluble oxalate—mg anhydrous acid per 100S	
	2nd carbonatation juice	Thick juice
Brigg	11.1	5.1
York	8.4	5.0
Selby	7.1	5.7
Spalding	11.0	7.0

The filtered standard liquor is still saturated with respect to calcium oxalate and so more calcium oxalate is precipitated as evaporation continues in the crystallization stages. Some of the precipitated oxalate forms scale in the vacuum pans, while some remains suspended in the sugar end syrups and is in part separated with the low product sugars in the centrifugals. Separation of solid material from a 50°Bx solution of Spalding afterproduct sugar showed that this sugar contained 8.8 mg of calcium oxalate per 100 g sugar.

² CARRUTHERS *et al.*: Paper presented to 9th Tech. Conf. British Sugar Corp., 1956; I.S.J., 1957, 59, 106.

³ CARRUTHERS & OLDFIELD: Paper presented to 12th Tech. Conf. British Sugar Corp., 1959; I.S.J., 1959, 61, 376.

⁴ CARRUTHERS *et al.*: Paper presented to 12th Tech. Conf. British Sugar Corp., 1959; I.S.J., 1959, 61, 376.

CONSTITUENTS OF STANDARD LIQUOR FILTER CAKE

The calcium oxalate removed by standard liquor filtration therefore originates partly from precipitation of calcium oxalate in the evaporator stage and partly from the calcium oxalate precipitated in the sugar end and returned to standard liquor with the remelt sugars.

Minute amounts of calcium oxalate are also precipitated with the first product sugar and can contribute turbidity to this sugar. With sugars exhibiting relatively high turbidity it is possible to distinguish turbidity due to calcium salts from that arising from other sources, by measurement of the turbidity of a 50°Bx solution of the sugar before and after acidification with hydrochloric acid. Calcium oxalate is soluble in dilute mineral acid and the calcium salt turbidity is removed on acidification.

Even when the turbidity is relatively high, the amounts of insoluble matter are very small indeed and are normally estimated in terms of mg per kilo of sugar rather than the mg per 100 sugar quantities reported above. The limit proposed by the Codex Alimentarius Commission for insoluble matter in first grade white sugar is 20 mg per kilo and the choice of this limit has been influenced more by the difficulty of measuring smaller levels rather than by any difficulties in achieving the standard in production sugar.

By selecting sugars exhibiting relatively high turbidity, it proved possible to separate insoluble material by continuous-flow, high-speed centrifugation. A sample of between 9 kg and 18 kg of sugar was required to produce an adequate yield of insoluble matter. The sugar was dissolved in an equal weight of water and passed through a continuous-flow rotor on a high-speed centrifuge operating at 17,000 r.p.m. The recovered solids were washed with water, extracted with methanol, dried and weighed.

The analysis of the insoluble material is recorded in Table VII.

Table VII. Insoluble material from sugar exhibiting relatively high turbidity

Sample	Turbidity	Total solids mg/kg	Acid-insoluble matter, mg/kg	Calcium oxalate, mg/kg
1	18	7.2	0.9	3.5
2	22	6.4	—	3.4
3	18	6.0	0.6	3.4

The calcium oxalate recovered, even from these selected sugars, represents less than one tenth of the oxalate removed by standard liquor filtration, as recorded in Table III.

If the standard liquor were not filtered, the additional calcium oxalate passing through into the first product crystallization stage would therefore give rise to unacceptable levels of turbidity in the white sugar.

Other calcium salts

In addition to calcium oxalate, other calcium salts are removed in standard liquor filtration as shown in Table III; at least part of this material was found to exist as calcium carbonate.

The calcium carbonate cannot be precipitated by the same mechanism as is responsible for calcium oxalate precipitation. Much of the carbon dioxide in the second carbonation juice is evolved during evaporation and molasses contains very little carbon dioxide. Also, as the pH is lowered, both by sulphitation and by chemical changes in the evaporators and sugar end, the carbonate:bicarbonate:carbonic acid equilibrium is increasingly shifted away from the carbonate ion form, so that only a very small percentage of the total carbon dioxide exists as carbonate ion in standard liquor. It is not therefore to be expected that the juice would become saturated with respect to calcium carbonate at any stage subsequent to thin juice sulphitation.

The calcium carbonate probably arises from minute leakages of precipitate from the second carbonation filters. Other calcium salts may also be returned to the standard liquor with the after-product sugar, if lime is added to the crystallizers for pH adjustment.

The calcium salts, other than calcium oxalate, would also give rise to unacceptable turbidity in the white sugar if standard liquor filtration were omitted.

Oil

It is often necessary to add antifoam oil to control foaming in continuous diffusers. At Nottingham factory about 40 lb of "Kilfoam R/V" per 100 tons of beet is employed. This oil is almost completely removed in the carbonation procedure. Very much smaller quantities of purified vegetable oils are sometimes required to control frothing in the evaporators; for this purpose cottonseed oil is added to the thin juice at Nottingham factory and the average usage is equivalent to about 0.7 to 1.5 lb per 100 tons of beet.

The natural oils in the beet itself provide a third possible source of oil in the process. To obtain samples of beet oil, washed fresh beet were shredded and dried in a current of warm air and then extracted with diethyl ether. The ether extract was washed with acid and alkali, and dried in the same manner as employed to isolate the oil from filter cake. After removal of the solvent the residue represented a yield of about 0.009% on beet, equivalent to about 20 lb per 100 tons of beet. If completely extracted in diffusion, the beet oil would be equivalent to about half of the total antifoam oil usage.

Samples of the three oils, and of the cake oil, were hydrolysed with alcoholic potassium hydroxide, to liberate the component fatty acids which were separated by ether extraction and then converted to methyl esters by treatment with methanol in the presence of boron trifluoride. The individual fatty acids were detected and estimated by gas chromatography and the concentrations of the component fatty acids are expressed as proportions of the palmitic acid content in Table VIII.

Linolenic acid was detected only in the beet oil, but the remaining fatty acids, all of which are common constituents of most natural oils, were found to be

present in all the oil samples. The relative proportions of the fatty acids differ in the four oil samples, but these differences cannot be used to deduce the source of the cake oil because, if traces of the beet oil or "Kilfoam" survive the clarification procedure, fractionation may have occurred so that the fatty acid composition of these traces may differ from that of the original oil.

Table VIII. Relative proportions of fatty acids in hydrolysed oil samples, palmitic acid = 1.0

Sample	Myristic	Palmitic	Stearic	Oleic	Linoleic	Linolenic
Cake oil	0.04	1.0	0.16	0.34	0.07	—
Beet oil	trace	1.0	trace	0.36	3.92	0.54
Cottonseed	0.04	1.0	0.10	0.60	2.10	—
"Kilfoam"	0.23	1.0	0.51	0.52	trace	—

From the low linoleic acid content of the cake oil, however, it appears unlikely that the cake oil arises exclusively from the cottonseed oil added after clarification.

The three oils were also compared with the filter cake oils by thin layer chromatography on "Kieselgel H". After development in 85:15:10 heptane:diethyl ether:acetic acid, the plates were sprayed with concentrated sulphuric acid and heated at 110°C for 5 minutes to detect the components.

Qualitatively, all of the filter cake oils yielded similar patterns with components at R_f values of 0.16, 0.26, 0.29 and 1.0. The latter spot consisted of three different coloured bands running virtually with the solvent front. The three oil samples were also resolved into multiple components and all gave components at R_f 0.29 and 1.0 but only the beet oil yielded a component at R_f 0.24 to match that in the cake oil.

The bands at R_f 1.0 were eluted from the chromatogram and redeveloped in water-saturated heptane. In this solvent the fast bands from the cake oil were resolved into components at R_f 0.4, 0.5, 0.7 and 0.9 which coincided with the fast bands from "Kilfoam", whereas the same bands from beet oil and cottonseed oil remained close to the start line.

From the chromatographic evidence, it seems probable that the filter cake oil contains components from beet oil, cottonseed oil and "Kilfoam". The total amounts of oil are quite small but the standard liquor filtration serves a useful purpose in removing these traces.

Protein

Before hydrolysis, the cake samples were washed until no free amino acids and no pyrrolidone carboxylic acid could be detected in the washings. After hydrolysis, the amino acids produced were resolved by electrophoresis of hydrolysed raw juice protein.

Protein is not, however, the only potential source in standard liquor which could yield amino acids on hydrolysis. It has been shown⁶ that amino acid deoxyfructoses are produced in the sugar end from the free amino acids in the syrup and these compounds are intermediates in the colour-forming Maillard reaction in beet process liquors. The possi-

bility arose that amino acids obtained on hydrolysis of the filter cake originated as Maillard reaction complexes.

The majority of the constituent units in vegetable protein are α -amino acids⁷. One of the free amino acids in beet juice is γ -amino butyric acid and this γ -amino acid is not a normal constituent of protein⁸. The free γ -amino butyric acid in beet juice readily forms a deoxyfructose compound in the sugar end⁶ and so it would be expected that γ -amino butyric acid would be a constituent of the Maillard complexes.

γ -Amino butyric acid was not found on hydrolysis of raw juice protein or of standard liquor cake. The vast majority of the raw juice protein is removed in clarification but it appears that the nitrogenous material removed by standard liquor filtration consists of protein and is not a Maillard reaction product.

Dilute solutions of protein are normally surface active and so the quality of the white sugar would probably be reduced if these traces of protein were not removed in standard liquor filtrates.

Summary

Calcium oxalate, other calcium salts, protein and traces of oil are shown to be components of standard liquor filter cake. In the absence of a standard liquor filtration stage, insoluble material equivalent to 0.007 to 0.016% of the white sugar production would be passed to the vacuum pans.

Thin juice is not saturated with respect to calcium oxalate but, with the removal of water in the factory process, the juices became saturated and calcium oxalate is precipitated. The calcium oxalate removed by standard liquor filtration originates partly from precipitation in the evaporator stage and partly from calcium oxalate precipitated in the sugar end and returned to standard liquor with the remelt sugars.

The other calcium salts consisted predominantly of calcium carbonate and probably arose from minute leakages of precipitates from the second carbonation filters. The calcium oxalate and other calcium salts would give rise to unacceptable turbidity in the white sugar if standard liquor filtration were omitted.

Natural beet oil and antifoam residues probably give rise to the traces of oil in the standard liquor filter cake. Most of the raw juice protein is removed in clarification but small amounts of protein residue are trapped on the standard liquor filters.

Acknowledgments

The authors wish to acknowledge the assistance which they have received from their colleagues: R. PARSLow for analytical work by gas-liquid chromatography and R. K. HEANEY for amino acid analyses.

⁵ CARRUTHERS *et al.*: Paper presented to 7th Tech. Conf. British Sugar Corp., 1954; *I.S.J.*, 1954, **56**, 218.

⁶ CARRUTHERS *et al.*: Paper presented to 15th Tech Conf. British Sugar Corp., 1962; *I.S.J.*, 1962, **64**, 343.

⁷ SPRINGALL: *Royal Institute of Chemistry Monograph*, 1954, (4).

⁸ MEISTER: *Biochemistry of the Amino Acids*. (Academic Press Ltd., London.) 1957.

Agricultural

Abstracts

Ratooning of sugar cane in Bihar. S. K. PAREKH. *Indian Sugar*, 1966, **15**, 671-673.—The writer considers that insufficient attention is given to ratoon crops in Bihar where growers are inclined to lavish much attention on plant crops but neglect ratoons, regarding them as a gift from nature. The importance of manuring and irrigating ratoons to obtain high yields is stressed.

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The influence of climatic conditions on the maturity of sugar cane in Tucumán. F. A. FOGLIATA. *La Ind. Azuc.*, 1966, **71**, 17.—Weather and climatic conditions over a period of six years (1958-63) are discussed in relation to sucrose content and maturity in the variety N:Co 310. Factors discussed include light intensity, relative humidity, temperature, rainfall and evaporation.

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The spacing of monogerm sugar beet seed. H. W. STRICKER. *Zucker*, 1966, **19**, 171-174.—A study was made of sowing pelleted monogerm sugar beet seed at different spacings, 3, 4.5, 7 and 8 cm apart, and correlating this with subsequent singling and other labour costs and final yield. Increasing the spacing from 4.5 cm to 7 or 8 cm reduced the manual labour requirement for singling by an average of 25.6 and 38.1% respectively.

* * *

Control of root-knot nematodes on sugar cane in Florida. J. A. WINCHESTER. *Sugar J.* (La.), 1966, **28**, (10), 22-23.—Much of the increased planting of cane in Florida in recent years has been on land previously used for vegetables. While pre-plant nematocide treatment may give good nematode control to the plant cane, the effects have worn off by harvest time and with the ratoon crop. An account is given of experiments with three nematocides (ethylene dibromide, dichloropropene and B-25141) applied two weeks after harvesting. All were effective in reducing nematode populations and increasing cane growth and vigour. B-25141 gave the best control.

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Additional hosts of the beet water mould. C. L. SCHNEIDER. *J. Amer. Soc. Sugar Beet Tech.*, 1965, **13**, 469-477.—This mould or fungus (*Aphanomyces cochlioides*) is associated with black root disease of sugar beet in the Great Lakes region. A study of alternative hosts is recorded. In all, 28 new experimental hosts were discovered, ranging over 8 families (all Dicotyledons); 19 species were found to be natural hosts.

Production practices affecting yield and sugar content of sugar beets grown in Ontario, Canada, 1961 and 1962. C. S. BALDWIN, C. E. BROADWELL and J. F. DAVIS. *J. Amer. Soc. Sugar Beet Tech.*, 1965, **13**, 509-522.—Data were collected from some 1700 farmers, representing 20,000 acres of sugar beet, and later analysed. Several different sugar beet production practices were correlated with yield of roots, sugar per acre and sucrose content. Coarse or fine textured soil made little difference.

* * *

Host-parasite relations of *Nacobbus batatiformis* on the sugar beet and other hosts. M. L. SCHUSTER, R. SANDSTEDT and L. W. ESTES. *J. Amer. Soc. Sugar Beet Tech.*, 1965, **13**, 523-537.—This little-known nematode is economically important with sugar beet in western Nebraska and elsewhere. Further studies on the nature of the pest (cytopathology and histopathology) are recorded.

* * *

Sugar beet breeding lines combining resistance to bolting and disease. J. S. MCFARLANE and I. O. SKOYEN. *J. Amer. Soc. Sugar Beet Tech.*, 1965, **13**, 555-562.—A list of breeding lines available to sugar beet breeders in the United States is given in three sections, viz. open pollinated selections, multigerm in-bred lines and monogerm in-bred lines. Notes about each line are supplied.

* * *

Infield rail-car transport. ANON. *Victorias Milling Co. Expt. Sta. Bull.*, 1966, **13**, (3 & 4), 3.—Details are given of a new method of transporting cane, still in the experimental stage. A specially designed trailer, with tyres 24 inches wide, has a length of rail track built into it. This can be made to connect to the usual rail track and a loaded cane truck or car is drawn on to the trailer by means of a winch or tractor. It is claimed that under Philippine conditions this "infield rail-car transport", as it has been called, could save time, labour and money.

* * *

Variety yield performance in the Philippines. ANON. *Victorias Milling Co. Expt. Sta. Bull.*, 1966, **13**, (3 & 4), 4.—The performance of different cane varieties in the Victorias Milling Company's five haciendas or estates during the 1964-65 crop year is discussed. The Philippine variety Phil. 54-60 gave the best overall performance, with an average of 76.82 tons/ha and 118.27 piculs (8.3 short tons) of sugar per hectare. The American variety CP 29-116 was next in performance and yield, followed by the Barbados variety B 37172. Results with nine other varieties are reported.



Reduction in yield due to cane tasselling can be minimized. ANON. *Victorias Milling Co. Expt. Sta. Bull.*, 1966, **13**, (3 & 4), 7.—It is pointed out that losses due to tasselling or flowering in the Philippines, which can be heavy with some varieties, may be largely avoided by planting only during certain months, i.e. July to October.

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Performance of some imported cane varieties (in Brazil). F. DE MENEZES VEIGA. *Brasil Açuc.*, 1966, **34**, (3), 46-47.—A brief account is given of the performance so far of some recently imported sugar cane varieties. Figures showing yield, sucrose content, juice purity, etc., are given in tabular form. Of the 18 varieties dealt with, 12 originated from Coimbatore, 2 from Barbados and 4 from Guyana.

* * *

Cytology of *Saccharum robustum* and allied species and hybrids. S. PRICE. *Brasil Açuc.*, 1966, **34**, (3), 48-58.—The importance of the wild cane *Saccharum robustum* to the sugar cane breeder in his quest for better or disease-resistant varieties of cane is explained and the rôle it has played in the past discussed. The chromosomes, or chromosome counts of different varieties or geographical races of this wild cane, are dealt with at some length.

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L 60-25 is released. L. L. LAUDEN. *Sugar Bull.*, 1966, **44**, 252.—This new variety, bred at Baton Rouge, Louisiana, U.S.A., was released in May 1966. Its three outstanding features are early maturity, high sugar content and high yield. It also produces well on all soil types. Unfortunately it shows high susceptibility to mosaic disease.

* * *

Field properties of sugar beet seed and considerations on the mechanization of thinning. B. EHNROT. *Socker Handl.*, 1965, **20**, 19-38.—Laboratory and field experiments with sugar beet seed showed a consistently lower germination rate with field-sown than with laboratory-sown seed, varying from 16% to 40%, with an average of 27%. Seed sown by hand in the field gave better germination than seed sown by machine. On average, germination of machine-sown seed was only 90% of that sown by hand. The mathematics of thinning is discussed in some detail.

* * *

"Meringa" has transformed North Queensland sugar farming. ANON. *Producers' Rev.*, 1966, **56**, (5), 21. The many facets of sugar cane research being carried out at the Meringa Experiment Station are briefly described, especially pest control. The now successful control of grey-back grubs with BHC dust, saving the industry £2,000,000 annually, was initiated there. Other lines of work are concerned with the control of the french grub, funnel ants, grasshoppers and army worms.

25% sugar yield increase possible. ANON. *Producers' Rev.*, 1966, **56**, (5), 9.—The views of an Australian sugar cane scientist are quoted as supporting this belief, viz. that new varieties of cane, yet to be bred, will be capable of this increase in yield.

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Evaluation of "Phosfon" and maleic hydrazide as late season yield stimulants on sugar cane. N. E. DELFEL, E. ORTIZ-TORRES, C. COLBERG and G. SAMUELS. *Trop. Agric.*, 1966, **43**, 199-210.—Most cane growers have to cut at least part of their crop before or after it reaches maximum sugar content in order to meet milling schedules. Chemical treatment to hasten or to delay sugar production in the plant might be a solution to the problem. Separate stools were treated and details are given of the special spray equipment devised and used. "Phosfon" (2,4-dichlorobenzyltributyl phosphonium chloride), a plant dwarfing agent, at 0.04, 0.2 and 1.0 lb/acre had no beneficial effect. With maleic hydrazide at 3 lb/acre after 4 weeks and at 15 lb/acre after 6 weeks, stools contained 5% and 12% more sugar, respectively, than untreated stools. Sugar in juice, purity and Brix were also correspondingly higher.

* * *

How to control the sugar cane borer. E. A. CENCIENNE and S. HENSLEY. *Sugar Bull.*, 1966, **44**, 268.—Insecticides are recommended only for 2nd and 3rd generation borers and only when field inspection indicates, i.e. when 5% of the stalks show small borers on leaf sheaths. The following insecticides, formulated as 30/60 mesh granules, are recommended at the rate of 15 lb per acre—"Endrin 2%", "Guthion 7%", "Sevin 20%" and "Thiodan 3%".

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Preharvest water stress for (San Joaquin) Valley sugar beets. G. V. FERRY, F. J. HILLS and R. S. LOOMIS. *Calif. Agr.*, 1965, **19**, (6), 13-14; through *Biol. Abs.*, 1966, **47**, 5049.—Moderate water stress (dehydration) prior to harvest did not significantly reduce sugar production. Higher sucrose content and lower production costs could make this practice profitable if stress is not prolonged.

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An analysis of production practices of sugar beet farmers 1961-1963. C. S. BALDWIN, J. F. DAVIS and C. E. BROADWELL. *Mich. Agr. Expt. Sta. Quart. Bull.*, 1965, **48**, (1), 36-63; through *Biol. Abs.*, 1966, **47**, 4636.—Data from a three year survey are given. Among factors associated with high yield are tile drainage, previous crop (especially when beet follows corn), manure application, depth of ploughing, early planting and adequate K and P fertilizer. Production was maximum when beets were harvested after 15th October. In two out of three years the sucrose content of roots was reduced as the amount of nitrogen applied was greater than 70 lb/acre.

Introduction of the sugar cane borer parasite *Diatraeaophaga striatalis* to Réunion and Madagascar. J. BRÉNIÈRE et al. *Agronomie Tropicale*, 1966, 21, 361-384.—The first attempts at introduction, direct in the field in 1964, to control the borer *Diatraea saccharalis* were unsuccessful because of dry weather. Subsequently the parasite was again introduced and successfully bred under laboratory conditions. A detailed, illustrated account of the methods employed is given.

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Weeds of Mauritius. 13. *Plantago lanceolata*. E. ROCHECOUSTE and R. E. VAUGHAN. *Mauritius Sugar Ind. Research Inst. Leaflet*, 1964, (9), 3 pp.—This weed, one of the plantains, also called "herbe caroline", grows at all altitudes in Mauritius and has become troublesome in cane fields, probably because of its tolerance to the auxin-type weed killers MCPA and 2,4-D. "Simazine" and "Atrazine", at 3 lb active material per acre, give satisfactory control.

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Weeds of Mauritius. 14. *Eleusine indica*. E. ROCHECOUSTE and R. E. VAUGHAN. *Mauritius Sugar Ind. Research Inst. Leaflet*, 1965, (14), 4 pp.—This grass, sometimes called goosegrass or "chiendent", is a common weed in warm countries. It is fully described and illustrated. In Mauritius it is troublesome in gardens and along roadsides but is not a serious problem in sugar cane fields. With its shallow root system it is easily eradicated by hand cultivation.

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Sugar beet—an economic study based on a survey in Yorkshire 1963/4 and 1964/5. J. W. WOOD. *Univ. of Leeds, Dept. Agric., Econ. Section Rpt.* 1966, 72 pp. All aspects of sugar beet cultivation and production in Yorkshire, England, over two seasons are discussed in turn. A surprising conclusion is that sugar beet in Yorkshire is over-fertilized and that "lower application of plant nutrients, particularly nitrogen, would substantially enhance the profitability of sugar beet".

* * *

Results of comparative sugar beet variety trials carried out in Belgium from 1961 to 1965. N. ROUSSEL and R. VAN STALLEN. *Pub. Tech. Inst. Belge pour Amél. Betterave*, 1966, (1), 1-20.—A detailed account is given of field trials carried out at three centres (Hesbaye, Hainaut and Flandres), with multigerm and with polished and graded seed. Results are given in a series of 26 tables.

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Growth, pests and diseases of the sugar beet in Belgium during 1965. L. VAN STEYVOORT. *Pub. Tech. Inst. Belge pour Amél. Betterave*, 1966, (2), 21-49.—An account of weather conditions is given, the season being less favourable than 1964, with less sunshine. Average yield of roots was estimated at 38.78 kg/ha. The season favoured attacks by millipedes and by *Ditylenchus dipsaci* (on seedlings). Losses due to virus yellows were light owing to late appearance of aphids. These were estimated at 1.2%, as in 1964.

Sugar beet yellows in 1965 (in Belgium): results of trials carried out with systemic insecticides. L. VAN STEYVOORT. *Pub. Tech. Inst. Belge pour Amél. Betterave*, 1966, (2), 51-67.—During the 1965 season aphids appeared late owing to weather conditions and increased little. Consequently systemic insecticides had little effect, confirming the belief that when aphids do not appear until June their use may not be justifiable or economical.

* * *

Problems of maintaining soil fertility with special regard to sugar beet cultivation. A. HERKENRATH. *Zucker*, 1966, 19, 66-70.—The writer points out that beet growers in Germany make good use of the wide range of fertilizers produced by German manufacturers but do not always, in his view, make sufficient use of lime, especially on the heavier soils. The matter is discussed.

* * *

Taxonomy, morphology and anatomy of *Saccharum* and varietal identification. U. VIJAYALAKSHMI. *Coimbatore Summer School for Sugar Cane Scientists, Summary of Lectures*, 1966, (2), 5 pp.—The five species of *Saccharum*, as at present recognised, are briefly described and the importance of morphological characters of leaf and stalk stressed. The value of certain anatomical characters in classification and in disease or pest resistance is discussed. The number of improved sugar cane varieties now in commercial cultivation in India is over 30. Six of these are approved varieties for Madras and Kerala States. The diagnostic characters of these six are described and a key for their identification given.

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Sugar cane breeding. J. THULJARAM RAO. *Coimbatore Summer School for Sugar Cane Scientists, Summary of Lectures*, 1966, (3), 4 pp.—The history of sugar cane breeding in India is briefly sketched and the present importance of different commercial varieties referred to. This is followed by a discussion of breeding objectives, breeding techniques and breeding systems.

* * *

Sugar cane genetics. S. S. SHAH. *Coimbatore Summer School for Sugar Cane Scientists, Summary of Lectures*, 1966, (4), 2 pp.—Many cultivated sugar cane varieties are inter-specific hybrids. The genetics of sugar cane is complex for several reasons. These are discussed. Nevertheless the genetics of several characters has been studied. Juice quality is important, having been derived from *Saccharum officinarum* in the case of cultivated varieties. Location may be important in regard to juice quality.

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Sugar cane physiology. K. M. NAIDU. *Coimbatore Summer School for Sugar Cane Scientists, Summary of Lectures*, 1966, (6), 4 pp.—The life cycle of a sugar cane crop may be regarded as consisting of four phases: germination, tillering, grand period of growth and the ripening phase. These are discussed in turn. Nutrition, water requirements, metabolism and flowering are also considered.



Sugar - House Practice

Cartier's new refinery serves Canada. R. H. DANON. *Sugar y Azúcar*, 1966, **61**, (5), 52-53.—Information is given on the processes and equipment used at the Montreal refinery of Cartier Refined Sugars Ltd. The refinery supplies primarily industrial and commercial users and also produces liquid sucrose and liquid invert. It is designed to produce 150 tons of sugar daily, but output exceeds this figure.

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Lusita's modern techniques cut costs. ANON. *Sugar y Azúcar*, 1966, **61**, (5), 55.—Details are given of the processes and equipment used at the Lusita refinery of José Cojuangco & Sons Inc. at San Miguel, Tarlac, Philippines. This Stork-Werkspoor refinery uses a 3-massecuite system and has a rated capacity of 250 metric tons of brown sugar per day, with a possible increase to 750 tons/day.

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Segura's accelerated natural circulation evaporator. J. FERNÁNDEZ C. *Sugar y Azúcar*, 1966, **61**, (5), 56-59.—Details are given of the Segura evaporator¹.

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Separation processes in sugar refining. TATE & LYLE REFINERIES LTD. *Filtration & Separation*, 1965, **2**, 364-368; through *S.I.A.*, 1966, **28**, Abs. 110.—The sugar refining process as currently used at Plaistow refinery is described with special reference to the carbonatation process and to the bacteriological filtration of liquid sugar. Carbonatated liquor is filtered in leaf filters, preferably of the rotary vertical-leaf type at a normal rate of 3.5 gal/sq.ft./hr. The chalk cake is sluiced out when compact and is sweetened-off in filter-presses, the sweet water being used for melting. Bacteriological filtration is carried out either in vertical-leaf type pressure filters using CaSO₄ as filter-aid, or in horizontal-leaf filters using a kieselsol-perlite-asbestos pre-coat. Some disadvantages of existing continuous centrifugals with regard to separation are briefly considered. A flow diagram of the process is given.

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The problem of frozen cane in the Argentine. W. E. CROSS. *Sugar J.* (La.), 1966, **28**, (10), 8-14.—The nature of the chemical changes that take place in frozen cane is discussed and the difficulties in factory processing associated with such cane, especially as a result of dextran formation, are considered. Measures for dealing with the difficulties are recommended. They include addition of formalin to prevent excessive fermentation, particularly in the juice pans under the mills, adding sodium carbonate to neutralize the acid juices, and boiling the juice vigorously for a few

minutes in open pans to improve settling and filtration. A second sulphitation of filtered clarified juice will reduce viscosity, as will phosphoric acid solution added to the vacuum pans. Should it be impossible to boil 2nd and 3rd strikes, 1st product sugar only should be produced and the molasses used for alcohol production, if the factory has a distillery, as is usual in Argentina. Special steps to take in juice analysis are also recommended.

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Some thoughts on the reduction of extraneous matter. A. G. CLAIRE. *Proc. 33rd Conf. Queensland Soc. Sugar Cane Tech.*, 1966, 25-27.—A scheme is suggested in which the amount of dirt and trash accompanying cane into the mill would be reduced. It assumes mechanical cutting and loading of the cane, which would be tipped from the trucks onto a feeder table and passed via an aligning drum, if necessary, to the knife station. The feeder table and drum would have slots and holes through which the dirt and trash would fall. The knife station would cut the cane into short lengths which would then fall onto a slotted conveyor taking the pieces to the main carrier. The separated dirt and trash would be collected and weighed in order to obtain a correction factor to be applied to the cane gross weight. After the cane trucks had passed over the weighbridge, certain ones would be tagged for sampling, giving better representation of the bulk of the cane than would a grab sample. Since these trucks would be treated separately, the cut cane en route to the main carrier could be washed with high-pressure water jets. In an alternative scheme, after the weighbridge all the cane would be tipped out, passed by elevator to the aligning drum and cut into short lengths which would then be delivered to one of a number of bins on rails. These would move round a closed-loop circuit during which they would be weighed on another bridge, and the cane emptied onto the main carrier. The identity of the sample trucks would be maintained.

* * *

Operating bulk sugar installations with rubber-tyred loaders. P. T. CAPRA. *Proc. 33rd Conf. Queensland Soc. Sugar Cane Tech.*, 1966, 35-40.—Two rubber-tyred loaders, one with a 2-cu.yd. bucket and the other with a 5-cu.yd. bucket, transfer sugar from the bulk warehouse at Mackay at a combined rate of 666 tons of sugar/hr. being occasionally assisted by one face shovel. The machines operate both as bulldozers and loaders, and their advantages in cost and flexibility over the face shovels they have replaced are discussed.

¹ See *I.S.J.*, 1966, **68**, 118.

Some effects of sour storage rot on cane juice quality.

B. T. EGAN. *Proc. 33rd Conf. Queensland Soc. Sugar Cane Tech.*, 1966, 11–20.—Information from 4 years' investigations on the effects of cane sour storage rot has been collated and an analogy drawn with the effects of freezing of cane in Louisiana. Both whole-stalk and chopped-up cane was examined. It was found that sour storage rot causes a substantial drop in pH, probably as a result of acetic and lactic acid formation, causes rather large increases in titratable acidity, results in considerable polysaccharide formation (probably dextrans), considerably increases the reducing sugar content, and causes substantial drops in pol and purity. Of these factors, the most important is considered to be formation of gum, since this is the main source of difficulty in juice processing. Thirty references are given to the literature.

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The collection and disposal of flue dust and grits from bagasse-fired sugar mill boilers.

J. C. KILLEEN. *Proc. 33rd Conf. Queensland Soc. Sugar Cane Tech.*, 1966, 41–48.—The causes of increase in bagasse furnace *grit and dust are discussed and the characteristics of the component particles briefly considered. Dust collector dynamics and precautions to be taken with the collectors are discussed and extraction and disposal methods described. Among the extraction methods are mechanical and pneumatic extraction from hoppers, while the disposal methods include adding the flue dusts to the boiler ash system and sluicing with water. The adverse effect of dirt particles brought in with the cane on dust collectors, etc. is mentioned.

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Steam turbine lubricants.

R. F. HOLMES. *Proc. 33rd Conf. Queensland Soc. Sugar Cane Tech.*, 1966, 49–55. The requirements of a high-quality turbine oil are discussed, including lubricating quality, viscosity, heat conductance, oxidation stability, rust inhibiting, demulsifying and anti-foam properties, and a sufficiently high ignition temperature. A discussion of turbine oil specifications and service tests is followed by a brief consideration of new developments in turbine oil.

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Developments in the combustion of liquid fuels.

A. C. GATTEGNO and C. J. SANDO. *Proc. 33rd Conf. Queensland Soc. Sugar Cane Tech.*, 1966, 57–67.—The general principles of oil firing for steam generation are discussed and the various types of burners available described, including mechanical (pressure jet and rotary cup) and twin-fluid types. The design of air registers is briefly discussed and the advantages of the parallel-throat design mentioned. More modern developments discussed include gas recirculation, and high-intensity, stoichiometric and two-stage combustion.

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Factors in clarification.

J. P. KOMEN. *Proc. 33rd Conf. Queensland Soc. Sugar Cane Tech.*, 1966, 97–102. Some of the factors in juice clarification are considered. Four categories of liquid-solid separation are discussed: (i) Class I clarification, in which each

particle maintains its entity during settling; (ii) Class II clarification, in which the particles coalesce or flocculate during settling; (iii) zone settling, in which interparticle forces are strong enough to drag each particle along in the same relative direction; and (iv) compression, in which the particles are supported by layers of solids below. Detention efficiency (defined as effective detention time divided by nominal detention time, where the former factor is the time it would take in a batch operation to produce a degree of flocculation equal to that actually obtained in a given continuous basin and the latter is the time required to pass, at a given feed rate, a volume equal to the volumetric capacity of the basin) and sedimentation volume are two controlling factors also considered. Emphasis is placed on the proper treatment of the juice before it is fed to the clarifier in order to achieve a maximum settling rate.

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Mud concentration within a clarifier tray.

K. J. NIX. *Proc. 33rd Conf. Queensland Soc. Sugar Cane Tech.*, 1966, 103–110.—The concentration of mud in the juice was measured at 12 sampling points within tray C (3rd compartment from the top) of a Dorr A.T.V. clarifier of 24 ft diameter and 59,000 gal capacity. A high mud level was maintained in the compartment so that as many sampling points as possible were "dirty". Clarifier feed, clarified juice at each overflow weir and primary mud were also sampled. Flow rate measurements of clarified juice and primary mud for each tray were taken before and after sampling. The results are tabulated and given in the form of mud concentration profiles in tray C. A graph showing mud concentration variation across the tray is also given. From the tests, it is concluded that the feed entering each tray mixes with partially settled mud before moving out over the tray to be clarified. Hence, the profiles of mud solids concentration were essentially horizontal. However, there is insufficient information on the settling mechanism to justify any change in the design theories.

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Pilot plant diffusion experiments.

D. H. FOSTER and J. W. HILL. *Proc. 33rd Conf. Queensland Soc. Sugar Cane Tech.*, 1966, 111–119.—The diffusion process in which liquid is applied to the top side of a moving bed of bagasse was simulated by an arrangement incorporating a 6-ft high, 15-inch diameter vertical tower in which about 75 lb of loosely packed bagasse was irrigated with a series of artificial juices. These were prepared in steel drums and were initially heated by direct steam injection, then maintained at a constant temperature before being pumped to the top of the tower. After the juices had been applied and the bed allowed to drain, the tower was tipped and the bagasse removed, weighed and sampled; a sub-sample was pressed to give moisture contents of 45–50%. The extraction times were 20, 24, 30 and 36 min. Temperatures of 80° and 90°C were used, except in one case, where 95°C was used for cell killing. At mean particle thicknesses in the range 3.7–6.9 mm, the extraction from 1st mill bagasse varied from 82.81 to 93.73% (mean of approx. 88%) in bagasse. The

overall extraction increased with reduction in particle thickness (by 2.1% with a reduction from 6 to 4 mm), with rise in temperature (by 0.4% with a 10°C rise) and with increase in retention (by 0.65% with a 10-min increase). Experiments on 2nd mill bagasse extraction at three factories gave results generally in the range of 80–85% pol in bagasse, giving an overall extraction of 96–97%. Tests were also conducted on bagasse from various cane varieties to determine their effect on bed permeability, which fell steadily with decreasing average particle thickness. Those canes with tough vascular bundles gave bagasse permitting consistently higher flow rates than did the soft varieties. The juice content of a bed was found to have a significant effect on the amount of juice required to wet the bed completely, and varied from 600% on fibre when well drained to 1200% when flooded.

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Cane shredder hammer performance. D. S. SHANN and W. MCWHINNEY. *Proc. 33rd Conf. Queensland Soc. Sugar Cane Tech.*, 1966, 151–156.—A number of hammer sets (each containing 4 hammers) of various compositions and treatments were installed in a Searby shredder and their performance evaluated on the basis of mass loss. Results of three separate investigations are discussed. The four main categories were: (i) solid alloy hammers, including chromium carbide types; (ii) hammers having a mild steel base with insert; (iii) hammers with an arc- or flame-applied high chromium carbide deposit; and (iv) hammers with miscellaneous arc- and flame-applied deposits, including tool-steel types and those with tungsten carbide deposits. The solid alloy hammers and those with alloy inserts of the high chromium carbide type suffered smaller mass loss than those with deposited materials, while the types with the high chromium carbide deposit were the best in the deposit-type category. On the basis of the results, Racecourse mill, where the tests were carried out, has installed 9-lb hammers with flame-applied chromium carbide deposits. As a result, the weekly refurbishing time has been reduced by 50% compared with that previously required. The comparative economics have not been evaluated.

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Some thoughts on milling. J. A. MCGINN. *Proc. 33rd Conf. Queensland Soc. Sugar Cane Tech.*, 1966, 173–178.—Cane milling control is discussed under two main aspects: rate control of the milling train, and control of the individual mill. The first of these includes metering of the fibre rate at No. 1 mill, which system produces a constant fibre rate at constant mill speed, and metering of the cane rate at the carrier, which method is a development of the mechanized carrier system, whereby the cane crushing rate is regulated according to the weights of individual cane trucks as measured at the weighbridge. Each truck would be discharged over a period of time proportional to its net weight, while the cane carrier system would run at constant speed, the first mill being controlled as the other mills in the train. Individual

mill control is sub-divided into constant speed control and killer plate control. Constant speed control has the disadvantage of possible failure in feeding at too low a speed and consequent damage to the pressure chute, if one is used, while the advantage of speed reduction in improving extraction is the main advantage. The choice of a compromise speed is therefore required, although it is pointed out that wide variations in the fibre rate will necessitate choosing a high speed. The disadvantages of killer plate control, where the blanket thickness is used to regulate engine speed, are given as: severe speed fluctuations caused by control instability, inadequate sensing and control of the mill fibre loading, and risk of mechanical damage to the pressure chute. Various devices which might help to overcome the difficulties are described.

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Circulation movements in sugar vacuum pans. P. G. WRIGHT. *Proc. 33rd Conf. Queensland Soc. Sugar Cane Tech.*, 1966, 179–184.—Circulation in vacuum pans was studied by means of a radio-active capsule inserted into the massecuite, the movement of the capsule being followed by gamma ratemeters. Of the types of pan used for high-grade boiling, the best for circulation (i.e. with high percentage of true circulation movement, a minimum of stagnation and a maximum circulation and eddy speed) was one with a central downtake calandria, a streamflow bottom, circumferential steam distribution and low head above the top tube plate. This was better than other central downtake calandria pans and was also better than two floating calandria pans as regards the percentage of true circulation movement. Of the various types of low-grade pans studied, no conclusions could be drawn, but it is suggested that if the circulation characteristics of the best high-grade pan mentioned above were as good as those of the two coil pans tested, it would be the most economical design for both high- and low-grade boiling. The investigations suggest that the annular downtake is not as efficient as a large downtake, since the heating process in the calandria tubes is not continuous but involves a period of relative stagnation of the massecuite, possibly with a slight downward movement, until sufficient superheating of the heated layers occurs, whereupon a large burst of vapour erupts and ejects the contents. That part of the vapour which does not collapse when it meets the cooler massecuite above will reach the surface and result in flashing. Fresh massecuite enters the tube from above as well as below, and the greater the proportion of massecuite from above the greater will be the reduction in true circulation and the greater will be the increase in the downcoming eddies above the calandria. Hence, it appears that the floating calandria is less favourable to ideal circulation but depends more on the eruptive boiling and return by eddy currents above the tube plate. While a steam jigger helped increase circulation, louvres increased the tendency for short circuiting to the tube elements, while retarding downward movement through the extra frictional drag surfaces.

BEET FACTORY NOTES

The Findlay flume and condenser water system. S. L. FORCE. *J. Amer. Soc. Sugar Beet Tech.*, 1965, **13**, 478-491.—In the system introduced in 1956 at the Findlay beet factory of The Great Western Sugar Co. in Ohio, the flume water is separated at the beet wheel and is pumped to a liquid cyclone for grit separation. The over-flow passes over Link-Belt vibrating screens, which remove light solids, tops, weeds, etc., and is then treated in two Dorr clarifiers, each measuring 63 ft 8 inches in diameter \times 12 ft high. The water is retained 6½ min, after which it is pumped to the spray pond, which is provided with four headers, each feeding 30 spray nozzles 15 ft apart, where the temperature of the water is reduced by an average of 21°F. The water is then returned via an excess water pond to the main water "sump" which pumps it to the "main water tank" supplying all the condensers and other stations not requiring clean water. The condenser water is collected in sealed tanks and piped to the "flume water tank", whence it is pumped to the flumes. Water accumulating in the clarifier sludge pond and the lime mud pond is re-utilized in the system to prevent excessive accumulation of water from these sources. Data obtained with the system are tabulated and advantages and disadvantages of the scheme noted.

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Ion exclusion purification of molasses. J. B. STARK. *J. Amer. Soc. Sugar Beet Tech.*, 1965, **13**, 492-502. At 90°C, "Dowex 50 W" resin in K⁺ form (X-4 cross-linkage) separated 50% of the sucrose in molasses by static ion exclusion. On a commercial scale these fractions, having purities of 80 or higher, could be returned to intermediate pans. More than 65% of the sucrose in cane molasses was eluted at a purity of at least 68, compared with a maximum purity of 48 using the X-12 cross-linkage form of the resin. Fractions of intermediate purity could, it is suggested, be used to dilute fresh molasses and then recycled on the column. The method was found to be also applicable to invert recovery from cane molasses.

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Problem of juice deliming. S. ZAGRODZKI. *Gaz. Cukr.*, 1966, **74**, 25-29.—The relative proportions of anions and cations in beet juice at various stages of purification (from raw to second carbonation juice) are shown diagrammatically, expressed in normalities, and discussed. The successive reactions taking place in 2nd carbonation after CO₂ addition are given in equation form and the effects of various factors on deliming considered, in particular exchange of the CO₃⁻ anions for OH⁻ anions when the pH falls, with dissolution of the resultant Ca(OH)₂. In this connexion, determination of the optimum alkalinity or pH in 2nd carbonation is discussed and various methods are described. The adverse effect of over- and under-saturation is considered. Laboratory tests have shown that a Ca salts content exceeding 70 mg CaO/100°Bx will lead to an increase in the rate

of scale deposition on evaporator tubes, while below 40 mg CaO/100°Bx some of the scale will dissolve in the juice. At a Ca salts content below 17 mg CaO/100°Bx there is risk of corrosion of the heating surface. A brief survey is presented of supplementary deliming methods.

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Processing of beet tails in the Polish sugar industry. J. LEKAWSKI and S. RZUCHOWSKI. *Gaz. Cukr.*, 1966, **74**, 30-32.—Data from all 77 Polish sugar factories on beet tail processing are summarized. These cover the proportion of tails to beets, the methods used to convey the beets and tails to the factory, equipment used in handling the tails, difficulties in tail processing, etc. Only 26 factories processed all the tails, while 35 did not process them all, and the others only partially.

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Evaluation of waste waters from washing of sugar factory (filter) cloths. K. SKALSKI. *Gaz. Cukr.*, 1966, **74**, 32-35.—The quantity of waste water emanating from filter cloth washing has been found by various authors to vary within the range 4-15% on beet. The characteristics of this waste water, according to various authors, are tabulated and the N, P and K contents compared with those of flume water and the average contents in sugar factory waste water. The N and K contents are much higher than in the other waste waters, while the P content is lower. Methods of purifying the waste water are discussed.

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Extension of Leśmeriz sugar factory. I. HARGESHEIMER and Z. RAKOWIECKI. *Gaz. Cukr.*, 1966, **74**, 35-37. Details are given of the new equipment installed in this white sugar factory to raise its capacity. In 1964/65 the factory produced a total of over 14,700 tons of sugar during a 101-day campaign, with a daily beet slice of over 1100 tons. Molasses yield was 3.24% on beet and molasses purity 60.2, while total losses were 3.25% on beet. The final aim is a daily slice of 2000 tons of beet.

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Thermal calculation of beet pulp drying. III. Reduction in fuel consumption. T. BALOH. *Zucker*, 1966, **19**, 190-197.—It is shown with the aid of graphs and a Sankey diagram that the fuel consumption in beet pulp drying can be reduced to as little as 700 kcal/kg of water by using boiler flue gases. The saving in fuel will be higher the greater is the amount of flue gas used and the higher its temperature. In contrast to conventional drying, the fuel consumption will be lower with lower input temperature of the gases. The use of flue gas is also claimed to reduce fluctuations in the heat economy of a sugar factory. The fuel consumption can also be reduced by using gas circulation, which at high temperatures (800-100°C) will give the same reduction in fuel consumption as will the use of boiler flue gas. However, with increased quantities of circulation gas and correspondingly

lower input temperatures, the reduction in fuel consumption will not be as great as with flue gas usage, since some of the saving in fuel will be offset by the greater energy requirements for gas circulation.

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Process optimization even in the sugar industry?

W. DIEKERS. *Zucker*, 1966, **19**, 197-202.—The subject is dealt with under four headings: (i) stages in process automation and automatic process optimization; (ii) advantages of using process computers for sugar factory operation; (iii) costs of an automatic process optimization unit with process computer; and (iv) step-by-step methods of realizing an optimization programme. Diagrams are given showing an on-line, open-loop computer for evaluating the measuring value, calculating the characteristic quantity, etc., an on-line, closed-loop computer for forward optimization and for backward optimization, respectively, and a direct digital control.

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Optimal crystallization by cooling. D. SCHLIEPHAKE. *Zucker*, 1966, **19**, 219-225.—The possibility of optimizing crystallization by cooling (as opposed to evaporation) is examined using the technique of computation of variance. It is assumed that the crystallization rate can be represented as a first order reaction. Deviation of actual crystallization of sucrose from that calculated by a simple formula is discussed qualitatively. It is shown by means of time-temperature curves that optimal crystallization (a maximum quantity of sugar obtained in the shortest possible time) is possible only if the required supersaturation remains within the metastable zone; should the supersaturation exceed a certain upper limit, fine grain will be formed. Hence, if the limit curve for fine grain formation lies above the calculated optimal curve, the calculated optimum is not feasible. Laboratory studies and factory practice showed that the crystallization rate will always be limited by fine grain formation, so that the above-mentioned calculation cannot be used for optimization. Comparison of the calculated optimal curve with the fine grain formation limiting curve demonstrates the importance of the degree of dispersion of the crystal surfaces presented during crystallization. With a fine dispersion surface it is possible to approach the optimal curve more closely than with a coarse dispersion. While mass transfer results in slower crystallization than with a pure surface reaction and tends to push the optimal curve up towards the limit curve, the effect of the growing surface will be to push the curve down again as a result of increase in the de-sugaring rate.

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Importance of limestone and coke distribution in shaft lime kilns. J. MRÁZEK. *Listy Cukr.*, 1966, **82**, 88-93. Lime production in sugar factories is briefly considered and feeding of limestone and coke into shaft kilns discussed. A new type of feeder is described, use of which at one Czechoslovak sugar factory resulted in reduced coke consumption, increased yield and quality of lime and elimination of risk of under-filling.

Plate heat exchangers in the sugar industry. A. BIELER. *Zucker*, 1966, **19**, 249-252.—The advantages of plate heat exchangers are discussed and possible applications in the sugar industry considered. Methods of cleaning the heat exchangers are also dealt with.

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Observations on a beet pulp drum dryer heated by waste gas. II. E. LEBEDA, L. NESVADBA and W. VON PROSKOWETZ. *Zeitsch. Zuckerind.*, 1966, **91**, 263-266. Further tests were carried out during the 1965/66 campaign to determine various factors involved in pulp drying in a gas-fired drum at Bruck a.d. Leitha white sugar factory, Austria. The results are compared with those from the previous campaign¹. From pressed pulp of 81% moisture content 9,802 kg of water, i.e. 96.77% of that in the pressed pulp, was evaporated per hr at a dry pulp throughput of 2,712 kg/hr. These figures compare with the 1964/65 values of 79%, 7,705 kg of water evaporated per hr (96.3% of the pressed pulp moisture content), and 2,432 kg dry pulp throughput/hr.

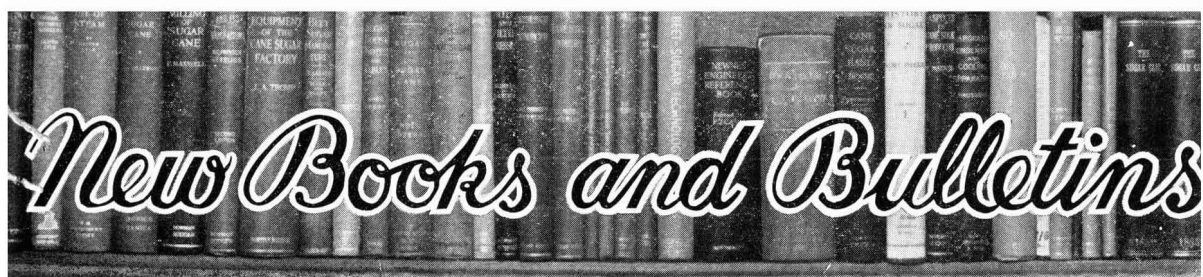
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Control of mass change during sugar beet storage by determining the content of invariable constituents, particularly potassium. H. D. WALLENSTEIN and G. KAGELMANN. *Zeitsch. Zuckerind.*, 1966, **91**, 189-194, 266-271.—Direct and indirect methods of determining the weight change in stored beet are described and their disadvantages noted. Determination of the potassium content as an indirect measure of weight change was carried out by drying a 20-g sample in a vacuum oven to constant weight, determining the dry solids content and adding conc. H_2SO_4 dropwise in sufficient quantity to moisten the sample. The sample is then held over a small flame until a loose, flaky ash is obtained. This is added to 20 ml of 1N H_2SO_4 and 150 ml water in a beaker and heated at boiling point for 2 min. After cooling, the sample is filtered into a 200-ml cylinder and made up with water, the K content of this solution being determined by flame photometry. The result, in g $K_2O/100$ g beet is given as $200 (K_2O/E)$, where K_2O is the weight in mg read off a standard curve, and E is the weight of the brei in mg. Accuracy in the measurement of two solutions of known K content (19 measurements per solution) was to within 0.5-0.6% and mean reproducibility 0.65%. It is necessary to consider the effect of dirt which is not completely removed even when the beet is washed. Any dirt adhering to the rasp may also be included in the brei. The corrected value

of K_2O is given by $\frac{100 \cdot K_2O_B - S \cdot K_2O_S}{100 - S}$, where

K_2O_B and K_2O_S are respectively the measured K content of the brei and the residual dirt and S is the dirt content of the brei. Tests on full-scale piles at two factories gave a reproducibility of sugar loss determination greater with K_2O determination than with most weighing methods.

¹ See *I.S.J.*, 1965, **67**, 376.



Zuckertechniker-Taschenbuch (Sugar Technologist's Pocket Book). E. WERNER. 501 pp.; $4\frac{1}{2} \times 7$ in. (Verlag Dr. Albert Bartens, Lückhoffstr. 16, Berlin-Nikolassee, Germany.) 1966. Price: DM 42; £3 16s 0d.

This is the 7th edition of a book first published in 1940 under the title "Zuckertechniker-Kalender". The 6th edition appeared in 1956, and the present one is some 170 pages longer than that edition, with a slightly larger format. It contains 220 diagrams and 350 tables and is divided into nine sections: mathematics, physics and chemistry; sugar technology; sugar factory control; machinery and equipment; heat engineering; materials strength and building; electrical engineering; lifting and conveying gear; and measuring and control engineering. The sugar technology section of 103 pages is sub-divided into sections on sugar and its properties (chemical and physical properties of the leading sugars, solubility of sugars and various substances in sucrose solutions, sucrose solution densities and concentrations, specific rotations, etc.), sugar recovery from beet, cane and palms (information on the various processes in sugar manufacture), and water and waste water treatment. The factory control section, containing 68 pages, has as its sub-sections one on analytical factory control, covering sampling and analysis techniques for raw material, intermediate products and end products, a section on microbiological control and a third one on mathematical control, with the basic equations needed for drawing-up of balances. The other main sections concern the mechanical, chemical and civil engineering aspects of sugar factory operation. The 24-page index is well set out. The book is well printed and offers a wealth of data on sugar technology, presented in a very clear and concise manner. It has been prepared in collaboration with some of the leading German specialists in the field and the reviewer has no hesitation in recommending it. It must be remembered, however, that the book is in German; but this should not deter the more ambitious sugar technologist with a different mother-tongue.

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Sugar Year Book 1965. 354 pp.; $3\frac{1}{2} \times 5\frac{1}{2}$ in. (The International Sugar Council, 28 Haymarket, London S.W.1.) 1966. Price: £2 0s 0d.

The 19th edition of this publication contains sugar statistics for 122 countries (nine more than in the last edition), in most cases covering the period 1959-65. The data are submitted by member countries of

the International Sugar Agreement under the Rules of the Agreement, while those of non-member countries are either supplied by the governments concerned or are extracted from statistical publications. They cover world centrifugal sugar production in metric tons (expressed, where possible, as 96° pol raw sugar) tabulated by countries in alphabetical order. More general information is also given, including world sugar production, imports, exports and consumption (absolute and per caput), sugar stocks in selected countries, world sugar prices 1952-65, British Commonwealth Sugar Agreement export quotas 1965 and 1966, U.S. sugar quotas 1964 and 1965, white refined sugar retail prices in selected countries 1964-66 and equivalent weights and measures. Despite the steep rise in the price of the book, which has doubled since the previous edition, it is still a valuable asset for those needing a handy source of world sugar data.

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Zucker-Jahrbuch 1965/66. (Sugar Yearbook.) Ed. H. HAHN. 960 pp.; $6 \times 8\frac{1}{2}$ in. (Verlag Alfred Röper, Schlüterstr. 56, Hamburg 13, Germany.) 1966. Price: DM 32.-; £2 18s 0d.

The latest edition of this international sugar yearbook is in the same form as previous editions, with the information brought up to date. The sections include addresses of the world's sugar factories and trade organizations. Unfortunately, as is pointed out, the data on the East German factories are out of date, despite efforts to obtain more recent information. The Soviet Union is represented only by the food import and export organization, Prodintorg, and the section on the Chinese factories gives only a selection of three factories and one refinery. On the other hand, the data on the West German industry is very detailed, with information on the equipment at most of the factories. The remainder of the book contains details of yeast plants, sugar industry chemists and sugar sampling chemists, trade concerns, importers, exporters, etc., national and international organizations, authorities and institutes, regulations governing sugar and beet in West Germany, and two surveys by Dr. DANKOWSKI of the world sugar market and the sugar economies of EEC and EFTA. Information on West German sugar equipment manufacturers is followed by a Buyers' Guide. The list of the contents is given in English and French as well as German, as are the names of the countries. The book is well presented, but its importance lies more in the detailed information on the West German industry than in the data on the rest of the world.

Laboratory Methods and Chemical Reports

Masseccuite flow. A. I. GROMKOVSKII and S. Z. IVANOV. *Sakhar. Prom.*, 1966, **40**, (4), 17-23.—The deformation of an artificial masseccuite of known crystal content as a contribution to flow studies was determined with a modified rotary viscometer. The masseccuite was thoroughly mixed in the outer cylinder replacing the thermostat vessel and the central rotary cylinder immersed in the masseccuite concentrically with the outer cylinder. The angle of rotation of the gauge plate between the two cylinders was measured after the inner cylinder had been rotated through 360°, thus giving a measure of the slip occurring. A theoretical deformation curve for a Newtonian liquid was plotted. Similar curves (angle of rotation of the plate vs. cylinder radius) were plotted for molasses and for the plate after a given number of rotations. While the molasses curve coincided with the theoretical curve, the curves for the masseccuite diverged from the theoretical curve by an amount increasing with the number of rotations. Maximum divergence occurred at the 11th rotation, after which the curves were identical as was the time of rotation, which dropped with each rotation. The pattern of the curves was found to be largely a result of slip of the inner cylinder, and, to a lesser extent, a result of crystal deposition. Masseccuites containing up to 58% crystal by volume (1st and low-grade masseccuites) exhibited greater slip as the crystal content increased. At crystal contents of about 60% the degree of slip was unity, i.e. masseccuite flow, if any, was a result of slip and there was no displacement between the layers in the viscometer cylinder. Hence, masseccuite flow can be regarded as not belonging to any known classic form of flow, but is nearest to Newtonian flow.

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The significance of aerobic and anaerobic thermophilic spore-forming organisms as sources of infection in beet sugar factories. III. The level of sucrose losses caused in sugar factory juices by aerobic and anaerobic spore-forming organisms. H. KLAUSHOFER and E. PARKKINEN. *Zeitsch. Zuckerind.*, 1966, **91**, 125-130. Four species of thermophilic organisms were cultured on two substrates and in juices from various sections of a tower diffuser. Both a continuous and a batch process were used at 66-68°C, i.e. the temperature range applying in diffusion. The sucrose losses occurring during continuous culture, i.e. under conditions obtaining in diffusion towers, were always greater than in batch culture. *Bacillus stearothermophilus* in continuous culture caused 10-100 times as much sucrose destruction as did *Clostridium thermohydrosulfuricum* and *Cl. thermosaccharolyticum*. *Cl. nigrificans* did not cause any sucrose losses.

Thin-layer chromatography of sugar beet carbohydrates. H. G. WALKER, B. A. RICCI and J. C. GOODWIN. *J. Amer. Soc. Sugar Beet Tech.*, 1965, **13**, 503-508. Solutions adjusted to 8-10% dry solids were chromatographed by the ascending technique and the absorbent plates developed first by 65:23:12 ethyl acetate:2-propanol:water and then, after air drying, by 55:30:15 ethyl acetate:2-propanol:water. Total time for the double development was 3 hr. The plates were coated with a slurry made from 10 g each of "Celite" and CaSO₄ mixed with 50 ml of 0.02M sodium acetate. Stahl's indicator (0.5 ml anisaldehyde, 0.5 ml conc. sulphuric acid and 9.0 ml 95% ethanol) was used as spray reagent. Separation of fructose, glucose, sucrose, kestose, raffinose and galactinol in beet juices was good, but appeared to depend on the presence of only extremely small quantity of carbohydrates. When amounts of sugars commonly used in paper chromatography were applied to the plates, good separation was impossible. Semi-quantitative analysis of the sugars was also not sufficiently precise. Hence, the method is considered suitable as an alternative to paper chromatography for rapid qualitative analysis, but further work on thin-layer chromatography for quantitative analysis of beet sugars is required.

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Measuring errors in research and chemical analysis in the food industry. Z. NIEDZIELSKI. *Gaz. Cukr.*, 1966, **72**, 53-57.—Methods of calculating measuring errors in research and food product analysis are described and the fundamentals of error theory explained. Methods of estimating dispersion of results as a measure of their reproducibility are presented, as are the procedures for determining dispersion according to the number of determinations.

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Microbiological examination of white sugar. I. JANUSZEWICZ. *Gaz. Cukr.*, 1966, **74**, 57-59.—A brief survey is presented of the conditions under which bacteria develop in white sugar. The types of micro-organism found in white sugar and the microbiological standards laid down for sugar used for preserves and non-alcoholic beverages are detailed.

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Chromatographic analysis of cane molasses. W. W. BINKLEY. *Zeitsch. Zuckerind.*, 1966, **91**, 195-196. The application of chromatography to cane molasses is reviewed with 46 references to the literature. The column technique has been used for isolation of sugars, sugar alcohols and other constituents, paper chromatography for detection and identification of amino acids and non-N organic acids, and thin-layer and gas-liquid chromatography for rapid quantitative analysis of certain constituents.

Contribution to sugar beet evaluation—formulae for molasses and white sugar yield. L. SCHMIDT, A. HAVRÁNEK and B. VORLÍČKOVÁ. *Listy Cukr.*, 1966, **82**, 81–84.—Formulae for calculating molasses yield and white sugar rendement from pre-campaign analyses are surveyed. Comparison of calculated values with one another and with the true values for the years 1940–1964 show that the best formulae for calculation of molasses yield (M) are those of SMOLENSKI $\left[0.5 M = \frac{4.25 Pp (Dg - 1)}{Dg}\right]$ and ŠANDERA $[M = 8 Pp, \text{ and } M = 11 (Pp - 0.120)]$. For white sugar rendement, the most suitable formula is considered to be $B = Dg - 1.1 - 3.6 Pp$. In the formulae Pp = sulphate ash in thick juice per 100 pol and Dg = digestion (sugar content).

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Long-chain aldehydes of sugar cane wax. J. A. LAMBERTON. *Austral. J. Chem.*, 1965, **18**, 911–913; through *J. Sci. Food Agric. Abs.*, 1966, **17**, i–191. Nuclear magnetic resonance spectra of solutions of sugar cane wax in CdCl_2 at 60° reveal the presence of free aldehydes in a concentration approx. equivalent to that in a solution of freshly distilled wax. Spectra of semicarbazones prepared from undistilled and distilled wax are also similar. The main constituent of the wax is probably an unsaturated aldehyde having a hemihydrate structure and dissociating easily in solution^{1,2}. This aldehyde is also present in sugar factory mud and in the unsaponifiable fraction obtained by heating the wax with ethanolic KOH.

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Comparative composition of crowns and topped beets. P. L. DEVILLERS. *Sucr. Franç.*, 1966, **107**, 123–125. The composition of the five major parts into which individual beets from one batch were divided (periderm, crown, collar, heart and tail) is shown diagrammatically, including the weight %, marc + hydration water content, sugar content and purity. A second batch of beets were dissected in various ways and the above contents determined more precisely, as well as the press juice composition [raffinose, alcohol-precipitable substances (pectins and proteins), acid-precipitable substances (saponin), betaine and amino acids, organic anions and mineral cations]. The tabulated results show that if the crown has a higher marc content and lower sugar content than the root, it will also have higher contents of raffinose, precipitable substances, betaine and amino acids. The same also applies, to a lesser extent, to the periderm. There was little difference between the middle section of the beet and the tail. The average weight of the crowns was 21% of the roots, while their sugar contents were 2% lower (absolute) and their marc contents 2% higher than those of the roots. The crown purities were much lower, their contents of reducing sugar, alcohol-precipitable and soluble N compounds almost double, while their acid and metal ion contents were only slightly higher. While the impurities content was approximately double that of the root, the purified juice from the crown was of 90 purity.

Determination of the water content of beet raw sugars by drying at 100–120°C and atmospheric pressure. W. SCHIEBEL. *Zucker*, 1966, **19**, 245–249.—Samples (10 g) of 1st and 2nd product sugar from the 1965–66 campaign were dried in a desiccator at 100, 110 and 120°C for a period in the range 1–4 hr. The weight loss in drying was plotted for both products. During drying the weight loss increased linearly with time and with rise in temperature. Differences in the weight losses between samples of the same sugar were generally small, although not so between the different sugars; it is suggested, however, that sugars from other campaigns and having other non-sugar contents than those tested would not behave differently from those tested. Raw sugar samples (1st product) were kept in open air for 2, 3 and 16 days. Those that had not been dried lost moisture while those that had been dried absorbed some moisture. The moisture content of samples dried at 100°C reached practically the same level as did the undried samples. On the basis of the tests, a temperature of 110°C and a drying time of 2 hr are suggested as optimal for both 1st and 2nd product sugars.

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Changes in volume of sugar beet slices in buffer solutions—effect of storage temperature. H. J. DELAVIER. *Zeitsch. Zuckerind.*, 1966, **91**, 262–263. Further tests³ were conducted on 8-mm dia. samples cut from the middle sections of beets and stored under controlled conditions for 14 days at 0, –10 or –24°C. The residue from the middle section was mashed and its water content determined. After storage the slices were immersed in a buffer solution of pH 6 and 60°C and changes in volume during the treatment period (up to approx. 115 min) determined using the same method as before. There was no significant difference between these and the earlier results. Other slices were heated for 15–90 min in a buffer solution of pH 5 and 70°C, and the volume measurements related to the results obtained with the beet stored at 0°C, given as 1. For up to approx. 70 min the volume change in the slices stored at 0 and –10°C was identical, after which the beet stored at the lower temperature underwent a greater volume reduction. The volume change in the slices stored at –24°C was smaller than with the slices stored at 0°C for up to 70 min, after which the volume fell sharply in step with that in the slices stored at –10°C, although to a lesser extent. On the basis of the results, it is concluded that cosettes subjected to diffusion conditions of pH below 6, a temperature exceeding 60°C and a retention time greater than 60 min can be expected to suffer a volume reduction of up to 10%. However, should the moisture content exceed 30%, a volume increase can be expected with heating and/or at a pH of 5–6 or greater than 6. The volume of cosettes from beets stored at 0°C or below without moisture loss will

¹ See *I.S.J.*, 1961, **63**, 59.

² KLASS *et al.*: *J. Org. Chem.*, 1963, **28**, 3029.

³ See *I.S.J.*, 1965, **67**, 250.

fall as the process time increases. A possible explanation of the basic cause of the volume change, involving the beet tissue, is given.

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Chemical composition of Egyptian cane molasses. E. E. M. TAHA, K. WOGER and D. A. ZAKI. *J. Chem. U.A.R.*, 1963, **6**, 163-179; through *S.I.A.*, 1966, **28**, Abs. 126.—Fairly detailed analyses of molasses from three factories and one refinery are given; the constituents of the ash and some trace elements are included. Individual sugars, organic acids, and amino acids were analysed qualitatively only, using paper chromatography. Separate determinations of aldose and ketose reducing sugars show an excess of 28-350% of aldoses over ketoses, except at one of the factories where the proportions were nearly equal.

* * *

Rapid method for determination of sulphurous acid in molasses. H. OLBRICH and S. PEETZ. *Mitt. Versuchstation Garungsgewerbe*, 1965, **19**, 83; through *S.I.A.*, 1966, **28**, Abs. 162.—Five ml of molasses are diluted with 5 ml of water, and 15 ml of 25% H_3PO_4 are added. The mixture is placed in a flask connected to a reflux condenser with an attached bulb containing a neutral solution of H_2O_2 (10 ml of 0.3% H_2O_2 and 60 ml of water). Air or gas is bubbled through the molasses for 15 min and the displaced SO_2 is collected in the bulb and oxidized to H_2SO_4 which is determined by titrating with 0.01N NaOH. Free SO_2 is displaced in the cold and bound SO_2 is displaced during heating. The self-decomposing Brazilian molasses¹, analysed by this method, contained 0.008% of free SO_2 and 0.060% of bound SO_2 (maximum limit 0.15% of SO_2).

* * *

Estimation of nitrate and nitrate nitrogen in sugar cane juice. B. A. LAKHDIVE and R. PRASAD. *Current Sci.*, 1965, **34**, 513; through *S.I.A.*, 1966, **28**, Abs. 163. The colorimetric method of BRAY² was adapted to cane juice analysis. One g of Bray's powder (4 g of sulphanic acid, 2 g of α -naphthylamine, 10 g of $MnSO_4$, H_2O , 2 g of Zn powder, 100 g of $BaSO_4$ and 75 g of citric acid) was added to 50 ml of cane juice clarified with lead sub-acetate. The mixture was centrifuged after 15 min and the colour was measured at 525 m μ . The results include nitrate and nitrite N. The latter is separately estimated by adding a powder mixture as above but in which the Zn, $MnSO_4$ and citric acid components are absent; drops of glacial acetic acid are added 5 min before colour measurement. Cane juices contained 0.4-1.9 p.p.m. of N as NO_3 and 0.02-0.4 p.p.m. of N as NO_2 . Gur samples contained 1.3-3.0 p.p.m. of NO_3 N and 0.15-0.20 p.p.m. of NO_2 N.

* * *

Supersaturated aqueous solutions of sucrose. Determination of activation energies from viscosity measurements. N. TIKHOMIROFF and M. CHABAS. *Compt. Rend.*, 1965, **261**, 701-704; through *S.I.A.*, 1966, **28**, Abs. 177.—Viscosities of sucrose solutions of super-

saturations (σ) 1.00-1.70 over the temperature range 20-80°C were measured using a capillary viscometer, and results are shown in graphs. At constant temperature and supersaturation, viscosity increased during the induction time and reached a constant value when crystallization began. The viscosity-temperature curves had a hyperbolic form when $\sigma = 1.00$. For $\sigma = 1.30$ and $\sigma = 1.50$, the curves showed points of inflexion at 58°C and 66°C. For supersaturations 1.50 and 1.70, the curves had a hyperbolic form, in the former case showing a slight point of inflexion at 94°C. Curves of the relation between induction time and temperature were of parabolic form. Minimum times occurred at 58°C for $\sigma = 1.10$ and at 66°C for $\sigma = 1.30$; for $\sigma = 1.50$ a minimum would occur at 94°C; for $\sigma = 1.70$, induction time decreased with temperature and would reach a minimum at ∞ . Activation energies were calculated from the slopes of the curves of $\log \eta$ against $1/T$, using GUZMANN's formula: $\log \eta = \log A + E_a/RT$, where E_a = activation energy. Values found were $E_a = 4250$ cal/mole for $\sigma = 1.00-1.10$ and 7990 cal/mole for $\sigma = 1.30-1.70$, indicating that the process is one of diffusion at low supersaturations, and is a mixed diffusion and kinetic process at higher supersaturations.

* * *

Colouring matter and its formation during sugar manufacture. V. PREY. *Ind. Sacc. Ital.*, 1966, **59**, 55-70.—See *I.S.J.*, 1966, **68**, 29.

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Determination of suitability of granulated sugar for beverages by polarography. R. D. MOROZ and Y. YANKELEVITS. *Proc. Tech. Session Cane Sugar Refining Research*, 1964, 2-10.—Solutions of granulated cane sugar in sterilized bottles were buffered with a citrate solution treated with carbonated water, sealed and observed daily for 14 days for appearance of floc. The results of this floc test were compared with results of each of four polarographic methods: (i) using 26 g of sugar per ml and 0.002N K_2SO_4 electrolyte as proposed by VAVRUCH³, (ii) using 10 g of sugar/ml and a citrate buffer electrolyte, (iii) using 10 g of sugar/ml and 0.001N KCl electrolyte, and (iv) using 10 g of sugar/ml and no electrolyte. While correlation between the polarograms obtained with (i) and (ii) and the floc test results was unsatisfactory, correlation in the case of method (iii) was good. Floc-positive sugars gave a curve with one value of h smaller than 5 μA (h = maximum height as measured from the highest point in the curve to the upper part of the diffusion current) or with more than one maximum in the range 5-6 μA . All other samples were floc-negative. In method (iv) floc-positive sugars were indicated by the absence of maxima and a current greater than 4 μA at a voltage of -3 V or by a maximum at which the h :voltage ratio was smaller than 1. Floc-negative samples

¹ See *I.S.J.*, 1966, **68**, 246.

² *Soil Sci.*, 1945, **60**, 219.

³ *I.S.J.*, 1951, **53**, 84.

gave curves without maxima and a current of less than 2 μ A at -3 V or with a maximum at a $h:V$ ratio greater than 1. It is concluded that method (iii) can be used for assessment of floc components in granulated sugar.

* * *

Review of colour-forming reactions. W. W. BINKLEY. *Proc. Tech. Session Cane Sugar Refining Research*, 1964, 10-14.—The reactions leading to formation of melanoidins (browning polymers) which take place during clarification, evaporation and boiling are briefly described. They include fragmentation of hexoses (D-glucose and D-fructose) to dihydroxyacetone, glyceraldehyde and pyruvaldehyde; condensation of these products with other carbonyl compounds or with glycosylamino acids to yield melanoidin precursors; rearrangement of glycosylamino acids to form a highly-reactive structure which can change readily to other glycosylamino acids and react further to become melanoidin precursors; and a LOBRY DE BRUYN-VAN EKENSTEIN rearrangement between D-glucose, D-fructose and D-mannose. It is pointed out that while some melanoidins are formed during clarification, the most significant production takes place during boiling and re-boiling, where the reactants are in higher concentrations.

* * *

The pH sensitivity of sugar colours and ease of decolorization. N. H. SMITH. *Proc. Tech. Session Cane Sugar Refining Research*, 1964, 14-26.—Changes in the pH sensitivity of colouring matter during refining were studied in laboratory tests. The indicator value (I.V.), i.e. the ratio of the attenuation of raw sugar liquors at pH 9 to that at pH 4, was used as a measure of pH sensitivity. The results, expressed in graph form, indicate that generally the colour of washed raw liquor at pH 7 was inversely proportional to the I.V., and higher amounts of colour remaining after bone char treatment corresponded to lower I.V. Since the I.V. increased by an average of 60% in affination, by an average of 10% in phosphoric acid defecation and increased during crystallization, the colouring matter removed during these processes was mainly pH-insensitive, the pH-sensitive colouring matter tending to be incorporated in the crystal. While the fact that a large decrease in turbidity produced only a small increase in the I.V. would account, at least partly, for the slight change in I.V. during defecation, the effect is considered too small to account for variation in the I.V. between different raw sugars. Bone char column tests showed that the I.V. of the effluent increased with that of the on-liquor, while, in the case of low I.V. samples the I.V. of the effluent was lower than that of the on-liquor and increased toward that of the on-liquor as more liquor was displaced. In the case of on-liquors with high I.V., the effluent I.V. was higher after three displacements than that of the on-liquor. Decrease in the I.V. after one displacement indicated preferential absorption of pH-sensitive colouring matter. Ab-

sorbancy curves of raw sugar liquors in the range 220-500 $m\mu$ at pH 4, 7 and 9 before and after bone char treatment generally resembled those of caramel, melanoidin and alkaline degradation products of invert sugar. A more pH-sensitive colouring substance was also indicated, possibly derived from naturally-occurring polyphenols.

* * *

Crystal chemistry of calcium phosphates. W. E. BROWN. *Proc. Tech. Session Cane Sugar Refining Research*, 1964, 26-34.—The crystal structures and chemical properties of eight sparingly-soluble calcium phosphates connected with juice purification are discussed, special emphasis being placed on hydroxyapatite and octacalcium phosphate. The conditions under which the compounds are formed are also discussed. Hydroxyapatite and tetracalcium and octacalcium phosphates have similar structures and can form mixed crystals, hydroxyapatite being the stable form occurring either directly precipitated or as hydrolysed octacalcium phosphate. It is the prototype for the chief crystalline material in bone char and probably also for the final precipitate formed in defecation.

* * *

A method for analysis of the reducing sugars of molasses. L. F. MARTIN and J. P. MADACSI. *Proc. Tech. Session Cane Sugar Refining Research*, 1964, 34-45.—A method for quantitative determination of glucose, fructose and mannose in cane and refinery molasses is described. The reducing sugars were adsorbed on a carbon-"Celite" column and desorbed in fractions with aqueous ethanol washes of increasing concentration. The polarization of each 5-ml portion of the effluent was measured with an ETL-NPL polarimeter. Glucose was determined from the polarization before and after its oxidation by glucose oxidase, and the fructose and mannose then calculated from the remainder of the net polarization and the remainder of the total reducing sugars as determined with the modification by HEIDT *et al.*¹ of the Somogyi cuprimetric technique. Equations are given for calculating the weights of the fructose and mannose and a worked example is presented. While psicose present in the molasses and reducing sugar mixtures causes error in the results, paper electrophoresis has shown that it is present only in small amounts. Comparison of the reducing sugar concentrations in two raw factory and three refinery molasses samples are tabulated together with duplicate analyses of four of the samples. These indicate good reproducibility. One blackstrap and one refinery sample yielded a negative calculated fructose content. The discrepancy is attributed to the presence in the reducing sugar mixture of a non-reducing compound tentatively identified by chromatography as a levorotatory diheterolevulosan associated with deterioration of raw sugar in transit and during storage.

¹ *J. Amer. Chem. Soc.*, 1949, **71**, 2190.

Patents

UNITED KINGDOM

Extraction of sugar from sugar cane. SOC. DES SUCRERIES ET DE DISTILLERIE D'EGYPTE, of Cairo, Egypt. **1,024,428.** 28th September 1962; 30th March 1966.—A horizontal endless slat conveyor 7 moves over a fixed screen 9 between the driving pulleys 8 and 12, and between stationary side walls 10 higher than the level of bagasse, which is limited by adjustable rotating drum 13. Below the screen are juice collecting tanks 1a, 2b, 3c, 4d and 5e, and juice from these is transferred by pumps 1, 2, 3, 4 and 5 to distributing devices a, b, c, d and e over the conveyor, the juice from tank 5e being sent to device e by way of a heater 20. Sweet water is supplied by way of device g and fresh water through device f. Diffusion juice collecting in tank 6f is pumped to process by pump 6. Bagasse is supplied by the belt weigher 15 to a classifier which ensures correct dimensions of the bottom layer of bagasse to prevent blinding of the screen. It passes along the conveyor and has its sugar content removed before reaching the discharge roller 11. The central control panel receives information on the weight of bagasse feed from weigher 15 and on diffusion juice flow from flowmeter 16, and governs the rate of water addition which is measured by flowmeters 17 and 18.

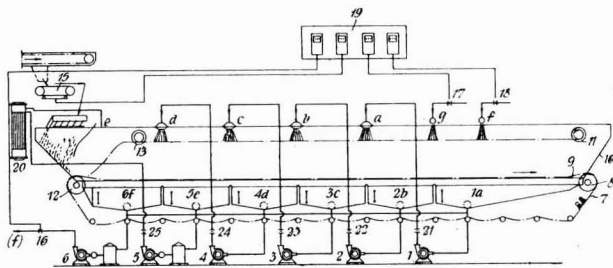
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Purifying sugar solutions. ASAHI KASEI KOGYO K.K., of Osaka, Japan. **1,027,583.** 14th December 1962; 27th April 1966.—See U.S.P. 3,196,045.

* * *

Purifying liquids (Bone char treatment of sugar solutions). TATE & LYLE REFINERIES LTD., of London E.C.3. **1,029,084.** 8th April 1964; 11th May 1966. The liquid (sugar solution) is passed upwardly through a bed of granular adsorbent (bone char) in which the upper surface of the bed is constrained by an at least partly foraminous member. Fresh adsorbent [5–50% (10–20%) by volume at a time] is introduced at the top of the bed and contaminated adsorbent removed from the bottom, liquid flow being interrupted for the period. The liquid is passed upwards at a velocity sufficient for buoyancy and viscous drag to overcome the weight of the granules. The bed of adsorbent is subjected to vibration (at or near its upper end) at least during introduction of fresh adsorbent. The reactor for this treatment has a circular cylindrical body portion and a conical top with the apex uppermost, the inlet for

fresh adsorbent passing through this apex and to the underside of the foraminous member (also of conical shape, with the apex uppermost). The bottom of the cylinder is joined to a conical base (apex downward) by way of a radiused section. Within the reactor and at the level of the radiused section is a hollow cone (apex uppermost) which covers the ends of the liquid feed pipe and a vent pipe. The treated liquid leaves through a pipe above the foraminous member and the spent adsorbent through a pipe connected to the bottom of the lower cone.



Producing D(-)-lactic acid. KYOWA HAKKO KOGYO Co. LTD., of Chiyoda-ku, Tokyo, Japan. **1,030,740.** 7th May 1963; 25th May 1966.—D(-)-lactic acid is produced by culturing *Sporolactobacillus inulinus* ATCC 14897 in a medium containing glucose, fructose, sucrose, etc., as a source of assimilable carbon, leucine or valine as a nitrogen source, mineral salt or salts, and biotin and pantothenic acid or a pantothenate as growth factors (and also *p*-amino-benzoic acid).

* * *

Producing L-glutamic acid and L-pyrrolidone carboxylic acid. KYOWA HAKKO KOGYO Co. LTD., of Chiyoda-ku, Tokyo, Japan. **1,031,046.** 5th March 1963; 25th May 1966. An L-glutamic acid-producing micro-organism, which requires biotin for its growth, is cultured in a medium containing assimilable carbon (e.g. as beet molasses), a source of assimilable nitrogen and mineral salts, and less biotin than required to support the growth of the micro-organism. After 5–24 hr (5–11 hr) are added biotin to more than the amount required for growth and also a substance which inhibits growth of the micro-organism (penicillin).

* * *

Beet harvester. SOC. D'ETUDES ET DE CONSTRUCTIONS MECANQUES DE CORBEIL, of Corbeil-Essonnes (S. & O.), France. **1,022,091.** 21st September 1964; 8th March 1966.

¹ I.S.J., 1966, 68,349.

Copies of Specifications of United Kingdom Patents can be obtained on application to The Patent Office, Sale Branch, Block C, Station Square House, St. Mary Cray, Orpington, Kent (price 4s 6d each). United States patent specifications are obtainable from: The Commissioner of Patents, Washington, D.C. 20231 U.S.A. (price 50 cents each).

TRADE NOTICES

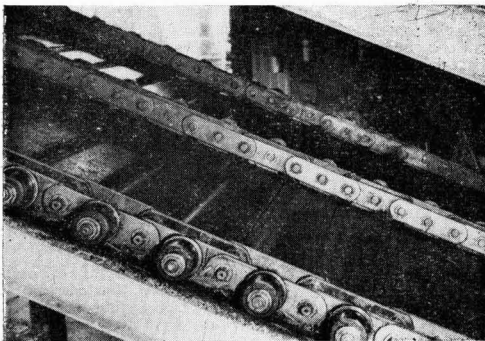
Statements published under this heading are based on information supplied by the firm or individual concerned. Literature can generally be obtained on request from the address given.

Renold chains in Philippine sugar mills. Renold Chains Ltd., Renold House, Wythenshawe, Manchester.

The illustrations show Renold chains being applied at two of the sixteen Philippine cane factories that use them for handling cane or bagasse. The Bogo factory of Bogo-Medellin Milling Co. Inc., at Cebu, is equipped with a 66-in wide cane carrier handling 3000-35000 t.c.d. which uses three strands of Renold outboard roller chain. This chain is one of the latest Renold products and has a breaking load of 100,000 lb. The bagasse carrier at the mill also employs Renold bagasse carrier chain.



Return strand of cane carrier at Central Binalbagan-Isabella.



Cane carrier at Bogo using three strands of outboard roller chain.

Power transmission at Central Binalbagan-Isabella, the third largest sugar factory in the world, is almost exclusively by means of Renold chain, while the cane carrier uses Renold chain of 100,000 lb breaking load and the bagasse elevator uses heavy-duty 60,000 lb Renold chain. Twenty-three centrals now use Renold transmission chain for mill and locomotive drives.

"Fabcon Cane Milling Aid". Fabcon Inc., 314 Public Square Bldg., Cleveland, Ohio, 44101 U.S.A.

"Fabcon Cane Milling Aid" is a strong liquid germicide now used in other food products. It specifically destroys bacteria and enzymes which cause inversion and loss of sucrose in juice. With the use of 5 p.p.m. "CMA", reduction in sugar losses can be seen from the reduction in purity drop between the first expressed juice and mixed juice. There is an even greater reduction in the increase of reducing sugars in mixed juice compared with first expressed juice. Slime on mill and juice runways is eliminated, and odour in the mill area becomes clean and refreshing.

An independent examination by Rumsu Technical Services Ltd., of Kingston, Jamaica, reports that the "CMA" test made at Innswood shows a significant previous sugar loss at the mills which was reduced by the use of "CMA" as sanitizing chemical. The average purity drop from 1st expressed to mixed juice was 1.26 units with 5 p.p.m. "CMA", compared with 1.50 units without treatment. This is equivalent to a 96° sugar saving of 0.572 tons per 1000 tons of cane at 5 p.p.m. "CMA".

* * *

Palletizing machine. Carl Drohmann G.m.b.H., 7 Stuttgart-Bad Canstatt, Postfach 360, Germany.

The P3 palletizer is a high-speed fully-automatic machine which has no pneumatic or hydraulic drives but is operated on a numerically-controlled programme on a minimum of floor space.

* * *

Drive head for sedimentation plant. Dorr-Oliver Inc., Stamford, Conn., U.S.A.

A new drive head design, now incorporated in all Dorr-Oliver thickeners and clarifiers and related equipment, has anti-friction bearings running in self-contained independent oil baths. These bearings replace conventional large annular ball thrust bearings. The bearing assembly is so arranged that tilting of the rake assembly is prevented and the need for oil seals obviated.

Variable sets of cylindrical torsion guides transmit a balanced driving torque and stabilize the rake mechanism at all sludge levels, permitting the rakes to be freely raised or lowered under varying load conditions.

The drive head is pre-assembled and adjusted for connexion to the rake mechanism, whether a lifting device is required or not. It is made in a range of sizes covering the torque range of 1000 to 4 million ft.lb. All units have a minimum endurance rating of 1,700,000 cycles. Interchangeability of components

permits the drive head to be adapted to a variety of thickener sizes from laboratory models up to 400 ft diameter units.

* * *

Sliding-vane pump for viscous fluids. Jobson & Beckwith Ltd., 62 Southwark Bridge Rd., London S.E.1.

The "Rotavane" pump comprises two reciprocating vanes rotating slowly within specially-shaped cast iron liners in an alloy casing. The heavy-duty shaft runs in pre-packed roller bearings, isolated from the pumping chamber by mechanical seals, and an air gap. An adjustable relief valve integral with the casing is hydraulically balanced, is completely positive in action and prevents seeping of the fluid below the pre-set operating pressure. The maximum pressure is 110 p.s.i. In tests with 80°Bx molasses, the pump transferred 14 tons in 50 min at a speed of only 171 r.p.m. and against a total manometric head of 80 ft. The pump can be directly coupled to a geared motor or driven by a synchronous speed motor through pulleys. It is available made of cast iron, aluminium and cast iron, or all bronze.

* * *

PUBLICATIONS RECEIVED

VARIABLE SPEED CONTROL. Allspeeds Ltd., Royal Works, Clayton-le-Moors, P.O. Box 43, Accrington, Lancs.

The "Kopp" variable speed drive, Series B, described in a brochure, is a stepless speed regulator in which the input and output drive shafts are mounted co-axially in ball races. Power is transmitted from an input drive cone to the identical output drive cone through a number of drive balls mounted on spindles. When the ball spindles are parallel to the drive shafts, i.e. the diameters of the balls at the point of contact with the drive cones are equal, the ratio is 1:1. Speed changes are effected by tilting the ball spindles so as to alter the relative diameters of the ball at the point of contact with the input and output drive cones. One direction of tilt reduces the output speeds and increases the output speeds, while the other direction has the opposite effect. Tables are given showing the standard output speeds available and the input h.p. at varying input speeds.

* * *

ELECTRONIC SPEED CONTROL. Chadburns (Research & Development) Ltd., Elswick House, Mellor, Blackburn, Lancs.

The Mk. 1 controller described in a recent bulletin is constructed on a modular system and consists basically of a rack-mounting panel with attached chassis, normally housed in a cabinet and fitted with plug-and-socket connectors at the rear. It may be used to provide one of three basic systems: ratio, speed or synchronization control.

* * *

EASY MOISTURE MEASUREMENT. Shaw Moisture Meters, Rawson Rd., Westgate, Bradford 1, Yorks.

A new publication details the company's hygrometers and dewpoint meters for use in measuring, detecting and recording water in air, gas or such liquids as SO_2 , oil and petrol.

* * *

HELIX STORAGE TANKS. Thermo Plastics Ltd., Dunstable, Beds.

The first number of a new publication, to be issued three times a year by the company, gives details of a new spiral winding process for manufacturing large storage tanks. The

process uses high-density polyethylene for making tanks of up to 3300 gal capacity, although tanks of 10,000 gal capacity have been supplied. The tanks are cheaper and lighter than stainless steel tanks and will hold many substances for which mild steel and other materials are unsuitable, including sugar solutions. The tanks are operable at temperatures up to 70°C, or higher for short periods.

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POLISH SUGAR MACHINERY. CEKOP, Warszawa, Mokotowska 49, P.O.B. 112, Poland.

Details are announced of two types of milk-of-lime grit separation equipment (a vibratory and a decanting type), a grasshopper conveyor for white sugar transporting, a vacuum pan of 10 cu.m. effective capacity, and a pre-liming tank (type CPB-1C).

* * *

LDEP SERVICE TO INDUSTRY. Lancashire Dynamo Electronic Products Ltd., Rugeley, Staffs.

This is the title of a loose-leaf catalogue giving details of the company's control and instrumentation products. These are grouped under four main categories: instrumentation (recorders, controllers and servo-mechanisms), photo-electrics, level controls, and timers and supervisory equipment.

* * *

AIRMEC INDUSTRIAL ELECTRONICS. Airmec Ltd., High Wycombe, Bucks.

A loose-leaf catalogue contains leaflets describing a large number of electronic devices. These include counting, nucleonic and photo-electric equipment, switches, frequency meter, temperature controllers, and time control and measurement devices.

* * *

BMA INFORMATION. Braunschweigische Maschinenbaustalt A.G., Braunschweig, Germany.

This is No. 5 in the series of well-prepared publications giving information on BMA equipment and design theory. The first article in this issue gives a description of the equipment and processes in the 1500-2000 t.c.d. cane factory supplied and erected by BMA for Habib Sugar Mills Ltd. at Nawabshah, Pakistan. The second article deals with solid-liquid separation in clarifiers, while the third gives details of a new high-speed evaporator, which is compared with other types. Briefer mention is made of BMA tower diffusers, the largest of which is to be put into operation during the 1966 campaign at Elsdorf factory, Germany, and will have a diameter of 6.8 m (22 ft 4 in) and a daily throughput of 5510 short tons; the BMA beet washer, now provided with an automatic stone ejector; the Braunschweig juice purification system; and a forced-circulation vacuum pan, which in tests at the Rain-am-Lech factory of Süddeutsche Zucker A.G. has given improved sugar quality with increased heat transmission and more uniform supersaturation.

* * *

NORTHROP 5004 DIESEL TRACTOR. Northrop Tractors Ltd., Church St., Ware, Herts.

Leaflet No. 485/O.E.C.D. of the National Institute of Agricultural Engineering is a detailed report on tests carried out on the Northrop Model 5004 turbo-charged, four-wheel drive diesel tractor. This is based on the Ford 5000 tractor and has established a reputation for remarkable traction and pulling power.

* * *

INHIBITORS FOR ACIDS. Glovers (Chemicals) Ltd., Wortley Low Mills, Whitehall Rd., Leeds 12.

A new brochure gives details of two new inhibitors for acids for use in chemical cleaning and scale removal. "Duoteric S" is design to reduce the attack of sulphuric, sulphamic, phosphoric and citric acids on steel and does so by up to 99.9% at 200-210°F. "Duoteric H" is intended for use with hydrochloric acid, metal corrosion being reduced by up to 99% depending on temperature, acid strength and inhibitor concentration.

BREVITIES

Polish sugar crop, 1965/66¹.—Official Polish statistics indicate that sugar production from the past five campaigns in Poland was as follows:

1961/62	1,508,000 metric tons, white value
1962/63	1,222,400 " " " "
1963/64	1,309,800 " " " "
1964/65	1,653,900 " " " "
1965/66	1,354,400 " " " "

The area sown to beet during 1965 has officially been put at 476,000 hectares as compared with 443,600 hectares sown during 1964, while the tonnage of beet lifted during 1965 and 1964 amounted to totals of 12,314,000 and 12,574,000 metric tons, respectively.

* * *

Sugar factory planned for Dahomey².—The erection of a sugar factory is planned by the Governments of Dahomey and Togo. The project depends, however, on the production of suitable sugar cane. The French aid funds (Fonds d'Aide et de Coopération—F.A.C.) will establish test plantations in the district of the river Mono, which is the border between the two countries. The present imports of sugar into Dahomey amount to some 10,000 tons per year, while 5000 tons are annually imported by Togo.

* * *

Mozambique sugar proposal³.—It is reported that a sugar organization will commence operations in the Vila Machado area, approximately 40 miles from Beira. The sugar mill is expected to have an annual output target of 150,000 tons.

Stock Exchange Quotations

CLOSING MIDDLE

London Stocks (at 17th November, 1966)	s d
Anglo-Ceylon (5s)	4/4½
Antigua Sugar Factory (£1)	6/3
Booker Bros. (10s)	17/3 (x.d.)
British Sugar Corp. Ltd. (£1)	21/3
Caroni Ord. (2s)	1/6
Caroni 6% Cum. Pref. (£1)	14/-
Demerara Co. (Holdings) Ltd.	2/6
Distillers Co. Ltd. (10s units)	17/9
Gledhow Chaka's Kraal (R1)	13/9
Hulett & Sons (R1)	11/3
Jamaica Sugar Estates Ltd. (5s units)	3/9
Leach's Argentine (10s units)	11/6
Manbré & Garton Ltd. (10s)	28/1½
Reynolds Bros. (R1)	15/-
St. Kitts (London) Ltd. (£1)	15/-
Sena Sugar Estates Ltd. (5s)	8/6
Tate & Lyle Ltd. (£1)	24/4½
Trinidad Sugar (5s stock units)	1/9
West Indies Sugar Co. Ltd. (£1)	8/1½

CLOSING MIDDLE

New York Stocks (at 16th November, 1966)	\$
American Crystal (\$5)	38½
Amer. Sugar Ref. Co. (\$12.50)	24
Central Aguirre (\$5)	27½
Great Western Sugar Co.	38½
North American Sugar (\$10)	7¼
South P.R. Sugar Co.	22½
United Fruit Co.	29

Cuban sugar production, 1966⁴.—The Cuban Ministry of the Sugar Industry has announced that a total of 36,839,806 metric tons of cane was crushed during the 1966 season, as against 50,686,485 tons crushed in 1965. Sugar production totalled 4,455,255 tons, about a quarter less than the 1965 outturn of 6,050,532 tons. These figures reflect the effects of the 1965 drought, the worst to hit Cuba for more than half a century. The yield of sugar on cane, however, was 12.09%, an increase over the 11.94% achieved in 1965.

* * *

Malawi sugar factory⁵.—The opening of the factory of the Sugar Corporation of Malawi (SUCOMA) at Chikwawa took place in August when the first cane from the £3,000,000 sugar scheme in the Shire valley went through the mill. At present there are more than 3000 acres of land under cane which by October will have risen to 4300 acres. From April 1967 SUCOMA will be capable of supplying the whole of Malawi's sugar needs and by then it will be doing its own marketing and distribution. The estate is expected to yield 25,000 tons of sugar next year, enough to leave about 5000 tons over for export.

* * *

The late F. I. Meyers.—FRED I. MEYERS, Editor-in-Chief of the Gilmore Sugar Manuals, died on the 31st October. He had been Editor of the Gilmore Manuals for 30 years, following many years as an Instructor in Advertising at Tulane University, and had maintained for the Manuals their reputation for reliable and detailed information which dated from the inception of the first Louisiana-Florida manual in 1909. He is to be succeeded as Executive Editor by C. O. DUPUY, Managing Editor for the past five years, and Director of Publications for the Hauser Printing Company Inc. It is planned to continue to publish the existing series of Manuals for Louisiana-Florida, Puerto Rico-Dominican Republic and Hawaii, and the new West Indies Manual, and to expand the series into every area where such a manual will serve a useful purpose to the sugar industry of that area.

* * *

Argentine sugar factory closures.—Sugar production in the province of Tucumán has been cut by a further 5% from the level of 75% of the 1965 outturn, according to a decree published in August⁶. This will bring a total reduction of 30% or 360,000 tons, which will help to reduce the sugar stocks which last year left an unsold carryover of 500,000 tons. The Government has taken over seven of the province's mills⁷ and has closed them, bringing the total closures to eight⁸. Several other private firms are seeking to modernize their mills, and to diversify their activities by producing cellulose from bagasse⁹.

* * *

Mauritius bulk loading proposal.—It is reported¹⁰ that the commission headed by Sir FRANK MILBOURNE recommends that bulk loading installations should be built at Port Louis. The main points in favour of bulk loading are undoubtedly that it would cut down on expenses borne by the sugar estates and it has been estimated that £1 2s 6d per ton would be saved if the proposal were adopted. In addition the loading time for ships in Port Louis would be reduced considerably. On the other hand, the main criticisms of the scheme are the capital cost (in this respect a sum of £2,250,000 has been mentioned) and the fact that 3000 people would have to be found alternative employment or phased out of employment by early retirement.

¹ C. Czarnikow Ltd., *Sugar Review*, 1966, (778), 162.

² F. O. Licht, *International Sugar Rpt.*, 1966, 98, (24), 18.

³ *Overseas Review* (Barclays D.C.O.), September 1966, p. 26.

⁴ *Cuba Economic News*, 1966, 2, (13), 7.

⁵ *Overseas Review* (Barclays D.C.O.), September 1966, p. 39.

⁶ *Public Ledger*, 9th August, 1966.

⁷ *ibid.*, 22nd September 1966.

⁸ *S.S.J.*, 1966, 68, 130.

⁹ *Fortnightly Review* (Bank of London & S. America Ltd.), 1966, 31, 561.

¹⁰ *Overseas Review* (Barclays Bank D.C.O.), September 1966,

INDEX TO VOLUME LXVIII

SOME REMARKS ON ITS USE

In using this Index it should be noted that the principal entries cover the several stages of production : CULTIVATION (see Beet; Cane; Diseases; Fertilizer; Irrigation; Mechanization; Pests; Soil; Transport; Varieties; Weeds, etc.); SUGAR PROCESSING (see Bagasse; Boilers; Boiling; Carbonation; Centrifugals; Clarification; Crystallization; Diffusion; Evaporators; Filter; Masseccuite; Mills; Milling; Molasses; Pans, Vacuum; Scale; Sucrose; Sugar; Sugars; Sulphitation; Water, etc.); REFINING (see Bone Char; Carbon; Refining; etc.); and By-Products (see Alcohol; Animal Fodder; By-Products; Fermentation; Paper; Pulp; Yeast, etc.).

Subjects covered separately include Ash; Bulk handling and Bulk storage; Colour; Control, Automatic and Chemical; Countries; Gur; Ion exchange; Juice; Micro-organisms; pH; Polarization; Weighing, etc. Glucose and Fructose are to be found under Dextrose and Levulose. Obituaries, Statistics and Trade Notices are collected together under those headings. "Sucrose" implies the pure chemical; "Sugar" the commercial product; and "Sugars" the chemical family, rather than grades of sugar. When looking under the author's name, it should be remembered that the surname may be the penultimate in Spanish.

(Abs.) indicates *Abstract*; (Brev.), *Brevity*; (B.B.), *Books and Bulletins*; (Corr.), *Correspondence*; (N.C.), *Note and Comment*; (Pat.), *Patent*; (Stat.), *Statistics*; (T.N.), *Trade Notice*.

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