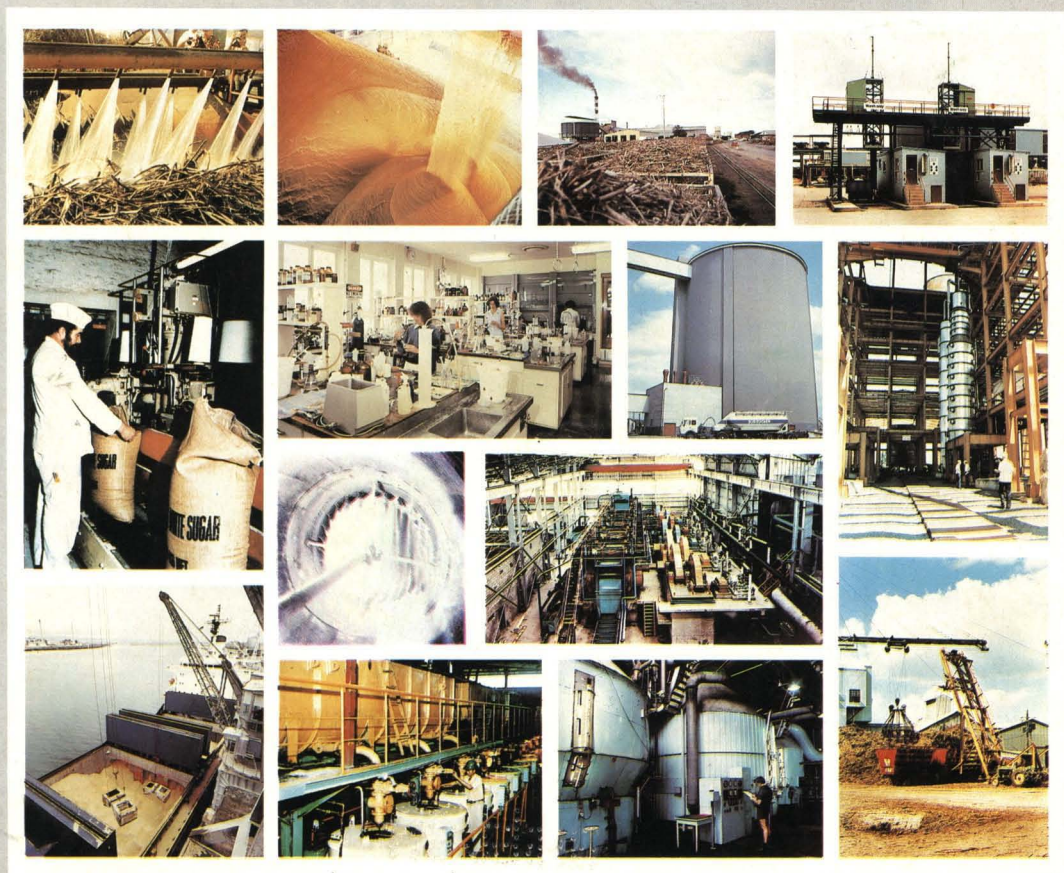
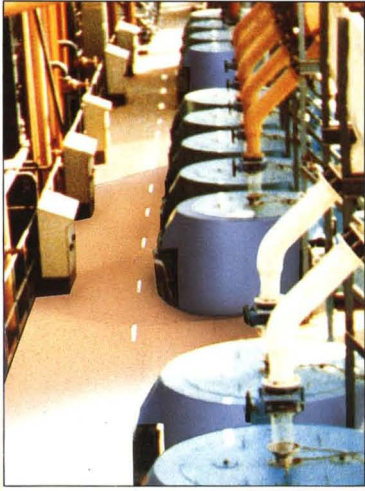


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**CONTENTS**

*Page*

217 News and views

\* \* \*

*Technical articles*

219 **ACONITIC ACID REMOVAL DURING CANE JUICE CLARIFICATION**  
By H. Hanine, J. Mourgues and J. Molinier (*France*)

221 **NEW DEVELOPMENTS IN THE PURIFICATION OF BEET SUGAR**  
By R. F. Madsen (*Denmark*)

224 **DETERMINATION OF SUCROSE USING A GLUCOSE ANALYSER**  
By T. A. Hsu and M. H. Bagley (*USA*)

\* \* \*

231 ICUMSA News

238 Facts and figures

\* \* \*

*Abstracts section*

119A Cane sugar manufacture

122A Beet sugar manufacture

124A Sugar refining

126A Laboratory studies

127A By-products

\* \* \*

Cover III *Index to Advertisers*

*Contenido*

*Contenu*

*Inhalt*

217 Noticias y opiniones / Nouvelles et opinions / Nachrichten und Ansichten

\* \* \*

*Articulos Técnicos / Articles Techniques / Technisch Artikeln*

219 Remoción de ácido aconítico durante la clarificación del jugo de caña / L'enlèvement de l'acide aconitique au cours de l'épuration du jus de canne / Beseitigung von Akonitsäure während der Rohrsaftklärung

221 Nuevos desarrollos en la purificación del azúcar de remolacha / Nouvelles développements dans la purification du sucre de betterave / Neue Entwicklungen in der Reinigung von Rübenzucker

234 Determinación de sacarosa usando un analizador de glucosa / Détermination du saccharose à l'aide d'un analyseur de glucose / Saccharosebestimmung mittels eines Glucose-Analysators

\* \* \*

231 Noticias de la ICUMSA / Nouvelles de l'ICUMSA / ICUMSA-Nachrichten

238 Hechos y números / Faits et nombres / Tatsache und Ziffern

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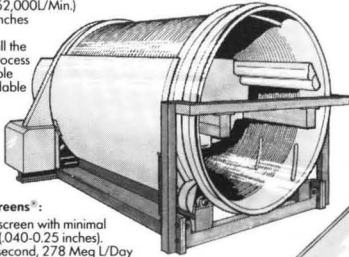
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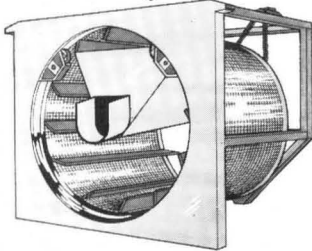
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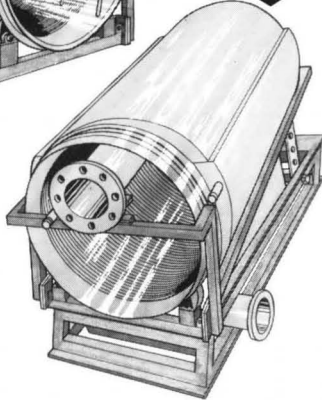
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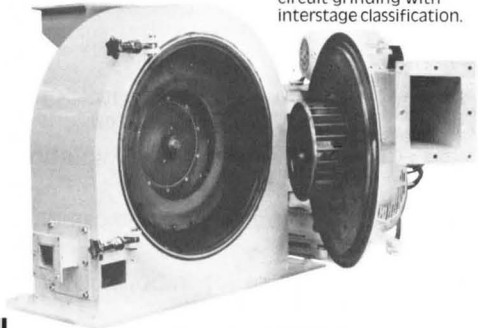
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# News and views

## European beet sugar production estimates, 1990/91<sup>1</sup>

The 1990/91 sugar beet crop started on a cautious tone as farmers showed only a modest response to the higher level of world prices. In the EEC the area sown to beet expanded by 2.4% while the area sown in Western Europe as a whole rose by 3.5%. By contrast, in Eastern Europe the area sown fell by almost 3% mainly as a result of the smaller area sown in Rumania and to some extent in the USSR. However, this latter will have little or no impact on production; last year's beet crop was far higher than the industry could absorb so that a smaller beet quantity harvested from a smaller area will not necessarily lead to lower production.

Last year's yields were very good in Western Europe and a return to normal could easily offset the extra hectares sown. Hopes for higher production were therefore not so much based upon the increase in area but rather on the very early sowings this year and good conditions during most of the vegetation period. Mild winter weather, followed by a warm spring, encouraged early drilling and the weather continued good until mid-July. The drought which came then and lasted several weeks has caused damage reported from France, Hungary, Czechoslovakia and West Germany. Nevertheless, production in Western Europe is expected to rise by a million tonnes from the 1989/90 figure, almost all accounted for by France, West Germany and Turkey. The Turkish Sugar Corporation is endeavouring to increase production both for domestic consumption and exports. To encourage farmers to plant more beets and increase their yields, support prices have been increased markedly and the area has risen by 7%. More beets have been sown in Yugoslavia too, and output is forecast to rise by more than 10%.

The situation in Eastern Europe is dominated by events in the USSR. Last year's enormous harvest of 97.5 million tonnes, of which 91.9 million tonnes were purchased for sugar manufacture,

proved too much for the industry to process. The majority of the more than 300 factories in the USSR contain machinery which dates from the last century, which again and again leads to stoppages during the campaign and resultant losses. The experience with last year's bumper crop apparently prompted the authorities to reduce this year's beet production target to 88 million tonnes and the area was reduced accordingly. So far, the weather has not been unfavourable and a sugar crop of the same size as last year is expected, or even above if all goes well. Such hopes could be ruined by adverse harvesting conditions which are always a possibility in the Soviet Union. Output in East Germany is expected to rise in spite of a smaller area, as yields last year were fairly poor. Elsewhere sugar production is expected to be about the same or lower.

The estimates appear below:

	1990/91	1989/90
	<i>tonnes, raw value</i>	
Belgium	1,070,000	1,038,000
Denmark	543,000	530,000
France	4,600,000	4,198,000
Germany, West	3,650,000	3,337,000
Greece	325,000	421,000
Holland	1,240,000	1,240,000
Ireland	239,000	233,000
Italy	1,707,000	1,880,000
Portugal	2,000	2,000
Spain	1,027,000	1,037,000
UK	1,415,000	1,377,000
<i>EEC</i>	<i>15,818,000</i>	<i>15,293,000</i>
Austria	478,000	458,000
Finland	141,000	160,000
Sweden	430,000	421,000
Switzerland	148,000	152,000
Turkey	1,740,000	1,378,000
Yugoslavia	1,025,000	917,000
<i>West Europe</i>	<i>19,780,000</i>	<i>18,779,000</i>
Albania	25,000	45,000
Bulgaria	60,000	67,000
Czechoslovakia	695,000	766,000
Germany, East	765,000	678,000
Hungary	595,000	577,000
Poland	1,850,000	1,865,000
Rumania	425,000	543,000
USSR	9,350,000	9,565,000
<i>East Europe</i>	<i>13,765,000</i>	<i>14,106,000</i>
Total Europe	33,545,000	32,885,000

## Brazil sugar situation

The Brazilian sugar industry, as well as the rest of the economy, is adjusting to the economic measures introduced by the new administration<sup>2</sup>. In aiming to control inflation, many strong austerity measures were implemented. Consumption for 1990/91 is estimated to grow at only 1.8% which is lower than both population growth and the average of the past five years. The impact has not been so severe on the sugar industry because the foreign market is currently more profitable than the domestic market for sugar or alcohol.

So far there has been no definition of the way the sugar market will work following the closure of the Sugar and Alcohol Institute (IAA) which for many years controlled the production quotas and cane prices and administered the marketing of sugar and alcohol. For the next crop year the Ministry of Economy will settle quotas for the domestic alcohol and sugar markets. Exporters, although free to deal directly with the buyer, will only be allowed to export after the fulfilment of their domestic quotas. The prices they will receive, however, are those of the international transaction. An export quota of 550,000 tonnes was announced in early September, all of it to be produced in the North-Northeast region. Exports from the Centre-South were explicitly banned as much as the cane in this area will be used to produce fuel alcohol as part of the quotas announced earlier<sup>3</sup>. Brazil's US quota of 240,578 tonnes for the year to end-September 1991 will leave only 338,000 tonnes of Brazilian sugar available to the world market.

## Another Ord River sugar industry proposal

Readers with long memories will recall that, whenever there is a relative shortage of sugar in the world and prices on the world market climb, proposals are made for establishment of a cane sugar industry in the Ord River irrigation area

1 F. O. Licht, *Int. Sugar Rpt.*, 1990, 122, 427 - 431.

2 *ISO MECAS Review*, September 11, 1990.

3 *I.S.J.*, 1990, 92, 174.

of Western Australia. The Government of Western Australia has recently issued a media statement announcing that it is looking for interested parties to establish an industry in the area in the north of the state.

The decision to test the market is based on the need to take advantage of a unique set of circumstances which include the availability of cane planting material at the Kununurra research station, strong market opportunities, a positive market outlook and the deregulation of the Australian sugar industry. Extensive research and development have shown that sugar cane is well adapted to the region and that high yields are obtainable. Almost a hectare of commercially acceptable cane varieties is currently planted at the government's research station and the state government is interested in submissions from parties interested in multiplying the material from 1991 with a view to establishing a viable sugar industry in the area. Contact should be made with the nearest Western Australia Government office or with Western Australia House, 115 Strand, London WC2R 0AJ, England.

### Indian sugar factory licensing policy

The Indian government announced fresh guidelines in August for the licensing of new plants and expansion of existing sugar factories during the 8th Five-Year Plan period. Factories may continue to be licensed for a minimum economic crushing capacity of 2500 t.c.d. but there will be no maximum limit on capacity. No relaxation of minimum capacity will be permitted in areas where there is a lack of sugar cane availability. Licences will be issued on condition that cane will be paid for on a basis of the sugar content.

The previous ban on building of factories within 40 km of an existing facility has been changed, reducing the distance to 15 km. This is contrary to the views of the industry which argued for increase in the separation. It is reported

by C. Czarnikow Ltd.<sup>4</sup> that this will put undue pressure on the finances of many existing factories. New factories are released from the obligation to supply low-priced levy sugar for the first seven years of operation and this gives them considerable leeway to compete with existing enterprises for cane supplies. Indeed, without the levy commitment, new factories are in a position to take advantage of any nearby cane infrastructure that may have been built up by an existing factory. The industry argues that, in reducing the minimum separation, the government may well bring about an increase in new capacity but the real cost will be that many existing factories will become severely under-utilized and run into financial difficulties. In time this will also discourage factories from making any investment in their local cane growing areas since any irrigation or transport infrastructure risks being taken over by a new factory.

### EEC sugar production quota for East Germany

The European Community's Executive Commission has proposed a sugar quota of 870,000 tonnes, white value, for East Germany in its plan to integrate the country into the EEC Common Agricultural Policy<sup>5</sup>. This applies to A- and B-sugar and represents a cut of about 15% on the average of the past five years. The Commission met in a special session to work out details of absorbing East Germany into the Community.

The total is made up of an A-quota of 665,280 tonnes and B-quota of 204,710 tonnes. The latter has been established on a similar ratio to the A-quota as applies for West Germany, i.e. 30%, in order to facilitate integration of the two sets of entitlements into a single quota for a unified Germany. It has been criticised<sup>6</sup>, however, as providing an unwarranted opportunity for increasing surplus sugar production by the Community.

A number of EEC countries and much of the Community's sugar processing industry allege that the Germans

are being offered an unrealistically high sugar production ceiling which will not only swell existing EC surpluses but land others inside Europe with a hefty bill for its disposal on world markets. Political considerations apart, it is difficult to find any justification for the East German figure as it stands. Under the European Commission's proposal, the quota will yield levies of roughly Ecu50m (£34.8 m) to pay for any exporting costs. Assuming an export subsidy of around Ecu400 a tonne, the EC will be able to afford 125,000 tonnes of surplus East German sugar within the current restraints of the regime.

Much depends on East German consumption, for which reliable figures do not exist. According to the International Sugar Organization, annual consumption in East Germany between 1984 and 1988 was 680,000 tonnes of white sugar equivalent, while the West German industry's own estimates suggest a figure of 665,000 tonnes on average over the past five years. Even taking the highest historic projection, there appears certain to be a much bigger difference between production and domestic consumption than the 125,000 tonnes "permitted" under the rules of the regime.

What is alarming and angering the industry and other member states is the knowledge that consumption in East Germany is falling sharply and is bound to plunge further as hitherto subsidized sugar prices move sharply up.

### Australian sugar exports reduction<sup>7</sup>

An extended dry period at the start of the growing season, followed by prolonged wet weather, has reduced the likely cane sugar crop in the 1990/91 season from the 3,520,000 tonnes of 1980/90. As a consequence, exports are likely to fall by some 300-350,000 tonnes from the 2,830,000 tonnes exported in the season July 1989/June 1990.

<sup>4</sup> Czarnikow Sugar Review, 1990, (1800), 123.  
<sup>5</sup> Reuters News, August 21, 1990.  
<sup>6</sup> Financial Times, September 24, 1990.  
<sup>7</sup> F. O. Licht, Int. Sugar Rpt., 1990, 122, 453.



# Aconitic acid removal during cane juice clarification

By H. Hanine\*, J. Mourgues\* and J. Molinier†

## Introduction

During the process of cane juice clarification, many compounds are eliminated, including polyphenols<sup>1</sup>, amino-acids<sup>2</sup> and wax<sup>3</sup>. No study regarding the elimination of aconitic acid, which is the main organic acid in sugar cane, has been done, however. The authors have taken an interest in the effects of this acid on the clarification process, its role in the buffer capacity of juice and its inhibitory effect on the precipitation of calcium phosphate<sup>4</sup>. Many studies have been made concerning the aconitic acid content of molasses<sup>5-8</sup> and its recovery for the manufacture of plastics, detergents and wetting agents<sup>9-11</sup>.

During white sugar manufacture, when purification by means of ion exchange resin is employed, it is also necessary to carry out a preliminary clarification process with defecation<sup>12</sup> or phosphatation with phosphoric acid and lime<sup>13</sup>. The fractions of the anion exchange regenerant liquor constitute an important source of aconitic acid<sup>9,12</sup>, but no study on its recovery has been reported. Before undertaking such a study, we wanted to examine the behaviour of aconitic acid during the preliminary clarification in order to choose the process allowing the removal of the smallest quantity of this acid. These investigations deal with raw material used (cane juice) clarified in accordance with various processes. A parallel between the aconitic acid removal and the clarification (turbidity, filtrability) has been drawn.

## Experimental procedure

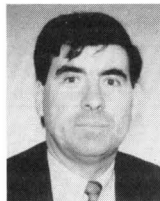
The samples of juice were obtained from French West Indies cane crushed in an experimental mill. The juices were subjected to clarification on a laboratory scale by defecation, double carbonatation, sulphitation and phosphatation, the last by use of a saturated solution of calcium superphosphate and also by means of phosphoric acid and lime at 98°C. All tests of clarification were made three times.

The aconitic acid content was



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determined with HPLC. The centrifuged samples were purified through a Sep-Pak C<sub>18</sub> cartridge and then filtered through a 0.22 µm Millipore filter. The quantitative analysis was done with a column HPX-87H at 45°C, eluted at a 0.6 ml/min flow rate with 0.6mM sulphuric acid and the aconitic acid measured at 214 nm with external standardization with two isomer forms: cis and trans. The results were expressed in mg of aconitic acid on 100°Bx.

Phosphorus was measured in the saturated solution of calcium superphosphate by the ammonium molybdate method<sup>14</sup>. Turbidity was measured with a standard turbidimeter in NTU units. The filtrability represents the volume which passed through a filter (diameter 47 mm; porosity: 0.45 µm; pressure: 0.45 bar) during 3 minutes<sup>15</sup>. The optical density of the clarified juice was meas-

ured at 420 nm with a cell of 1 cm path length without preliminary dilution of the samples.

## Results and discussion

According to results recorded in Table I, clarification by double carbonatation removes the largest quantity of aconitic acid (78.8%), while defecation removes 73.8%, phosphatation by addition of calcium superphosphate removes 10.2%, phosphatation by use of phosphoric acid and milk-of-lime removes 18.7%, and sulphitation removes 16.3%.

These results confirm observations concerning the aconitic acid content in molasses coming from juice of various origins and which have been clarified by

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Table I. Effects of different process of clarification on aconitic acid removal

Nature of juice	Aconitic acid content, mg/l on 100°Bx	Removal, %	OD at 420 nm	Filtrability V <sub>max</sub> , ml
Mixed juice at 15°Bx, pH 5.2	5392	-	0.422	70
Clarified juice				
(a) Defecation at pH 6.8	1411	73.8	0.335	20
(b) Double carbonatation	1141	78.8	1.950	13
(c) Sulphitation	4511	16.3	0.259	9
(d) Phosphatation at pH 5.5				
with super-phosphate	4311	20.2	0.180	2.6
with phosphoric acid	4385	18.7	0.199	2.4

different processes. The molasses from juice clarified by carbonation is less rich in aconitic acid than from juice clarified by defecation<sup>6</sup>. That clarified by defecation is less rich than that clarified by sulphitation<sup>7</sup>. However, no comparative data regarding the effects of phosphatation are known.

Phosphatation by addition of phosphoric acid and lime milk gives a better clarification than sulphitation for the same final reaction (pH 5.5); the removal of aconitic acid increases until the dose of phosphoric acid reaches 350 mg/l. Above this value, the removal of aconitic acid slightly decreases (Table II). Similarly, as seen in Table III, for the same quantity of phosphoric acid added, the removal was greater as the pH got closer to neutrality. The clarification is improved but the colour, measured at 420 nm, increases.

During the clarification process, a coprecipitation probably occurs of other insoluble calcium compounds with calcium aconitate hexahydrate (or trihydrate if heating takes place) associated with a calcium magnesium salt  $\text{Ca}_2\text{Mg}(\text{C}_6\text{H}_6\text{O}_6)_2 \cdot 6\text{H}_2\text{O}$  less soluble than other salts of calcium<sup>16</sup>.

At pH 6.5 in the case of defecation (Table I), as well as in the case of phosphatation (Table III), the level of aconitic acid content removed is high. At this pH the best yield is obtained<sup>9-11</sup> on recovery of calcium aconitate and of calcium magnesium aconitate from molasses rich in aconitic acid.

In the case of conventional sugar manufacture, it is in our interest to remove as much aconitic acid as possible because this compound inhibits the crystallization of sugar. It may play a role either in the viscosity of molasses<sup>17</sup> or by the production of a more or less polymerized organic complex with sugars<sup>4</sup> or by the specific inhibitory properties of aconitic acid salts, properties which are greater than those of sulphates, chlorides and phosphates<sup>18</sup>.

In the case of sugar manufacture using ion exchange resin, phosphatation by addition of phosphoric acid and lime milk to give a final pH between 5 and

**Table II. Effects of various quantities of phosphoric acid on aconitic acid removal during the process of clarification by phosphatation**

	Aconitic acid content, mg/l on 100°Bx	Removal, %	OD at 420 nm	Turbidity, NTU	Filtrability $V_{\text{max}}$ , ml
Mixed juice at 15°Bx	6452	-	0.950	121	8.82
Phosphoric acid used					
50 mg/l	5025	19.3	0.281	2.2	100
150 mg/l	4754	36.1	0.271	1.2	214
250 mg/l	4163	35.5	0.273	1.1	340
350 mg/l	3998	38.0	0.267	1.1	375
450 mg/l	4530	29.0	0.266	0.8	553
650 mg/l	4487	30.4	0.254	0.7	666

**Table III. Effects of the final juice pH on aconitic acid removal during the phosphatation process for an addition of phosphoric acid at 650 mg/l**

Final juice pH	Aconitic acid content, mg/l on 100°Bx	Removal, %	OD at 420 nm	Turbidity, NTU	Filtrability $V_{\text{max}}$ , ml
4.5	5423	16.0	0.272	3.7	90
5.5	4765	26.2	0.221	3.2	100
6.5	3833	40.6	0.335	0.9	200
7.0	4113	36.2	0.387	0.7	207

5.5 appeared to be one the best techniques of clarification<sup>13</sup>. It permits avoidance of an excess of calcium in the cane juice and preserves a large proportion of the aconitic acid which can then be recovered from regeneration eluants. However, defecation at a pH between 7 and 11 before passing through exchange resin<sup>12</sup> is not advisable.

#### Conclusion

The removal of aconitic acid during cane juice clarification varies with the process used. More is removed by the carbonation and defecation processes, and less by the sulphitation process. The phosphatation process is recommended to clarify cane juice for the production of sugar using ion exchange when recovery of aconitic acid is intended.

Phosphatation with phosphoric acid and with calcium superphosphate give the same results, but we prefer to use phosphoric acid since it allows easy pH regulation at 5.5. The final reaction pH must be maintained between 5 and 5.5 because more aconitic acid is removed closer to neutrality. Many preliminary tests must be done for each juice type in order to determine the minimum quantity of phosphoric acid to

be added.

Another possibility of cane juice clarification is that of ultrafiltration<sup>19</sup>. This might permit removal of materials in suspension and colloidal substances (proteins, wax, polysaccharides) without altering the aconitic acid content. Some investigations have been carried out<sup>20</sup> and the process deserves to be studied again in order to avoid the addition of lime and to give sufficient yield on filtration.

#### Acknowledgement

We particularly thank SIASS for furnishing sugar cane juice obtained in their experimental plant and Société Appléxion for suggestions and comments on the phosphatation procedure.

#### Summary

The variation of the aconitic acid content has been studied according to different cane juice clarification processes intended for the manufacture of sugar employing ion exchange resin

*continued on page 230*

16 Ambler et al.: *J. Amer. Chem. Soc.*, 1945, 67, 1 - 5.

17 Sandera & Patek: *Zeitsch. Zuckerind. Czech. Rep.*, 1933/34, 58, 26.

18 vanHook: *Ind. Eng. Chem.*, 1946, 38, 50 - 54.

19 Kishihara et al.: *I.S.J.*, 1981, 83, 35 - 39.

20 Madsen: *ibid.*, 1973, 75, 163 - 167.

# New developments in the purification of beet sugar

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## Introduction

The title of this paper is "New developments in the purification of beet sugar" and not "New development in juice purification", the reason for this being that, in reality, only a small part of the purification of the beet to white sugar takes place in the so-called juice purification. A major part of the purification takes place in diffusion, where the pulp dry substance is removed, and in crystallization, where the molasses non-sugars are removed, and in reality it is not too important where the non sugars are removed, as long as they are removed in the most economical way with the maximum utilization and added-value of the by-products.

There is another good reason for not speaking only about juice purification with lime. The only big changes which have taken place during this century are the introduction of the partly continuous systems, the use of automatic filters and controllers, and the improvement of the filtrability of the juice. The fundamental chemistry has hardly been changed; we have only been able to understand the processes a little better, to automate them, and to control them, to deliver sludge of higher dry substance and less sugar, but the fundamental chemical work was done by our grandfathers or great-grandfathers.

If we leave out process control and the introduction of new types of equipment and look at the inventions which have changed the chemistry in a sugar factory, we are left with hardly a handful of important developments having taken place during our time or even within the last century. A number of these developments involve the use of ion exchangers, others are the use of antiscalants and other processing aids.

A number of other fundamental changes have been tried here and there in the sugar industry, but the main impression is really that our generation has been quite good at developing new types of machinery and process control, yet we have been more or less a "post-chemical generation". Is it necessary that



R. F. Madsen

the future remains like this, or does there exist or can there be developed or introduced, major chemical changes in an economical way?

In a way it is a high-stakes game to play, because many odds are against us. First of all: Western European factories are, to a high degree through development, improving the efficiency of existing technologies to a point where the potential savings in operation costs are limited. Most factories are at the same time so big that fundamental changes in technology are very expensive, and the risk of scaling-up promising new methods is therefore great.

Even though the risks are great, a few important developments remain yet to be tried, and some very important developments are in the process of trial or development. In this paper I will go into some of the important developments which I foresee.

## Liming of cossettes

Liming of cossettes is not a new idea. In the 1950's, Brieghel-Müller limed the cossettes at the Højbygaard sugar factory in Denmark. The result was immediate. Filtration on first carbonation filters went from running to dripping within an hour, and it took about 8 hours to recover to normal conditions on the filter floor. Later, Randall *et al.*<sup>1</sup> and Camirand *et al.*<sup>2</sup> worked on the method.

The results published by Ponant *et al.*<sup>3</sup> and by Südzucker<sup>4</sup> and recent results in our own laboratories seem to show that somebody will come up with a method which is economically interesting. The potential is, first of all, that much more non-sugar will end in the pulp and much less in the sludge, and that, with traditional presses, pressed

pulp can achieve a dry substance of somewhere between 40 and 50%. This gives the potential for considerable savings in both fuel and lime. In the best results we have, we have further achieved an improvement in juice purity of about 1 point, although the results are not absolutely reproducible.

The main difference between the old and the new approaches is the understanding of the fact that the main reason for the terribly bad filtration obtained before with the method is that the limed juice in the diffuser was not prelimed, and not so much the fact that the alkaline conditions attacked the pulp. It seems possible to prevent the filtration disasters by the use of three different methods:

- (a) Removal of the surface juice from the cossettes before a cold liming of the cossettes,
- (b) Preliming the cossettes at a low temperature,
- (c) Liming the cossettes at a very low temperature.

At present it is unclear whether it is best to carry out the diffusion at normal temperature or, since the cossettes are denatured by the liming, to introduce a cold diffusion, which might take a longer time than the normal hot diffusion.

## Membrane-filtration

Membrane-filtration equipment and processes for the sugar industry in Europe have been developed mainly by our group. Although we built the first industrial plants in the enzyme and dairy industry as early as 1969<sup>5,6</sup>, being a sugar company we have not yet made commercial use of membrane-filtration in our sugar factories, even though two or three obvious applications exist.

Paper presented to the British Sugar Tech. Conf., 1990.

- 1 Zuckerind., 1982, 107, 38 - 45.
- 2 J. Amer. Soc. Sugar Beet Tech., 1981, 21, (2), 159 - 174.
- 3 Zuckerind., 1988, 113, 665 - 676.
- 4 Oral communication at CITS Meeting, 1990.
- 5 Madsen: "Hyperfiltration and ultrafiltration in plate-and-frame systems" (Elsevier, Amsterdam), 1977.
- 6 Idem: "Evaporation, membrane-filtration, spray drying in milk powder and cheese production", Ed. Hansen (North European Dairy Journal, Denmark), 1985.

What are the reasons? For many years, one obvious reason was that membranes could not operate at high temperature, and all experiments ended up, either in the utilization of large amounts of formaldehyde, or in strong bacterial growth.

Other reasons are that ultrafiltration of diffusion juice without a pre-purification is very difficult, and short campaigns together with high investment costs gave a poor return on investments.

During the past two years, two developments have been made which will improve the chances that membranes will be a part of future sugar factories.

(a) There is a reasonable chance that a juice purification using just about the lime needed for cossette liming could be possible if an ultrafiltration

stage were to be introduced somewhere in the juice purification.

(b) Nanofiltration membranes have been developed which have a high capacity, high rejection of sucrose and an intermediate rejection of sodium, potassium, etc. It seems possible to use these membranes at elevated temperatures (above 80°C). If the permeate (filtrate) from these membranes can be used as part of the diffusion water without re-introducing too much of the non-sugars into the juice, we might see a future beet sugar factory with a process scheme having principles like those shown in Figure 1, where the sludge is hard-pressed and supplied directly to the farmers, if no better use is found<sup>7</sup>.

Nanofiltration is proposed, partly because it gives a chance of a 10% reduction of losses in molasses without changing molasses quality, partly

because fluxes around 70 l/m<sup>2</sup>/hr can be achieved, instead of the 20 - 30 l/m<sup>2</sup>/hr possible by reverse osmosis.

The hard-pressed pulp of perhaps 45% dry matter content goes to a steam-heated dryer, from where the vapour supplies the evaporators. Most protein in the beet ends up in the pulp. 0.2 - 0.4% CaO is used for cossette preliming or additional liming of the juice. Purification steps follow, including heating, traditional carbonatation and filtration, ultrafiltration and either a traditional cation exchanger or an anion exchanger, in an order which is difficult to predict today. The purified juice goes to a nanofiltration stage, where approximately 30% permeate on beet is removed with a Brix of approximately 1 and a purity of approximately 58 - 60. This permeate is returned to the diffuser.

Owing to the introduction of hard pressing, it is a problem whether all the permeate can be returned to the diffuser, if a draft of only about 110 is needed. Excess permeate can be treated in a second nanofiltration where the permeate goes out as waste water with almost no BOD. The second concentrate of approximately 20° Bx can be used for molasses dilution. The concentrate from the first nanofiltration goes to the evaporators, where only some 65% water on beet has to be evaporated.

In principle, the sugar house as such is not changed, with the exception that, owing to changes in boiling procedures, it is possible to boil from a thick juice Brix of about 78.

Whether a Quentin process or another process is used to remove sugar from molasses is, for me, very doubtful. We have an excellent domestic market for good molasses, but a rather bad market for low-purity molasses. As molasses utilization does not compete with sugar it might also be uneconomical in the future to remove more sugar from molasses, because it will only reduce the beet production.

We will test whether it would be worthwhile to add some molasses to

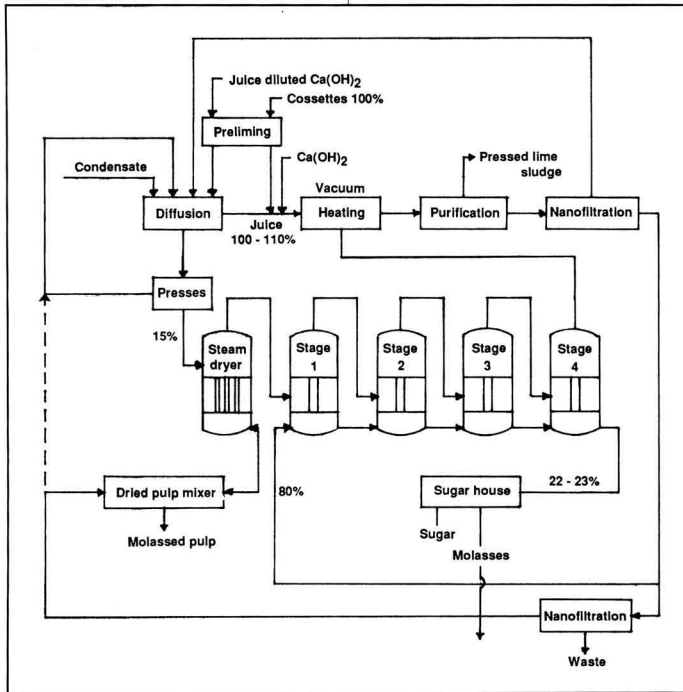


Fig. 1. Schematic imagination of a future beet sugar factory; all percentages are % on beet

<sup>7</sup> Madsen: *Zeitsch. Zuckerind.*, 1977, 27, 643 - 645.

diffusion supply water. In theory, owing to the counter-current principle, some molasses exhaustion should occur, sending molasses with a lower purity to the pulp. This would only be of interest with 100% pulp drying.

*Fuel economy with the predicted process*

Figure 2 gives an idea of the vapour scheme of the new process. The total steam production including that for drying will be around 21 - 22% on beet, corresponding to a fuel consumption of about 1.9% on beet of 7000 kcal coal.

The consumption of electricity will increase by approximately 2 kWh per tonne of beet. For a 10,000 tonnes/day factory, this means that the turbines will have to supply approximately 830 kW more than today. This would be possible if all steam for turbines was supplied at >60 bar and the necessary turbines existed, without having to buy-in electrical power.

Figure 1 shows an evaporator station of only 4 stages. It might be an advantage to use a fifth stage if only in order to reduce the temperature of the

thick juice before the sugar house (Figure 2). This also would secure a surplus of evaporation capacity.

The evaporator inlet temperature can be reduced to around 120°C, thus removing the need to use SO<sub>2</sub>. The liming of cossettes might prevent fermentation in diffusion and thus avoid the use of formaldehyde. The colour of thick juice would probably be reduced by 30 - 50% from the conditions we have today and, owing to the fact that proteins and polysaccharides are kept in the pulp, we would have a chance to produce by-products from lime sludge or anion-exchanger eluate.

An interesting consequence of other changes is that sugar factories having boilers with sufficient pressure (60 bar for turbines, 25 bar for drying) do not need new boilers, even when all pulp is dried by steam drying, because only approximately 22% standard steam is needed in total. Probably, all sugar factories can produce more than that.

*The economic perspective*

The reduction in production costs

and the gain by comparison with existing technology will, per 100 days for a 10,000 tonnes/day factory, be approximately as follows:

	ECU
20% coal at 100 ECU/tonne	2,000,000
1% limestone and coke saving at 20 ECU/tonne	200,000
0.8% dried pulp increase at 120 ECU/tonne	1,060,000
Other chemical savings	140,000
Reduction in molasses losses	700,000
<b>Total</b>	<b>2,700,000</b>

The potential is quite good. The difficult problem is to give a reasonable guess on the investment needed. I hope that the future will show that the investments are not so high that the process changes are unrealistic.

Nanofiltration with current costs would require an investment of approximately 2 million ECU; ultrafiltration probably around 7 million ECU. This indicates that it might be an advantage to operate with nanofiltration, but not with ultrafiltration.

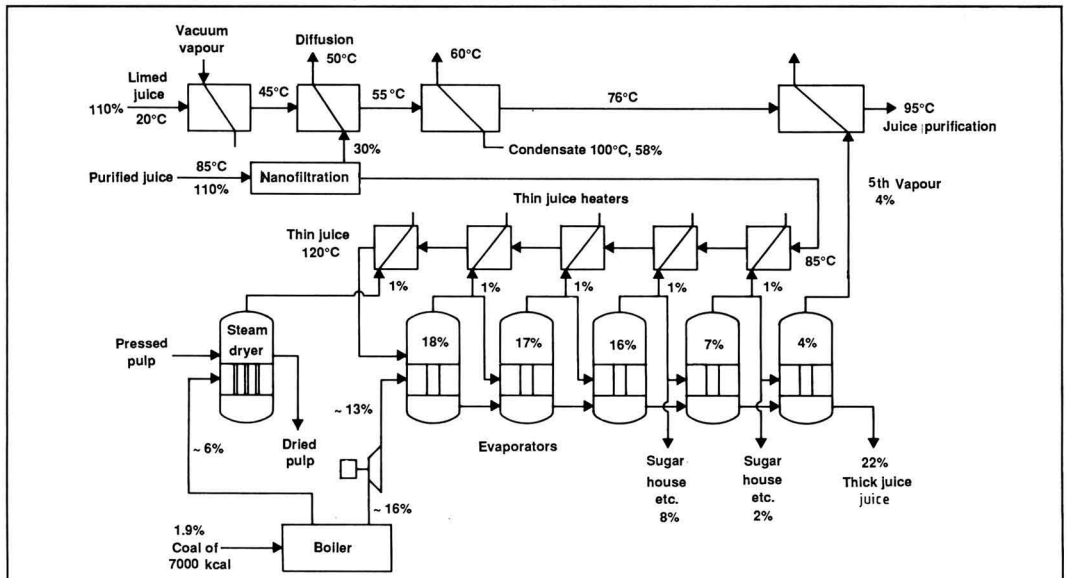


Fig. 2. Schematic vapour scheme for predicted sugar factory; condensate and juice flashing not calculated

# Determination of sucrose using a glucose analyser\*

By Teh-An Hsu and Mark H. Bagley

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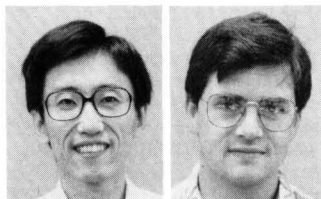
## Introduction

The currently used method of sucrose analysis in the Hawaiian industry<sup>1</sup> is the Jackson & Gillis Method IV, using the Walker inversion method (hereafter referred to as the HST method), which is a modified version of the original Clerget method proposed in 1846 for the determination of sucrose. It is a double polarization method with the inversion induced by hydrochloric acid.

The original Clerget method is well known for its shortcomings in analysing sucrose in impure samples, such as molasses, and, hence, numerous modified versions, as summarized by Browne & Zerban<sup>2</sup>, have been proposed to improve its accuracy. In all of these methods the pol of the sample is measured before and after inversion of sucrose, with the inversion induced by either hydrochloric acid or invertase.

When hydrochloric acid is used, the problem of non-specificity arises, since this acid attacks not only sucrose but also other oligosaccharides in the sample, producing a source of error in the method. Furthermore, the introduction of hydrochloric acid changes the pH of the sample, which, in turn, affects the optical rotation of impurities (invert sugar and nitrogenous substances, for example) and causes errors in the double polarimetry methods<sup>3</sup>. Because of the inherent inaccuracies, the Jackson & Gillis Method IV was discussed at the 17th session of the International Commission for Uniform Methods of Sugar Analysis (ICUMSA), and some proposed that the official ICUMSA method status be withdrawn<sup>4</sup>. Subsequently, this method retained its official status, for the sole reason of its still being a US government method.

Invertase, in contrast to hydrochloric acid, is highly specific in attacking the "sucrose group" (one glucose molecule and one fructose molecule linked together) and has no effect on the polarization of other impurities. Therefore, it appears to be a better choice as the inversion agent. The reliability of the invertase method has



Teh-An Hsu

M. H. Bagley

been recognized to be superior to the hydrochloric acid method; however, invertase has not been adopted for routine analysis because of its cost, uncertain activity, and long inversion time<sup>5,6</sup>.

Since invertase specifically attacks the sucrose group, the saccharides in cane products, known to have a sucrose group – including sucrose,

kestoses, raffinose, and other species in the families of kestose and raffinose<sup>6,8</sup> as shown in Figure 1 – would be hydrolysed in the enzymatic inversion step and would interfere in sucrose analysis. However, except for kestoses and raffinose, the levels of saccharides of these two families in cane products

\* Published as Paper No. 690 in the journal series of the Experiment Station, Hawaiian Sugar Planters' Association.

- 1 "Sugar cane factory analytical control" Rev. Edn., Ed. Payne (Elsevier, Amsterdam), 1968, Ch. 5.
- 2 "Physical and chemical methods of sugar analysis". 3rd Edn. (Wiley, New York), 1941, Ch. 10.
- 3 Bates & Associates: "Polarimetry, saccharimetry, and the sugars" (U.S. Government Printing Office, Washington, D.S.), 1942, Ch. 8.
- 4 Dutton: *Proc. 17th Session ICUMSA*, 1978, 89.
- 5 Chen: "Cane sugar handbook", 11th Edn. (Wiley, New York), 1985, Ch. 20.
- 6 Binkley & Allenburg: *J.S.J.*, 1965, 67, 110.
- 7 Schiweck: *Proc. 17th Session ICUMSA*, 1978, 212.
- 8 "Sugar analysis", Ed. Schneider (ICUMSA Peterborough), 1979.

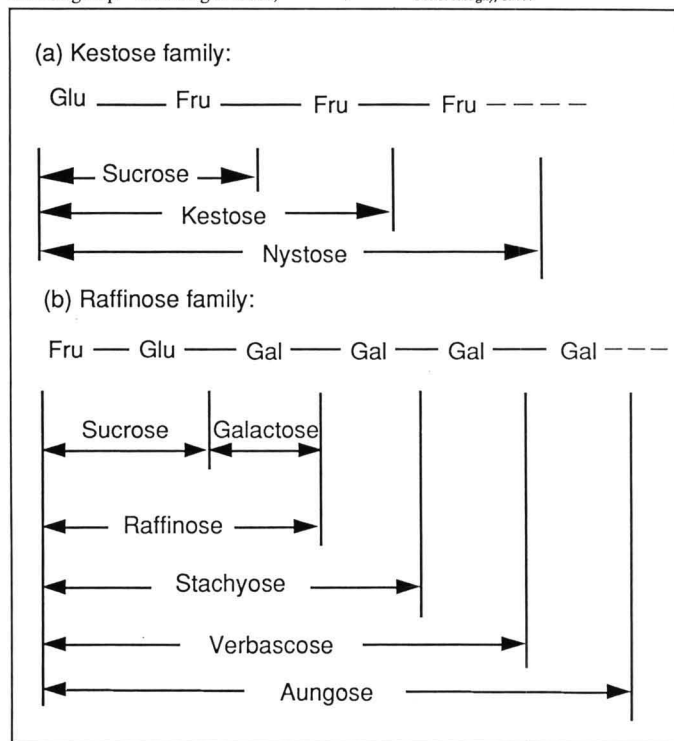


Fig. 1. Saccharides of (a) kestose and (b) raffinose families

are generally reported as negligible<sup>9,10</sup>. Therefore, for practical purposes, the only constituents in cane products to cause any error in sucrose analysis involving double polarimetry with invertase inversion are kestoses and raffinose<sup>11</sup>. According to the limited data found in the literature<sup>6,7,10-17</sup>, kestoses and raffinose in cane molasses, in which their levels are the highest among all cane products, range from 0.2 to 1.7% and 0.005 to 0.6%, respectively, as summarized in Table I.

Devillers *et al.*<sup>19</sup> proposed an alternative enzymatic method for the determination of sucrose, which incorporates invertase inversion of sucrose and colorimetric determination of glucose by glucosidase digestion of glucose (a coloured reagent is produced simultaneously). Since the action of invertase on raffinose does not produce any glucose (cf. Fig. 1) and since polarimetry is not involved, raffinose does not interfere with this method. The only constituents to interfere in this method of sucrose analysis would, therefore, be kestoses. Thus, the method of Devillers *et al.* represents a significant improvement over all the methods involving polarimetry. As a result, it was recommended

by ICUMSA that this enzymatic method be further studied<sup>13,20</sup>.

At present, commercial preparations of invertase with high activities are available at reasonable prices and, since invertase has the above advantages over hydrochloric acid, we felt it appropriate to look into the possibility of using invertase in sucrose analysis.

There is an additional factor which seriously threatens the continued use of the HST (and all other versions of the Clerget) method of sucrose analysis: the banning of the use of lead that appears to be imminent<sup>21</sup>. All methods of sucrose analysis involving polarimetry or colour determination call for clarification of the sample. Among the clarifying agents in use today, the most effective agent for sugar is a lead-containing salt. Because of increased environmental and health concerns, this agent is likely to be banned in the early 1990's.

In the current work, an enzymatic method similar to that of Devillers *et al.* is studied. The difference between the two methods lies in the way glucose is determined; instead of the colorimetric means, a glucose analyser is used. The principles of operating glucose analysers

are given elsewhere by various instrument manufacturers (e.g. operating manuals of Beckman Instruments Inc. and Yellow Springs Instruments Co.); the actual operation of the instruments is simple and rapid. In an effort to examine the possibility of developing a method simpler (less time-consuming) and better (more accurate) than, and be able to supplement or to replace, if necessary, the HST method of sucrose analysis, we investigated the enzymatic method with the use of a glucose analyser. This method is attractive because it does not involve a clarification step, thereby eliminating the use of a lead-containing salt.

#### Instruments, materials, and procedure

##### Instruments

Except for the Glucose Analyzer 2 unit manufactured by Beckman Instruments Inc., all instruments involved in this study are common to sugar industry laboratories. Currently, a Glucose Analyzer 2 unit costs \$5,200.

##### Materials

The enzyme used for inversion in this study is a high-purity, melibiase-free invertase (Cat. No. 14504) purchased from Sigma Chemical Company. The glucose oxidase reagent used in the glucose analyser is supplied by Beckman Instruments Inc. Other chemicals are all of reagent grade, purchased from various chemical companies.

##### Analysis of sucrose

Analysis of sucrose was carried out with the HST method<sup>1</sup>, by a proficient analyst, or with the glucose analyser. The procedures for the latter

Table I. Concentration of kestoses and raffinose in cane molasses

Kestoses, %	Raffinose, %	Origin of molasses	Reference
0.7	>0.005	Cuba	12
0.8	0.17 - 0.28	Brazil	4
0.8	0.08 - 0.25	Mozambique	4
	0.08 - 0.28	Mozambique/East Africa blend	4
0.5 - 0.6	0.08 - 0.21	Australia	4
0.7	0.08 - 0.24	East Africa	4
0.4 - 0.9		(Not specified)	13
0.8 - 0.9		South Africa	13
	0.12	Hawaii	14
	0.10	Australia	14
	0.12	Jamaica	14
	0.13	Taiwan	14
	0.10	South Africa	14
	0.10 - 0.20	(Not specified)	15
	0.35 - 0.60	(Not specified)	15
0.44 - 1.66		South Africa	18
0.9 - 1.0	0 - 0.13	(Not specified)	11
0.65	0.07 - 0.14	(Not specified)	16
0.21	0.04 - 0.14	Hawaii	16
0.6	0.06 - 0.25	(Not specified)	7
1.2	0.25	(Not specified)	17

9 Binkley: *I.S.J.*, 1965, 67, 204.

10 Gross *et al.*: *ibid.*, 1962, 64, 69.

11 Roche: *Proc. 15th Session ICUMSA*, 1970, 52.

12 Binkley: *I.S.J.*, 1964, 66, 185.

13 Dutton: *Proc. 18th Session ICUMSA*, 1982, 87.

14 Gross: *Proc. 15th Session ICUMSA*, 1970, 148.

15 Idem: *Proc. 16th Session ICUMSA*, 1974, 190.

16 Schiweck: *Proc. 18th Session ICUMSA*, 1982, 254.

17 Stachenko: *Proc. 16th Session ICUMSA*, 1974, 85.

18 Nurok & Reardon: *Proc. 49th Ann. Congr. S. African*

*Sugar Tech. Assoc.*, 1975, 94.

19 Devillers *et al.*: *Proc. 15th Session CITS*, 1975, 383.

20 Schäffler: *Proc. 19th Session ICUMSA*, 1986, 94.

21 Chou: *Sugar J.*, 1988, 50, (9), 4.

method are briefly outlined as follows:

1. Analyse the sample with a glucose analyser to determine the pre-inversion glucose content.
2. Use invertase to hydrolyse sucrose completely to invert sugar.
3. Analyse the sample after inversion to determine the post-inversion glucose content.
4. From the difference between the pre- and post-inversion glucose content, calculate the sucrose content in the sample.

Details of the method are given in the Appendix.

#### Experimental

**Effect of buffer solution on the glucose analyser:** A buffer solution is needed to adjust the sample to a pH at which the invertase is active. To determine whether the added buffer would affect the glucose analyser readings, synthetic glucose solutions prepared with and without the buffer were analysed and the results compared.

**Accuracy and precision of the glucose analyser:** A group of eight synthetic solutions, ranging from 50 mg to 400 mg glucose per decilitre (dl), was used to examine the accuracy and precision of the glucose analyser. The 150 mg/dl (0.15% on weight basis) solution was used as the standard of calibrating the analyser. After calibration, the eight solutions were repeatedly analysed in random order to give a total of 10 readings for each solution. The results were then statistically analysed.

**Sample dilution:** Beckman Instruments Inc. indicated that samples should be prepared (diluted) so that, for the analyser results to be reliable, the readings are less than 450 mg/dl. Since the glucose contents of any factory sample before and after inversion would differ quite substantially, we investigated the need of diluting the sample to two different concentrations (two-concentration dilution) for the pre- and post-inversion glucose determin-

ations. This was done by comparing analyses made by single-concentration dilution (sample diluted to only one concentration for the pre- and post-inversion glucose contents to be determined) and by two-concentration dilution.

**Interference of impurities in the glucose analyser and HST methods:** A group of synthetic sucrose solutions was prepared, in which known quantities of such impurities as cellobiose, maltose, raffinose, and starch were added (one saccharide added to each solution). The solutions were then analysed with the glucose analyser and the HST method for comparison of the interference of these impurities in the two methods. Next, the interference of cellobiose, maltose, and raffinose in molasses was examined by comparing the sucrose content analysed with the two methods before and after inoculation of known quantities of these saccharides.

**Comparison of accuracy and precision of the glucose analyser and HST methods:** To determine the precision of the two methods, one final molasses sample diluted to one-half of its strength (for ease of handling) with the buffer solution specified in the Appendix was repeatedly analysed for its sucrose content with the two methods

and the results statistically analysed. In a separate test, a subsample of the diluted molasses was fortified with a known amount of sucrose. We then compared how accurately the two methods could determine the fortified sucrose.

**Comparison of costs and labour requirements:** The costs for performing the two methods were estimated using available equipment and chemical costs. The labour required was estimated from our testing and operating experiences.

#### Results and discussion

**Effect of buffer solution on the glucose analyser:** A relatively strong buffer solution (as described in the Appendix) was selected in this study for enzymatic inversion. In an exploratory test, it was found that the glucose analyser readings varied slightly with the presence of the background buffer solution (Table II). Although the variations were within the precision limits specified by the manufacturer, repeated tests showed that the analyser reading was indeed dependent on the presence of the buffer. It was thus apparent that the performance of the analyser would be improved by preparing all samples, including the standards for analyser calibration, in the buffer. As a result, throughout the study, samples and standards were all prepared/diluted with the buffer. The make-up of the buffer solution was so selected that it would be sufficiently strong to provide a nearly uniform salts background in the two samples which resulted from two-concentration dilution and that inversion would be active without any further adjustment of pH.

**Accuracy and precision of the glucose analyser:** Table III shows the

**Table II. Effect of buffer solution on glucose analyser**

Glucose solution	Analyser readings	Average
150 mg/dl in distilled water	150, 151, 150, 150, 151, 149	150
150 mg/dl in buffer	152, 153, 151, 152, 152, 153	152

Note: Beckman Instruments specifies that, with a glucose standard of 150 mg/dl, the analyser precision allows readings to vary between 147 and 153

**Table III. Accuracy and precision of the Beckman glucose analyser**

	Glucose in synthetic solution, mg/dl							
	50.0	100.0	150.0*	200.0	250.0	300.0	350.0	400.0
Mean of 10 readings, mg/dl	49.6	99.6	149.8	198.4	248.6	296.7	345.6	395.5
Standard deviation, mg/dl	0.7	0.7	0.6	1.2	1.8	1.0	2.6	1.4
Coefficient of variation, %	1.41	0.70	0.42	0.59	0.71	0.32	0.75	0.43
95% confidence level error, %	± 1.01	± 0.50	± 0.30	± 0.42	± 0.51	± 0.23	± 0.54	± 0.26
Relative deviation of mean from true value, %	0.80	0.40	0.13	0.80	0.56	1.10	1.26	1.13

\* Solution used for calibration of analyser



statistical analysis results of the series of tests for determining the accuracy and precision of the glucose analyser. Data in the last row indicate that the analyser response is slightly non-linear and the deviation from linearity appears to increase as the reading becomes 300 mg/dl or higher. At the 95% confidence level, the error is  $\pm 0.5\%$  or less for readings greater than or equal to 100 mg/dl.

It therefore appears that the accuracy of the results obtained for unknown samples could be increased by using a standard curve constructed from multiple standards. (The manufacturer recommends a one-point calibration of the analyser with perfect linearity assumed.) Throughout this study, efforts have been made so that samples prepared would result in analyser readings between 100 and 300 mg/dl, and because the procedure is simple, multi-point standard curves have been prepared daily for use.

**Sample dilution:** The results in Table IV show that the glucose and sucrose contents could be better determined by two-concentrations dilution of the sample. In the case of one-concentration dilution, it was not unexpected for the analysed pre-inversion glucose content to show a relatively high error, since the analyser reading was low (as low as 50 mg/dl). As a result, two concentration dilution was employed for sucrose analysis in the enzymatic method in this study.

**Interference of impurities in the glucose analyser and HST methods:** When cellobiose, maltose, and raffinose were present, whether in synthetic sucrose solutions or in molasses, the sucrose measurement by the glucose analyser method, as shown in Tables V and VI, was not affected. The HST method, however, was strongly affected by raffinose. This was not surprising since, as was pointed out earlier, raffinose would interfere in the HST, but not in the glucose analyser, method. (As may be seen from Table I, raffinose in natural cane molasses is generally low; we used high raffinose levels in the

**Table IV. Glucose analyser analysis of a synthetic solution of 1.98% glucose and 16.77% sucrose**

Analysis No.	One- or two concentration dilution	BI/AI	Glucose, %	Sucrose, %
1	Two	BI	1.99	16.69
		AI	10.78	
2	One	BI	2.05	16.59
		AI	10.78	
3	Two	BI	2.00	16.83
		AI	10.86	

\* BI = before inversion; AI = after inversion

**Table V. Effect of cellobiose, maltose, and raffinose on the HST and proposed glucose analyser methods of sucrose analysis with synthetic solutions**

Sample description	Sucrose, %	
	HST method	Glucose analyser
10.00% sucrose	10.07	10.04
10.00% sucrose + 10.00% cellulose	9.98	9.96
10.00% sucrose + 10.00% maltose	10.15	10.04
10.00% sucrose + 8.48% raffinose	16.94	10.00
9.96% sucrose + 4.23% raffinose + 0.36% glucose	13.34	9.94

**Table VI. Effect of cellobiose, maltose, and raffinose on the HST and proposed glucose analyser methods of sucrose analysis with molasses\***

Saccharide added	Sucrose, %			
	HST method		Glucose analyser	
	Before saccharide addition	After saccharide addition	Before saccharide addition	After saccharide addition
2.64% cellobiose	23.13	23.07	22.37	22.55
2.68% maltose	15.01	14.63	14.13	14.09
2.21% raffinose	19.60	21.35	18.83	18.77

\* Various amounts of sucrose added to 1:1 diluted final molasses before addition of saccharides; dilution with buffer solution

study merely for the purpose of demonstrating the interfering effect.)

The last two rows in Table V show that the HST method measured raffinose as if it were 81.8% and 80.0% sucrose, respectively. Table VI shows that, in molasses, 79.3% of inoculated raffinose was detected as sucrose by the HST method. Since the slight difference among 81.8%, 80.0%, and 79.3% were insignificant (had the figure 81.8% been used, the sucrose contents of the 5th sample in Table V and the 3rd sample in Table VI would have been calculated to be 13.42% and 21.40%, respectively, which, compared with the analysed 13.34% and 21.35%, are within the analytical error limits), it was concluded that raffinose interferes with the HST method by over-estimating the sucrose content by an average of 80.4% of the

amount of raffinose.

Tables V and VI indicate that the effects of maltose and cellobiose in the HST method are probably insignificant.

Table VII shows that starch has a moderate but definite effect on sucrose analysis by the HST method and that the effect appears to increase with the amount of starch. The effect on the glucose analyser method, however, is clearly insignificant. This appears to be consistent with the previous note that starch should not interfere in the glucose analyser method.

**Comparison of accuracy and precision of the glucose analyser and HST methods:** Table VIII compares the repeated analyses of a diluted (1:1) molasses sample by the HST and the glucose analyser methods. The statistical analysis shows that the precision of the

**Table VII. Effect of starch in HST and glucose analyser methods of sucrose analysis with synthetic solutions**

Sample composition, %			Sucrose, %	
Sucrose	Starch	Glucose	HST method	Glucose analyser
9.97	4.99	1.99	10.76	9.91
10.01	3.00	2.00	10.44	10.01
10.01	1.00	2.00	10.27	10.02

two methods appears to be comparable.

The HST method, however, resulted in a statistically significantly higher sucrose content. Since there is no definitive method against which the accuracy of the two methods can be assessed when non-synthetic samples are involved, we relied on recovery experiments to evaluate which of the two methods determined sucrose more accurately. A subsample of the above molasses was fortified with a known amount of sucrose and then analysed by the two methods. The results in Table IX indicate that both methods are able to recover the fortified sucrose accurately. The differences between the two methods for sucrose determinations as shown in Tables VIII and IX thus suggest that the HST method has a tendency to overestimate the sucrose content in molasses. The observation is in agreement with the non-specificity of the HST method discussed previously. We conclude, therefore, that the accuracy of the glucose analyser method is better.

**Comparison of costs and labour requirements:** The instrument and the estimated operating costs for the two methods are given in Table X. We realise that a saccharimeter is commonly found in all sugar laboratories and it is used for not only sucrose analysis but also pol analysis; however, the operating cost (\$0.22 per analysis for the glucose analyser method *versus* \$0.70 per analysis for the HST method) shows that the glucose analyser method is significantly less costly.

Because the invertase recommended for use (that used in this study) is a highly pure commercial product with high activities, and since only a very small amount of it is required for each analysis, the cost of enzyme is extremely

low, at approximately 2 cents per analysis. Concern that the enzyme might be too costly and the activity uncertain,

**Table VIII. Accuracy and precision of the HST and proposed glucose analyser methods of sucrose analysis\***

	Sucrose, %	
	HST method	Glucose analyser
Mean (of 6 analyses)	16.85	15.53
Standard deviation	0.18	0.11
Coefficient of variation, %	1.05	0.74
95% confidence level error, %	±1.11	±0.77

\* Sample used was a 1:1 diluted final molasses; dilution with buffer solution

**Table IX. Comparison of the HST and proposed glucose analyser methods in recovering sucrose added to molasses\***

Sucrose added, g	Sucrose, %		Added sucrose recovered, g	
	HST method	Glucose analyser	HST method	Glucose analyser
0	16.85	15.64	-	-
23.0	22.98	21.93	22.8	23.1

\* Molasses sample used was a 287.0g 1:1 diluted final molasses; dilution with buffer solution

**Table X. Estimated cost of HST and proposed glucose analyser methods**

Cost item	Method	
	HST	Glucose analyser
Capital cost (Instrument)	\$12,200 (Saccharimeter)	\$5,200 (Glucose analyser)
Operating cost		
Reagent/chemicals	Nil	\$0.10/analysis
Invertase	-	\$0.02/analysis
Supplies	\$0.70/analysis (Filter aid and filter papers)	\$0.10/analysis (Pipette tips)

**Table XI. Estimated time requirements of HST and proposed glucose analyser methods**

HST method:	1 analyst can perform a maximum of 6 analyses/shift
Proposed glucose analyser method:	1 analyst can easily perform 10 - 15 analyses/shift

therefore, appears to be unjustified.

Table XI shows a comparison of the labour requirements of the two methods. It is clear that the glucose analyser method is significantly less time-consuming than the HST method.

**Other comments:** It was previously mentioned that one of the reasons invertase has not been adopted for routine analysis is that the reaction time is too long. This was found not to be a problem in this study. Apparently, primarily because of the low sucrose concentrations used, inversion (and the mutarotation necessary for glucose determination with the glucose analyser) was found to be complete within 30 minutes at room temperature.

Other than the advantages already discussed, the glucose analyser method has the following advantages:

1. Sample centrifugation, clarification, and filtration are not necessary. Because the method is insensitive to suspended solids and colour, samples

can be analysed directly after the necessary dilution. This represents a significant advantage over the HST method (and all other methods of sucrose analysis thus far known to be in existence).

2. Sample size required is small. Only a few grams of sample is required for each analysis. This implies that the method is particularly promising for



# Cane sugar manufacture

## The efficiency of a sieve plate scrubber for prevention of air pollution

C. J. Chang and J. T. Lee. *Taiwan Sugar*, 1989, 36, (6), 14 - 15 (Abstract only).

If flue gases from bagasse boilers are untreated, > 6000 mg/m<sup>3</sup> of dust particles are emitted. Since the gases are of high moisture content (10 - 14%), the wet scrubber is more suitable. In the past, water spray and impingement scrubbing systems have been the major methods used; but their efficiency (65 - 85%) has not been enough to allow the emitted gases to meet the requirements of a new air pollution control standard. For the water and gases to be thoroughly mixed so as to provide a high particle removal efficiency in wet scrubbers, the induced-draft fan has to be sufficiently powerful; however, since alteration in the flow path and acceleration of the gas flow in a Venturi-type scrubber will cause a noticeable pressure drop, the system is too costly and energy-consuming. In a sieve plate scrubber, the dust particles are accelerated to impinge on the water layer while the flue gases are divided into thousands of small bubbles by the orifices in the sieve plate; as the particles pass through the water, they collect in the bubbles, so that a 98.5% efficiency is achieved. Two scrubbers with a single sieve plate and a 20 mm high water layer installed at Hsinying and Chiali sugar factories had an efficiency greater than 98.5%, with an emitted dust load of 110 - 160 mg/m<sup>3</sup>. Modifications to 15 units at another 7 factories involved installation of double sieve plates with a water layer 50 mm high; their efficiency exceeded 99%, with a final dust load of 30 - 150 mg/m<sup>3</sup> which met the requirements of the new official regulation.

## Boiling low-grade massecuite by personal computer

Y. C. Hsiao, F. U. Liu and Y. H. Lin. *Taiwan Sugar*, 1989, 36, (3), 16 - 17 (Abstract only).

In the past, low-grade massecuite was boiled by an experienced operator; however, although he worked very carefully in the seeding stage, the crystals were not always very uniform in shape and quantity and it was not possible to stabilize sugar recovery. A computerized system, with software written in BASIC, has been designed for pan boiling. Each of six phases in the boiling cycle is based on pan level, conductivity or a sequence timer. Level and conductivity signals are sent to the computer which controls the feed valves. The program includes function modules for valve control, data acquisition, phase determination and control loop calculation. An experiment at Hualien sugar factory in 1988/89 showed that the conductivity curve for low-grade massecuite was much better than with manual boiling and no false grain or conglomerates were found during the graining or boiling phase.

## Calculation of factory data with computers

Z. P. Chiang. *Taiwan Sugar*, 1989, 36, (6), 17 (Abstract only).

Two main benefits of the Lotus 1-2-3 package software for calculation of factory data are indicated: (1) when the formula cells are copied to other cells, the formulae in the new cells will adopt the data of the cells located in the new relative positions; because each calculation step applies the same formula, keying-in can be reduced; and (2) the iterative system of calculation not only matches the cyclic processes such as imbibition, seeding, etc. but it avoids the necessity of having to solve simultaneous equations and simplifies the trial-and-error method by using the Newton approximation method. The step-by-step procedures applied to cane milling, evaporation and boiling are described.

## Effect of biocides on minimizing post-harvest deterioration in sugar cane

J. Kapur and K. P. Sharma. *Bharatiya Sugar*, 1990, 15, (3), 47, 49.

Experiments are reported in which topped and stripped cane stalks were soaked in various chemicals and then heaped for exposure to the sun during 6 days. Results showed that a 0.015% solution of Action ID (allyl phenoxy-polyglycol ether complex with 1.6% available iodine) had the best effect by reducing sucrose losses to 0.24% per day compared with 0.49% in the control and limiting the daily increase in reducing sugars to 0.30% (compared with 0.54%) and the hourly rate of dextran formation to 32 ppm (compared with 157 ppm).

## Wide-gap plate heat exchangers in the cane sugar industry

R. S. Deshmukh. *Bharatiya Sugar*, 1990, 15, (3), 51, 57.

Alfa-Laval wide-gap heat exchangers were used in trials on raw and sulphitation juice heating with 3rd effect vapour. Results showed a high heat transfer coefficient in the range 1800 - 2100 kcal/m<sup>2</sup>/hr/°C with a heat surface area less than half that of a shell-and-tube unit of the same duty, a low pressure drop, 30 days operation before cleaning was needed, no floc breakage in the sulphitation juice and the ability to use low-pressure vapour at 0.4 atm at temperatures down to 75°C. The heat exchanger is compact and easy to clean and has a low hold-up volume.

## Use of semi-Kestner and double-effect vapour cells for cane juice evaporation

D. S. Lande, M. B. Londhe and D. B. Jambhale. *Bharatiya Sugar*, 1990, 15, (4), 9 - 14.

The effects of replacing a conventional pre-evaporator with a semi-Kestner vapour cell followed by a quintuple-effect evaporator at one factory and with a double-effect vapour cell followed by a quadruple-effect system at another were determined and the results tabulated. It was found that the high temperatures used in the pre-evaporators at both factories caused increase in sugar

losses, juice colour, scale and entrainment while difficulty in maintaining stable conditions in the double-effect vapour cell caused marked fluctuation in the vacuum pan vapour load. It was not possible to obtain any indication of savings in bagasse fuel.

#### **Problem of scale formation in Indian sugar factories. II. Chemical composition of scale from 1st and 2nd evaporator bodies in Maharashtra sugar factories**

M. R. Shivde, S. D. Borawake, V. S. Keskar, J. D. Mane, A. Kumar and S. J. Jadhav. *Bharatiya Sugar*, 1990, 15, (4), 19 - 20, 23 - 24.

The procedures used to analyse scale taken from the 1st and 2nd effects at a number of factories are described and results for 42 samples are discussed. Calcium phosphate constituted up to 40% of the composition, so that the scale was soft; an average of 12% calcium silicate was also found followed by organic acid salts and magnesium phosphate (each 8%) and magnesium silicate (4%).

#### **Use of lime in juice purification - a panorama**

N. Mohan and A. Bajpai. *Indian Sugar*, 1990, 39, 739 - 743.

Aspects discussed include the required limestone composition, lime kiln operation, milk-of-lime preparation and treatment and factors affecting lime consumption in clarification.

#### **Ideal cane juice clarification**

S. B. Pendse, B. D. Sidnale and S. Y. Jadhav. *VSI Bull.* (Vasantdada Sugar Inst.), 1990, 1, (1), 13 - 15.

The sulphitation process and the physico-chemical changes that take place in the juice are described, its advantages over carbonatation indicated, the role of phosphates and flocculants examined and advice given on how to overcome the mud problem and obtain optimum clarification.

#### **Belize - a growing success**

F. Curtis. *Tate & Lyle Times*, 1990, (53), 4 - 8.

A brief account is given of the Belize sugar industry which involves one factory, Tower Hill, supplied by 4875 cane farmers. Market problems forced the closure of a second factory, Corozal, in 1985, since when cuts have been made in the US sugar quota; despite these, the industry has proved profitable and the outlook is very bright.

#### **Influence of minimum air temperatures and stale cane deliveries on the cane/sugar ratio in Jamaica**

M. E. A. Shaw. *Sugar y Azúcar*, 1990, 85, (4), 13 - 14.

It has been found that post-harvest deterioration of cane is by far the most important controllable factor affecting the Jamaican sugar economy. Statistical analysis also showed that 75% of the variance in the cane/sugar ratio between years in Jamaica was attributable to the effect of minimum air temperature, while 5% of the variance was a result of rainfall effect. Empirical equations are presented for the various relationships. A 3-year program aimed at reducing the cane/sugar ratio succeeded in 1987 when 66% of the cane was delivered within a target 48 hr, reducing the ratio by approx. 1.4. It is calculated that delivery of 80% of the cane within this time would give a ratio of about 10 in an "average juice year" and one of about 9.6 in a "good juice year".

#### **An overview of the sugar industry in Trinidad and Tobago**

V. Ramlogan. *Sugar y Azúcar*, 1990, 85, (4), 19, 22 - 23.

A brief account is given of the activities of Caroni (1975) Ltd. which produced 97,000 tonnes of sugar from 1.2 million tonnes of cane in 1989 when consumption was estimated at 58,000 tonnes. With restructuring, a reduction in the level of production to 75,000 tonnes in 1993, closure of one of the two factories

and expansion of the other are planned as well as a degree of diversification.

#### **Boiling of solutions and juices in a single-tube evaporator**

D. Clerch A., O. Herrera M. and R. González Q. *Centro Azúc.*, 1989, 16, (2), 3 - 12 (*Spanish*).

The design and operating parameters of an electrically heated vertical, single-tube evaporator are described. The unit was used to obtain values of the surface coefficient of heat transfer (SCHT) of sugar solutions and juices at 20° and 60°Bx as a function of temperature difference and heat flow. The results are intended for comparison with data from other experiments to see if surfactants increase values of SCHT.

#### **Storage of consumption white sugar obtained by the sulphitation method**

M. Muro M., I. Machado L., E. Garnica and A. Li A. *Centro Azúc.*, 1989, 16, (2), 13 - 17 (*Spanish*).

White sugar stored in polyethylene-lined jute sacks for 260 days at approx. 40°C was analysed for colour, pH, moisture, pol and ash. Results showed a progressive fall in pol and pH and increase in moisture and ash contents and a slight increase in colour. While the sulphite ion has a positive effect on sugar storage, this effect only lasts 3 months after which deterioration occurs in brightness, odour and taste.

#### **Extraneous matter and the inorganic non-sugars content in cane juices**

W. Burgos G., E. Valdés B., M. García G. and M. Leyva A. *Centro Azúc.*, 1989, 16, (2), 24 - 27 (*Spanish*).

The trash content was determined in cane samples taken during two months and extracted juice and mill juice analysed for Brix, pol, pH, reducing sugars, ash, silica, Ca<sup>++</sup>, Mg<sup>++</sup>, PO<sub>4</sub><sup>---</sup>, Na<sup>+</sup>, K<sup>+</sup>, Fe<sup>++</sup> and Cl<sup>-</sup>. Results showed a significant increase in impurities content

with trash content which also raised the acids content and buffer capacity and hence the milk-of-lime consumption.

#### **Study on insolubles in sugar factories of Matanzas province**

P. R. Pérez E., S. Díaz S., M. Torres and I. Díaz C. *Centro Azúc.*, 1989, 16, (2), 28 - 35 (Spanish).

The insolubles content was determined in filtered, limed, clear and mixed juices as well as clarifier mud at three Cuban sugar factories as an indication of purification efficiency. Values are tabulated but, for a number of reasons, it was not possible to obtain satisfactory correlation, and the investigations were to be continued.

#### **Evaluation of the process of juice purification by sulphitation at Argelia Libre agro-industrial complex**

A. C. Muñoz, G. Mayo P., J. Gil O., A. Enrich and J. C. Obregón P. *Centro Azúc.*, 1989, 16, (2), 36 - 40 (Spanish).

Analysis of mixed, sulphitation, limed and clear juice samples showed wide variation in pH and SO<sub>2</sub> content. The temperature of mixed juice and of SO<sub>2</sub> entering the sulphitation vessel did not conform to established norms, so that gas absorption was affected and cooling of the excessively hot gas was inefficient. Milk-of-lime dosing did not correspond to juice flow (explaining the fluctuations in pH) which was adversely affected by variations in the milling rate.

#### **Some thoughts on the energy consumption of prime movers in agro-industrial complexes of Villa Clara province**

R. Espinosa P., M. J. Carrillo A., S. Machado B. and A. Reymond A. *Centro Azúc.*, 1989, 16, (2), 47 - 49 (Spanish).

Analysis of the thermo-energy consumption of steam engines, turbo-sets and turbines in nine Cuban sugar factories demonstrated the adverse effect on thermodynamic efficiency of excessive

age of the equipment.

#### **Burning cane waste. I. Fuel properties**

A. Aguilar, U. Peña, P. Friedman and B. Brito. *CubaAzúcar*, 1989, (Jan./March), 40 - 47 (Spanish).

Although trash from a cane reception station or cane cleaner was found to contain 2 - 4 times more ash than bagasse, it contained less moisture and had a higher volatiles:fixed carbon ratio. However, its particle size must be reduced to that of bagasse for it to be suitable as a fuel, and some indications are given of the efficiency of various systems.

#### **Continuous centrifugalling of commercial massecuites**

M. Elena C., P. Pérez and J. Lodos. *CubaAzúcar*, 1989, (Jan./March), 49 - 56 (Spanish).

The effect of reheating *B*-massecuite (i) immediately after dropping from the pan was compared with that of (ii) simultaneous heating before and during purging in a continuous centrifugal. Results showed that, provided the pressure of heating steam did not exceed 6 lb/in<sup>2</sup>, method (ii) raised the massecuite temperature sufficiently higher than method (i) to reduce viscosity, increase centrifugal capacity and improve sugar quality without any change in the molasses properties.

#### **Material balance for the pan station as a means of establishing process efficiency**

C. Silverio R. and M. C. Santibáñez. *ATAC*, 1989, 48, (4), 24 - 27 (Spanish).

It is shown that the overall coefficient of retention obtained from the s-j-m formula of Noël Deerr as an indicator of sugar recovery fails to provide a sure guide to the efficiency of the pan station. The efficiency coefficient of David, which defines the quantity of sugar produced per 100 parts of solids in the system, is offered as an alternative; it

allows calculation of the quantity of material recycled, so that knowledge of the Brix of a given product will allow calculation of the quantity of water to be evaporated and hence the total vapour requirement. Its application is demonstrated by reference to two actual case histories.

#### **Effect of cavitation on *Bacillus subtilis* in pure solutions**

O. Rodríguez, P. V. Pérez and X. Meneses. *CubaAzúcar*, 1989, (Apr./June), 15 - 19 (Spanish).

*B. subtilis* is a thermophilic bacterium that occurs throughout the sugar manufacturing process and even creates difficulties in raw sugar storage. Experiments were conducted on its control by cavitation. Solutions of Tween 20 containing 10<sup>5</sup> cells/ml of the micro-organism isolated from crusher juice were fed by force pump up into a cavitation chamber and from there via a bent tube section down into a tank. Reduction of the microbial population by 90% needed only 3 min treatment at 50°C or 7 min at 40°C. By contrast, 4400 ppm of 37% formalin was needed to give the same results with vegetative cells and 10,000 ppm with sporulated cells at a much greater cost than that of the electricity used for cavitation treatment.

#### **Use of hydraulic drives for individual movement of cane mill rollers. Results from three seasons of experience**

J. F. Abón. *ATAC*, 1989, 48, (5), 40 - 50 (Spanish).

Experiences in the use of mill hydraulic systems in a Cuban sugar factory are discussed and their advantages indicated. Best results were obtained with use of different peripheral speeds in the tandem; this either reduced the power consumption or increased crushing and extraction. The average true power consumption figures did not match values calculated using the formulae generally used, and an improved formula is needed.

# Beet sugar manufacture

## The PSW-1000M vertical pulp press - its modifications, drive and control

K. Buck. *Gaz. Cukr.*, 1990, 98, 43 - 46 (Polish).

The main features of the Polish-built PSW-1000 M vertical pulp press and its variants are described. The models are designed to press to a pulp dry solids content of 18 - 21% at a daily throughput of up to 800 tonnes; spindle speed is up to 4.3 rpm.

## Energy savings in the carbonatation process - results of experiments and practical experience

W. Lekawski, J. Serafin and K. Urbaniec. *Gaz. Cukr.*, 1990, 98, 46 - 49 (Polish).

Experiments and results of factory experience in Spain and Poland are reported in which steam consumption in carbonatation was reduced by 0.8 - 1.6% on beet by moistening and heating the CO<sub>2</sub> from 32° to 70°C with condensate and conducting the carbonatation process in a pressure vessel at 0.15 MPa. CO<sub>2</sub> utilization efficiency also rose. Comparison is made with conventional use of dry, cold gas.

## Washing with syrup in white sugar centrifugals

G. Möller. *Lebensmittelind.*, 1990, 37, 82 - 83 (German).

At Güstrow sugar factory, experiments were conducted on replacement of some wash water with wash syrup in a batch white sugar centrifugal. From the results, optimum conditions were found to be: application of a 71°Bx syrup of 95 purity at 5% on massecuite over 30 sec, followed by a 10 sec interval and then application of 1.2% wash water over 15 sec. While the white sugar quality was unaffected, there was a 1% reduction in water consumption per charge, 30% less sugar was dissolved, the quantity of run-off was 34% lower, crystal yield was about 2% higher, electricity and fuel consumption fell, there was a decrease

in reboiling and associated losses and sugar house performance improved.

## The 1989 campaign in GDR sugar factories

D. Urban. *Zuckerind.*, 1990, 115, 353 - 358 (German)..

Results for the 41 out of 43 operable factories in East Germany that processed beet during the 1989 campaign (24 white sugar and 15 raw sugar factories plus two thick juice plants) showed an average sugar yield of 11.05% on beet at an average sucrose content of 15.39%. The average beet quality tended to be poor, with low sugar/non-sugars ratio and juice purity, inadequate effective alkalinity and high lime consumption and molasses losses. Raw juice quality was affected by prolonged retention in oversized DDS diffusers (as used in most factories) and a high non-sugars content in the water; where condensate was used, considerable quantities of HCl (employed instead of sulphuric acid and SO<sub>2</sub> because of their melassigenic effects) had to be used to combat the significant ammonia content, although treatment with pan vapour at some factories reduced the ammonia level to 30 - 40 mg/litre and saved acid. The use of flue gas SO<sub>2</sub> for diffusion water treatment at other factories was economically sound and reduced emission but was not advisable because of the non-sugars introduced with it. While a good half of the factories use conventional carbonatation, with slight modification in some cases, the rest employ a system that includes precarbonatation; a vessel for precarbonatation, juice circulation and intermediate liming at Haldensleben was to be tested in the 1990 campaign. Programmed filter-thickeners with backwashing at Haldensleben and Güstrow gave Fk values of 1 - 4 and achieved a maximum mud throughput of 1200 - 1300 litres/m<sup>2</sup>/hr; of major disadvantage was the time (3 min) needed to obtain a clear filtrate by backwashing (which necessitated check filtration) and the difficulty of detecting turbid runs sufficiently quickly. In order to overcome the

problem of pH fluctuations and hence possible overliming caused by a considerable time lag between juice feed to 2nd carbonatation and its flow past the thick juice pH measuring point, a system was devised in which some of the thin juice stream was heated and passed through a packed column to simulate the conditions in the evaporator, the ammonia and CO<sub>2</sub> rapidly driven off and the change in pH measured in just a few minutes. Only three factories achieved a specific steam consumption below 40% on beet; economy measures have concentrated on the use of secondary energy and more effective heating of intermediate products as well as cleaning of juice heaters every 8 - 10 days with 5 - 10% NaOH at 0.01 m<sup>3</sup> per m<sup>2</sup> heat surface area which completely removed scale formed from protein in 1 hr and restored heat transfer to its initial value. A HUB tubular heater developed at Humboldt University, Berlin, gave high heat transfer coefficients (2500 - 4300 W/m<sup>2</sup>/°C) and clean heating surfaces with high flow rates (2.6 - 2.8 m/sec) throughout the campaign, although preliminary results of juice heating in a contact heater showed a 35°C temperature rise at a dilution rate of 0.8% on Brix. Polystabil VZ antiscaling agent allowed particularly stable heat transfer coefficients at two out of five factories, and only at Güstrow was there need for boiling-out. Another product, Scopacryl LW 300, of similar composition, failed to give comparable results. Prolonged retention in conventional evaporators caused increase in colour and reducing matter, with inadequate alkalinity and a thick juice pH 0.46 units below that of thin juice having major effect; average thick juice purity was 89.1 (the lowest for the past 45 years). Washing with melt liquor in the white sugar centrifugals at Löbau increased the crystal yield by 5%, reduced crystal dissolution and ensured high sugar quality by comparison with water washing. Improvement in sugar quality also resulted from the use of centrifugal leaf filters despite some drawbacks. Affination of B- or C-raw sugar in continuous centrifugals has

been found to improve purity by 5 units and reduce colour and ash contents by >50%; the aim is to obtain a sugar that can be melted and processed directly to white sugar without recrystallization, thereby allowing steam economies.

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### The new Braunschweig juice purification scheme, System 89 Tulln

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G. Schäffer. *Zuckerind.*, 1990, 115, 371 - 375 (German).

A new BMA carbonation system developed in collaboration with Tulln sugar factory in Austria is described. Since CO<sub>2</sub> utilization with the previous system was unsatisfactory because of inadequate distribution at high juice throughput, each carbonation vessel was provided with an arrangement based on that of Selwig & Lange GmbH in which the gas was injected through slits on the underside of horizontal pipes just above the floor, with freely moving pieces of metal keeping the slits free from scale. A central pipe having a diameter that took up a major part of the vessel diameter was mounted above the distribution pipes; juice entered it tangentially, was forced upwards by the ascending gas stream and left the top of the pipe to flow down between it and the wall of the vessel, being thus subjected to about a 20-fold circulation. The juice was drawn off at the bottom and fed, via a ventilated pipe loop that maintained a constant juice level, to the next vessel. Besides the improvement in gas circulation and utilization, it was thought that this arrangement would largely prevent over- and under-carbonation and associated colour formation. Because of space restrictions, most of the vessels in the system were used for a combination of processes, with the intermediate liming tank (after precarbonation) also being used to pump out the juice, the 1st and 2nd carbonation vessels being used to pump out the corresponding juice and the main liming tank also being used for after-liming of filtrate from the filter-thickeners after 1st carbonation. Full details are given of the

entire purification process and of the performance of the station with indications of the improvements by comparison with the earlier system.

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### The use of infinite series for optimizing placement and operation of chromatographic separators

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M. Kearney. *Paper presented to 49th Ann. Meeting Sugar Ind. Tech.*, 1990, 12 pp.

The use of a chromatographic separator for sugar recovery from molasses is discussed. Location of a separator at the sugar end generally results in a type of recycle loop, and the equilibrium recycle conditions may be modelled using an infinite series which, because it is convergent, allows the maximum amount of recoverable sugar to be predicted. Examples of operational schemes are presented that are based on a molasses purity of 60. The Twin Falls factory of The Amalgamated Sugar Co. operates two Tascos separators having a daily processing capacity of about 350 tonnes of molasses of 80% dry solids. The annual program is separated into: (1) a beet campaign of 148 days during which all the extract from the separator is sent directly to the high melter and some discarded molasses is stored to prevent a non-sugar accumulation; (2) a thick juice campaign of 26 days which includes operation of the separator, again with storage of some discarded molasses; (3) a 142-day molasses campaign during which the molasses discarded in (2) and (3) is treated in the separator and all the extract is stored, and (4) an extract campaign lasting 15 days during which all the stored extract from (3) is passed through the sugar end which operates entirely on extract with no thick juice. All the molasses is discarded during this period to provide a final blow-down of poorly separated non-sugar components.

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### Foaming of carbonation juice

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T. Szekrényesy, T. Liktor, K. Hangyál and L. Dömötör. *Cukoripar*, 1990, 43, 48 - 53 (Hungarian).

See *I.S.J.*, 1990, 92, 31A.

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### Sugar solubility and molasses exhaustion. I. Simple calculations. The separation function of the sugar house. II. The relationship between parameters of low-grade massecuite cooling crystallization and solubility

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I. L. Megyeri. *Cukoripar*, 1989, 42, 143 - 149. II. *Idem. ibid.*, 1990, 43, 54 - 58 (Hungarian).

I. Mathematical formulae defining sugar solubility are presented as well as some simple worked examples. The significance of the separation function (the ratio of massecuite purity to mother liquor purity), which allows examination of any given boiling scheme in terms of sugar solubility, is discussed. It is stressed that any approach to molasses exhaustion should make best possible use of the theoretical possibilities arising from sugar solubility and the boiling scheme.

II. With the aid of calculations concerning low-grade massecuite cooling, it is shown that it is not necessary to maintain the parameters at strictly optimal levels, although the final temperature of the massecuite and the non-sugars/water ratio should be selected in ranges that are close to optimum so as to achieve a maximum mother liquor viscosity. Even where the massecuite is rapidly reheated before centrifugalling, it is possible to calculate the optimum cooling temperature. A 5°C rise with reheating reduces the mother liquor purity by 2%.

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### Environmental protection in the sugar industry

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E. Horváth-Kelemen. *Cukoripar*, 1990, 43, 74 - 75 (Hungarian).

A brief review is presented of cases of waterway pollution by waste and contaminated oil, agricultural chemicals and other substances such as oil rags at Hungarian sugar factories in 1985/88 (for which fines were imposed). Mention is made of noise levels that exceeded the official limits at two factories.



# Sugar refining

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## Use of dimensionless factors to obtain relations between crystallization variables

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T. Cruz C. and A. Delgado R. *ATAC*, 1988, 47, (6), 31 - 33 (*Spanish*).

Equations were developed for the calculation of refined sugar batch boiling parameters relevant to the feed material, the boiling process and the final massecuite. The intention was to compile tables and nomograms of help in formulating and controlling boiling processes.

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## Techno-economic evaluation of the use of surfactants in raw sugar refining in Cuba

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M. Cordovés and V. Chopik. *ATAC*, 1989, 48, (2), 48 - 53 (*Spanish*).

Use of quaternary amine salts at an average of 120 ppm and of polyacrylamide at 15 ppm in phosphatation at selected refineries reduced the consumption of steam, active carbon and kieselguhr and decreased sugar losses while greatly increasing refined sugar output by comparison with conventional treatment without them. The equivalent monetary savings are calculated.

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## A mathematical model and algorithm for control of the (drying and pressing) section

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V. B. Medzenovskii, A. O. Poltorak, Yu. M. Skakovskii and V. Ya. Muchnik. *Sakhar. Svekla*, 1990, (2), 9 - 12 (*Russian*).

A mathematical model and control algorithm are presented of the distribution of moist sugar to the drying and pressing plant. The aim is to ensure that there is sufficient material at all times in the feed hoppers to ensure normal operation of both dryer and press.

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## An automated line for continuous syrup decolorization

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Ya. O. Kravets, L. P. Zarudnev, S. A. Gurova, L. I. Akimova, V. V. Kim, N. S. Tishchenko, S. I. Povolotskii and E. A. Skorobogatov. *Sakhar. Svekla*, 1990,

(2), 53 - 55 (*Russian*).

The continuous line for decolorization with a moving bed of AGS-4 granular carbon and subsequent filtration has a nominal hourly throughput of 25 m<sup>3</sup> and is based on a working bed height of 8 m (half the effective height of the 3 m diameter counter-current column). Automatic control is applied to: syrup flow; levels in the feed and decolorized syrup, carbon slurry and diluted wash water tanks and in the wash chamber and feed hopper; temperature of the treated syrup; monitoring of pump operation; etc. Operation of the system is described. Results of tests on 1st and 2nd refinery syrups of 69°Bx and initial optical density of 81 and 215 units, respectively, indicated average decolorization efficiencies of 47.5 and 47.0%, respectively, and 83 - 84% reductions in turbidity.

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## VPK-101 polyelectrolyte for refinery syrup purification

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L. G. Vorona, V. V. Mank, T. A. Mikhaïlik, V. F. Chernenko, Yu. F. Tsyukalo, V. I. Shul'ga and T. K. Panchuk. *Sakhar. Svekla*, 1990, (2), 55 - 58 (*Russian*).

VPK-101 is a cationic, aqueous solution of poly-4-vinylbenzyltrimethyl ammonium chloride derived from a polystyrene by chloromethylation and subsequent amination with trimethylamine. As decolorizing agent it was compared in laboratory and preliminary full-scale tests with Soviet OU-A powdered carbon (of limited efficiency and therefore generally replaced by more expensive imported carbons), Norit and Japanese SW-50 carbons. In the laboratory, a maximum 40 - 50% decolorization of 1st refinery syrup was obtained with the polyelectrolyte at 0.3% and with Norit at 0.5% on Brix. At a dosage rate of 57 ppm and with syrup of varying quality, 37 - 48% decolorization was obtained in the full-scale experiments by comparison with 25 - 35% using carbon. The polyelectrolyte was thoroughly mixed within 10 min at a mixer speed of 9 rpm.

The colorant coagulate formed by the polyelectrolyte in the syrup was not dispersed during transfer by centrifugal pump to the filters.

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## Cane sugar refining - where are we going? I. Fundamentals of sugar processing technology

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C. C. Chou. *Paper presented to 49th Ann. Meeting Sugar Ind. Tech.*, 1990, 21 pp.

With a reduction that has already taken place in refined sugar demand in the USA, the author examines the future prospects for cane sugar refining in the face of increasing environmental problems (in which waste disposal is discussed) and a shrinking share of the market. The potential damaging effects of increased beet sugar yields, sugar recovery from beet molasses, and increased consumption of HFS and artificial sweeteners are discussed and the possibility of co-processing cane raw sugar in beet sugar factories considered. Proper training and practice are necessary for maintenance of refining efficiency; affination and decolorization are given as processes that would benefit from this approach. Staff attention to detail is also discussed, with sugar loss such as caused by a fall in pH and by microbial activity cited as example.

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## Quality management systems in Tate & Lyle Sugars. The international standard

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S. J. Gibbons and R. B. Ratcliffe. *Paper presented to 49th Ann. Meeting Sugar Ind. Tech.*, 1990, 20 pp.

Quality control as specified in British Standard 5750, Part 2 (identical with ISO 9002:1987) is explained and the procedure used to assess factory operations and certify that they comply with the standard specifications is described. The measures adopted at the Greenock refinery of Tate & Lyle plc to meet the certification requirements in 1989 are outlined. Advantages of the program include customer, senior management and employee confidence, improved

staff training and elimination of market disadvantage. It is calculated that savings will have exceeded the costs involved in certification within 24 months.

### **BC Sugar - the Vancouver refinery**

K. M. Foo. *Paper presented to 49th Ann. Meeting Sugar Ind. Tech.*, 1990, 9 pp.

Operations at Vancouver refinery (where there are no excess non-sugars for molasses production so that molasses has to be purchased occasionally to supplement the non-sugars in the processed high-grade raw sugars) are briefly described. (See also Chapman & Curdie: *I.S.J.*, 1990, 92, 84 - 87, 92.)

### **Clarification processes**

M. C. Bennett. *Paper presented to 49th Ann. Meeting Sugar Ind. Tech.*, 1990, 12 pp.

The mechanisms and importance of the phosphatation and carbonation processes as means of removing impurities and as pretreatments for decolorization processes are described and their efficiencies and major factors influencing them discussed. While carbonation achieves over 50% decolorization as against 25 - 30% by phosphatation, the use of cationic colour precipitants will give significant improvements. Improved aeration and secondary flocculation by polymers allow the phosphatation-floitation process to handle liquors having a Brix greater than 70° (leading to significant reductions in evaporation costs), while carbonation is restricted to a maximum of 66°Bx. The amount of invert sugar in liquor treated by phosphatation is typically about 0.025% as against 0.010 - 0.015% in carbonated liquor after filtration. However, the filter cake resulting from carbonation is more than 10 times greater than with phosphatation. The capital cost of a new carbonation/filtration plant is about double that of a new phosphatation plant, mainly because of the extra filters, sweetening-off mud presses, gas pumps and scrubbers required, while operating

costs of the two processes under similar operating conditions are almost identical. Improvements over the last 20 years include new methods of polish filtration and mud sweetening-off for phosphatation and the development of automatic pressure filters for carbonation mud.

### **Colonial Talofloc system**

C. A. Rousse. *Paper presented to 49th Ann. Meeting Sugar Ind. Tech.*, 1990, 15 pp.

Details are given of the Talofloc system installed at the Gramercy refinery of Colonial Sugars Inc. and of the Williamson clarification system it replaced (which had become too small to meet requirements and was in a very poor condition after some 50 years operation). Test data from the new system showed that the clarifiers removed more colour than the old ones but less turbidity than expected. However, the new station has substantially increased flow through the char filters (despite an increase in Brix after the char house and a fall in clarification temperature) with a dramatic reduction in the time required for sweetening-off and an increase in the melt rate. The amount of sweet-water generated by the char, sugar losses and fuel consumption have been reduced.

### **Ion exchange purification processes**

J. P. Sheahan. *Paper presented to 49th Ann. Meeting Sugar Ind. Tech.*, 1990, 7 pp.

The literature on liquor decolorization by polystyrene and acrylic resins (preferred because of their stability and resistance to fouling) is reviewed, including the mechanism of the process, acrylic vs. polystyrene resin and the benefits obtained at some refineries with use of a combination of both types, the advantages of resins over carbon and bone char, the costs of resin decolorization and problems such as chalk contamination, effluent disposal and inability to remove ash. The use of deionizing resins for ash removal in liquid sugar

production and to increase the output of a refinery is also mentioned.

### **Design and implementation of conditioning silos and bulk loading facilities at Lantic Sugar - Saint John refinery**

J. N. Lemon and D. A. Ryan. *Paper presented to 49th Ann. Meeting Sugar Ind. Tech.*, 1990, 20 pp.

Details and diagrams are presented of the facilities at Saint John refinery where all 1000 tonnes per day of sugar from the granulators is conveyed directly to a primary conditioning silo of 2000 tonnes capacity which normally remains full to provide a residence time of 48 hr; sugar is continuously extracted from this silo to a secondary or storage silo of 3000 tonnes capacity (although it may be diverted to a screening system) where the residence time varies from a few hr to 72 hr and from which the sugar goes to the screening system and thence to appropriate packaging bins and/or to bins for transfer to bulk railcars. The sugar entering the silos contains 0.035 - 0.40% moisture (oven dried) and has a temperature of 115 - 120°F; it is discharged at a moisture content of 0.025% and a temperature that depends on the ambient temperature and on the silo that is being used to feed the screening system. Computerized control is provided for the silos, conveyors, screening station and associated equipment, with ultrasonic level detection in the silos. Advantages include a dramatic reduction in manpower, a considerable improvement in industrial sugar consistency and quality (including the absence of lumps), an increase in bulk density, and a substantial increase in productivity as a result of the independence of melt rate from packaging, storage and screening operation. Disadvantages include a marked increase in dust generation, a high failure rate of the bearings on the rotary valves of the silo discharge system (although this has not hampered production) and the inability to transfer sugar from the granulators directly to the secondary silo.

# Laboratory studies

## Measurement of process parameters applicable in sugar factory automation and computerization

J. Dobrzycki, M. Ludwicki and S. Wawro. *Zesz. Nauk. Technol. Chem. Spoz.*, 1989, **42**, 21 - 34; through *Ref. Zhurn. AN SSSR (Khim.)*, 1990, (5), Abs. 5 R1480.

A short account is given of a number of rapid methods applicable to process automation which have been developed in Poland for determination of: the sugar content in pulp based on the density of press water; reducing sugars in various products; the sugar content in condensate; etc.

## Method for determining visible impurities in sugar

L. P. Ferrara, I. Shirose and F. Matano. *Colet. Inst. Tecnol. Alim.*, 1987, **17**, (2), 126 - 131; through *Ref. Zhurn. AN SSSR (Khim.)*, 1990, (5), Abs. 5 R1481.

A method was developed for determining impurities in sugar which was based on the principle of counting them after they had been spread on a specially designed tray. Samples of various types of sugar were investigated.

## Comparison of techniques for pol determination in cane juices

R. J. Rodríguez, F. Pérez and R. Torres. *CubaAzúcar*, 1988, (Oct/Dec.), 44 - 49 (*Spanish*).

Aluminium hydroxide was compared with basic lead subacetate as clarifying agent in the polarimetry of primary, mixed and clarified juice. Statistical analysis of the results showed no significant differences between the results for the two agents nor between the use of dry and wet lead. The Al hydroxide prepared *in situ* from anhydrous Al chloride and NaOH had the advantage over the lead acetate of being much less costly, causing no turbidity, being non-toxic, being unaffected by CO<sub>2</sub>, giving good filtrability and allowing the juices to be returned to process, although it had the disadvantage of

requiring more time for analysis than with dry lead.

## Study on the effect of basicity of basic lead acetate solutions on pol determination in raw sugars

J. Rodríguez L., F. H. Pérez S and Z. Hernández B. *Centro Azúc.*, 1989, **16**, (1), 9 - 17 (*Spanish*).

A review of the literature on raw sugar polarimetry using official ICUMSA methods showed that measurements were affected by the basicity of the lead acetate, particularly at lower levels of pol. However, statistical analysis of pol measurements of refined sugar, plantation white sugar and raw sugar samples using acetate solutions of 35, 40 and 45% basicity showed no significant differences between the effects of the three basicities when ICUMSA No.1 method was employed for the 41 samples.

## A new approach to measurement of colour in white sugar manufacture

M. B. Londhe, S. Y. Jadhav and A. A. Zende. *Indian Sugar*, 1989, **39**, 675 - 681.

Measurement of colour by ICUMSA Method 4 at 420 nm was found to be unsuitable on its own for boiling house products. Instead, the authors advocate (colour at 420 nm × solids % by weight) as a better criterion of colour removal. The results are discussed with the aid of tabulated data.

## Constant-potential amperometric detection of carbohydrates at a copper-based chemically modified electrode

S. V. Prabhu and R. P. Baldwin. *Anal. Chem.*, 1989, **61**, 852 - 856; through *Anal. Abs.*, 1990, **52**, Abs. 4J112.

A chemically modified electrode was prepared by coating a vitreous-carbon electrode with a Cu(II) layer, which decreased the overpotential for carbohydrate oxidation compared with that at unmodified carbon electrodes. The

electrode was used for constant-potential amperometric detection of reducing and non-reducing sugars separated by HPLC on a column (15 cm × 4 mm) of Dionex HPIC-AS6. The Cu layer catalysed the sugar oxidation in basic solution when a potential sufficiently positive to generate Cu(III) was applied. In flow-injection analysis and HPLC, electrode response was stable for >5 hr (signal loss <10%). With anion exchange chromatography in 0.15M NaOH, mono- and di-saccharides were separated and detected in sub-ng amounts at an applied potential of +0.48 V vs. Ag-AgCl.

## Additives to improve filtrability and reduce turbidity in polarimetric analysis. I. Raw sugar

F. H. Pérez S., Z. Hernández B. and M. J. Castro F. *CubaAzúcar*, 1989, (Jan/March), 11 - 17 (*Spanish*).

Since the use of basic lead subacetate in raw sugar polarimetry has failed to solve problems (aggravated by mechanical harvesting) associated with filtration velocity, turbidity and colour, a number of additives were tested. Out of six initially selected, sodium chloride, potassium sulphate and potassium ferrocyanide gave improvements and were subjected to further assessment. The best results (a dramatic increase in filtration velocity and an appreciable reduction in turbidity with no significant effect on pol) were obtained by adding potassium ferrocyanide at 0.05 g/26 g raw sugar together with the standard quantity of lead acetate.

## Fifty years of sugar research

M. A. Clarke. *Paper presented to 49th Ann. Meeting Sugar Ind. Tech.*, 1990, 11 pp.

A survey is presented of the work conducted by the Bone Char Research Project (inaugurated in 1939), Cane Sugar Refining Research Project Inc. (formed in 1963 when the BCRP was concluded) and Sugar Processing Research Inc. into which it was reorganized in 1981.

# By-products

## Investigations on oxidative cleavage of reducing disaccharides

H. Röger, H. Puke and M. Kunz. *Zuckerind.*, 1990, 115, 174 - 181 (German).

Investigations of oxidative cleavage of reducing sugars (particularly palatinose obtainable from sucrose by biochemical transglucosidation) to yield polyhydroxymonocarboxylic acids are reported.

## Production of microbial flocculants

S. L. Huang, J. S. Wang, Y. T. Chuang and L. H. Wang. *Taiwan Sugar*, 1989, 36, (6), 16 (Abstract only).

High polymer flocculants are widely used for waste water treatment and cane juice clarification. A promising strain of flocculant-producing microbe, S-4K', has been isolated from soil. Activated sludge containing the microbe gave a considerable improvement in the sedimentation of the fine solids from waste water at Pingtung pulp factory. Large quantities of the flocculant can be produced by culturing the micro-organism on many kinds of sugar as substrate, with K nitrate, diammonium phosphate and peptone as suitable N sources (but not urea). The flocculant-producing activity of the microbe may be improved by adding Ca<sup>++</sup>, Mg<sup>++</sup>, K<sup>+</sup> and Na<sup>+</sup>, but Cu<sup>++</sup> and Fe<sup>+++</sup> have a considerable inhibitory effect. Optimum pH was 7 - 8, and maximum cell growth under laboratory conditions was obtained within 32 hr.

## Production of liquid sugar

W. F. Lin and T. P. Hsieh. *Taiwan Sugar*, 1989, 36, (6), 18 (Abstract only).

The relationship between reaction temperature, flow rate and colour index was studied in the laboratory when a strong cation exchange resin in H<sup>+</sup> form and a weak anion exchange resin in OH<sup>-</sup> form were used in series to invert syrup of 55°Bx having a colour content of 90 - 103 RBU at a flow rate of 2.3 and 3.2 bed-volumes/hr during a cycle of 48 BV at 40°, 45° and 50°C and regeneration of

the cation exchanger with 5% HCl and the anion exchange resin with 4% NaOH, both at 0.6 BV/hr. Results showed that ash removal was independent of the reaction temperature and flow rate, that the inversion rate was proportional to reaction temperature at constant flow rate, that temperature and decolorization efficiency were in inverse proportion and that the relationship between reaction temperature, inversion rate and flow rate could be expressed by the formula:  $-\ln(1 - X_s) = 0.69 \times 1.1 (T - 40)/V$ .

## Manufacture of photocopying paper from bagasse pulp

W. F. Yee and L. H. Wang. *Taiwan Sugar*, 1989, 36, (6), 18 (Abstract only).

A practical process for producing photocopying paper of high quality from bagasse pulp has been developed. Chemical pulp having a brightness greater than 83% and a freeness (CSF) of 290 ml is used as fibre stock with which filler is mixed at 8% on product dry weight. A paper having a fine texture, higher brightness (87%) and opacity (87%) is thus obtained. Good control of the surface resistivity (10<sup>6</sup> mohm) and the smoothness (30 sec) of the paper ensure a good photocopying effect. When rosin soap, wax emulsion and alum are used as sizing agent, a product of 11 sec waterproofing and excellent writing quality is obtainable, but its tearing strength (not an important requirement for photocopying paper) is lower than that from wood pulp, but the addition of NBKP can make up the difference.

## Image processing for alcohol seed culture inspection

C. I. Wang. *Taiwan Sugar*, 1989, 36, (6), 20 - 21.

Successful alcohol fermentation depends largely on the preparation of the inoculum which must contain a certain level of total viable yeast cells. Usually, the inoculum is examined by microscope, but recently a computerized image processing system has been connected to

the microscope for total cell counts to be video-taped through the camera. Details are given of the procedure used and of the hardware employed. The system provides better seed inspection and culturing while also making available objective records of seed culture for examination after fermentation. However, the seed inspector must also be a skilled microcomputer operator, software is difficult to develop and the seed culture capacity must be much greater than the quantity of seed required by the fermenters. Nevertheless, it is more suitable for continuous fermentation and is to be used in automation of seed selection.

## Molasses fermenter scale-up

N. P. Shukla. *Taiwan Sugar*, 1989, 36, (6), 23 - 24.

The procedure used in scaling-up a molasses fermenter from pilot-plant data is described. In order for the volumetric mass transfer coefficient to be unchanged, it is necessary to predetermine the oxygen diffusion coefficient, surface tension and rheological properties. The impeller speed in most fermenters is of the same order, regardless of size.

## Critical examination of anhydrous ethanol as fuel in gasolines

F. Caballero P. *Rev. Asoc. Técn. Azuc. México*, 1989, 3, (3), 11, 13, 15 - 17 (Spanish).

The advantage of a 4:1 gasoline:ethanol mixture as motor fuel in reducing lead pollution of the atmosphere is discussed and the potential for alcohol manufacture from molasses or cane in Mexico examined. If molasses were fermented to alcohol rather than exported, 33.5 million litres could be produced in a distillery of 100,000 litres daily output, but this would provide only enough fuel for 13,500 minibuses; for the same volume of alcohol produced directly from cane, a 4500 tcd mill integrated with a distillery (also producing 100,000 litres daily) plus a cane area of 10,500 ha would be required. However, even

using both raw materials and possibly reducing the alcohol component in fuel to 10% would still cover only 11% of demand in the Valle de México; 11,680 million hectolitres would be needed to meet all the demands. The problem of carbon monoxide pollution and how to solve it using catalytic converters is also discussed.

#### Use of sugar industry by-products as calf feed at Cordoba, Ver.

R. Reta G., A. Aguilar Y. and J. C. Ojeda T. *Rev. Asoc. Téc. Azuc. México*, 1989, 3, (3), 17, 20, 22, 25, 28 (Spanish).

Trials are reported in which fattening calves fed on rations containing molasses, vinasse and bagasse pith for up to 258 days gained an average 60% in carcass weight at a feed conversion ratio of 17:1. In one trial, the inclusion of dried vinasse at 10% of total feed dry matter gave a daily weight gain of 1.4 kg compared with 1.3 kg when it was excluded; the other ingredients included 26% molasses and 19% bagasse pith. The economics are discussed.

#### Recent developments in ethanol production technology

D. R. McGaw, W. A. Mellowes and A. C. Pilgrim. *Sugar J.*, 1990, 52, (9), 16 - 20.

The potential for fuel alcohol manufacture by fermentation in the Caribbean is discussed. While cane and molasses are the most suitable materials, there are major drawbacks: (1) competition with sugar manufacture for cane supply, (2) high cost of cane and hence of the alcohol and (3) the fact that more than half of the molasses produced is used for rum manufacture. Other cane waste products considered are tops and trash (the amount of which in Trinidad is such that it could yield  $3.4 \times 10^7$  litres per year of 180 days) while bagasse could yield  $3.4 \times 10^6$  annually. Cellulose- and starch-based materials are also examined. The literature on aspects of the fermentation and distillation processes is

briefly reviewed and future prospects discussed.

#### Performance of sows fed different amounts of final molasses

C. P. Díaz and J. Díaz. *Cuban J. Agric. Sci.*, 1989, 23, 283 - 288.

Trials in which 130 sows were fed daily on final molasses at 1.1 - 2.1 kg dry matter per animal plus a protein supplement showed no significant differences in any of the reproductive traits studied nor any net gains at conception and during the first 60 days, from which it is concluded that molasses should not be used to increase the energy supply with the aim of obtaining greater weight gains during pregnancy.

#### Feed intake pattern in piglets fed sugar cane meal diets

N. Rodríguez, R. Bocourt, E. Lamazares and R. Larduet. *Cuban J. Agric. Sci.*, 1989, 23, 311 - 316.

Rations containing 0, 20 and 40% (dry basis) ground dehydrated cane stalk containing about 50% soluble carbohydrate and 42% neutral fibre were fed to piglets in the 5th week after weaning. Results showed that while the consumption pattern was not affected by 20% cane meal (by comparison with a cereal control), consumption, rate and frequency of ingestion and body weight fell significantly with 40% meal. A number of possible causes are mentioned, including the potential effect of the soluble carbohydrate content on palatability.

#### Dissolving of sugar

K. E. Austmeyer. *Zuckerind.*, 1990, 115, 250 - 260 (German).

While the drinks and confectionery industries use sugar as an aqueous solution, there are inadequate guidelines on the design of vessels and optimum procedure for dissolving sugar. The theory of the mass transfer process is examined and represented by dimensionless expressions. Stirrer systems and their application are described. In

experimental batch operation, a 3-bladed propeller having an angle of incidence adjustable in the range 25 - 45° and rotating at 700 - 1850 rpm dissolved sugar in the shortest time and used least power (3.9 kW/m<sup>3</sup>), proving superior to a 3-bladed propeller of fixed angle, a slanted 4-bladed and a splitter-type stirrer. An energy balance for the process covers both the heat of dissolution and the stirrer. A continuous column based on the investigations is described which consists of four stirred compartments, one above the other; where there is insufficient space, an alternative comprises a pair of interlinked two-compartment columns. Appropriate shape factors were derived which allowed for deviation of crystals from the equilibrium shape as a result of rounding of the corners and edges during dissolution.

#### Biotechnology - a strategy for the sugar world

Anon. *GEPLACEA Bull.*, 1990, 7, (4), 2 pp.

The potential in Latin America for diversification into the fermentation field is discussed with mention of ethanol, L-lysine, monosodium glutamate, citric acid, yeast, acetic acid, acetone-butanol, xanthan gum and dextran.

#### Preliminary study on corrosion of a system for irrigation with sugar industry waste

S. Gil F. and W. Francisco M. *Centro Azúc.*, 1989, 16, (2), 18 - 23 (Spanish).

A system for spraying canefields with a mixture of cane sugar factory effluent and waste from a yeast plant was found to be corroded; carbon steel components were mainly affected, although aluminium tubes and the main pumps also showed signs of deterioration and pitting.

#### Kinetics of degradation of stored bagasse

A. García R. and I. Vega R. *Centro Azúc.*, 1989, 16, (2), 41 - 46 (Spanish).

Investigation of wet storage of bagasse (for subsequent pulping) in wooden boxes in which it was subjected to a pressure of 70 - 80 kg/m<sup>3</sup> showed that degradation was more marked during the first three weeks and was greater when sprayed with a solution containing up to 2.5% sugar than with water. Partial depitching had no significant effect on degradation. The rate of degradation was similar to that found in autocatalytic reactions under the effect of biological process kinetics. A fall in microbial population (possibly a result of depletion of a substrate needed for development of micro-organisms and of the inhibiting effect of a metabolic product) accompanied a reduction in degradation from the 4th week.

#### **Study of mechanical and optical properties of newsprint produced from different types of raw material**

C. Moreda and A. Abril. *CubaAzúcar*, 1989, (Jan./March), 25 - 33 (*Spanish*).

The mechanical and optical properties of newsprint manufacture from various materials, particularly wood and bagasse pulp, are compared on the basis of data from Cuba and other countries. It is concluded that newsprint production from bagasse is a practical means of replacing pulp imports in Third World countries where there is a sugar industry and a ready supply of the raw material.

#### **Separation of glucose and fructose from invert sugar using calcium oxide**

A. Fariñas B. and E. Duarte P. *ATAC*, 1989, 48, (4), 19 - 22 (*Spanish*).

CaO was added to 200 ml of 25°Bx invert sugar (>90% inversion) at a molar ratio to fructose of 2:1 and the mixture stirred continuously at 0 - 5°C. A maximum fructose recovery of 91% was achieved after 90 min, and purity was approx. 90; after the same time, 75% glucose was recovered at a purity of 71, while maximum glucose recovery was 78% after 120 min at about 72 purity.

#### **Isolation of lignin in black liquors from chemical pulping of bagasse**

R. Cruz, H. Domínguez and A. Grau. *CubaAzúcar*, 1989, (Apr./June), 1989, 53 - 58 (*Spanish*).

A study of lignin isolation demonstrated the effects of initial black liquor concentration, of the final pH of the precipitate and of filtrate recycling on filtrability. A process is proposed for precipitation of the alkaline lignin in which the granulometry of the precipitate is such as to facilitate filtration and the lignin itself acts as coagulant.

#### **Modification to the system of preparing nutrient salts in a torula yeast plant**

L. A. López C. *ATAC*, 1989, 48, (5), 16 - 22 (*Spanish*).

Urea, phosphate and sulphate solutions used as nutrients in yeast fermentation are corrosive, particularly sulphate. Instead of three individual solutions, two mixtures were prepared containing optimum proportions of the nutrients, namely ammonium biphosphate: ammonium sulphate and ammonium biphosphate:urea. The biphosphate was found to act as corrosion inhibitor and the modified system was an economic improvement.

#### **Increasing the biological activity of yeasts**

L. P. Pashchenko and Yu. S. Serbulov. *Pishch. Prom.*, 1989, (12), 35 - 36; through *Ref. Zhurn. AN SSSR (Khim.)*, 1990, (8), Abs. 8 R1404.

In order to reduce the costs of yeast activation, it was recommended to use a feed consisting of an aqueous solution of beet molasses and meat broth enriched with animal protein. The biological characteristics of the pressed yeasts were thus improved without the use of seed yeast. The raising force improved from 12 - 14 to 5 - 6 min. The change in fermenting capacity of zymase and maltase complexes of yeasts from the start of their activation was determined

gasometrically by fermenting 10% solutions of maltose, sucrose, glucose and fructose. There was a 2 - 2.2-fold increase in maltase activity. The fermentation times for sucrose, glucose and fructose were cut by 30 - 35%, 50 - 55% and 40 - 50%, respectively.

#### **Use of carbonation mud as a new inorganic filler in plastics and rubber**

A. Smelík, G. Halásová and S. Füzü. *Listy Cukr.*, 1990, 106, 79 - 82 (*Czech*).

A filler for plastics and rubber was produced by carbonizing carbonation mud at 650 ± 50°C to give a greyish-black powder containing a minimum of Cu, Mn and Fe. The particle size of the product determines its use: the finest fraction (2 - 3 µm) is of application for hard PVC, polyethylenes require the 3 - 20 µm fraction while the coarsest fraction up to 60 µm is suitable for use as a rubber filler.

#### **The ethanol industry today and its future impact on the sugar industry**

W. Maloney. *J.A.S.T.J.*, 1988, 49, 15 - 17.

The economics of anhydrous ethanol production from molasses in the Caribbean (particularly Jamaica) and its marketing, especially in the USA, are discussed and future prospects considered. It is thought that ethanol manufacture should be developed in Jamaica as a means of increasing revenue within the sugar industry.

#### **Molasses traps in grassland pest control**

Anon. *Tech. Rpt. Inst. Cienc. Animal*, 1987/88, 8.

Molasses traps 30 cm in diameter and placed at a height of 1.5 - 2.0 m 500 - 1000 m apart proved effective as a means of detecting the appearance of *Mocis* sp. Use of a bait containing molasses, insecticide and water reduced the adult insect population by 80%.

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research purposes, since in research tests the entire mass undergoing testing is frequently very limited.

3. Flexibility in procedure. The HST method, similar to the original and all the modified Clerget methods, requires that analytical procedures be followed strictly<sup>5</sup>. We found, however, that the glucose analyser method can tolerate much more flexibility. Temperature variations within a few degrees Celsius or inversion times from 30 minutes to 24 hours do not have any significant effect on the analysis. The only steps requiring more exact techniques are sample dilution, which can be automated, and operation of the glucose analyser.

The enzymatic method described in this report has thus far been demonstrated with molasses only. However, since molasses is the factory product which is lowest in purity, we expect the method to be applicable to other factory products as well. For other factory products, the only modification needed in the analytical procedure would be to apply different dilution factors.

Kestoses, as mentioned earlier, could be the only constituents to interfere in the glucose analyser method. In view of the limited levels of kestoses in cane products (cf. Table I), it is apparent that, for general research purposes, the presence of kestoses will not cause significant errors in sucrose determined by the glucose analyser method. For routine analysis purposes, the effect of kestoses needs to be better defined. We are planning to conduct research in this direction and, currently, since kestoses are not commercially available, we are in the process of preparing them in the laboratory.

#### Conclusions

The study thus far has shown that, for sucrose analysis, the glucose analyser method is comparable with the HST method in precision, is significantly better in accuracy and time efficiency, and appears to be less costly to use. It is apparently promising for research purposes and for routine analysis use.

The effect of kestoses, however, requires further study.

#### Acknowledgements

The authors wish to extend their deep gratitude to Dr. Toshio Moritsugu for numerous helpful discussions during the course of this study. Also, the authors wish to thank Mr. T. S. Chao for conducting part of the experimental work and to thank Dr. R. Hammer of the Department of Anatomy, University of Hawaii, for allowing the authors to use the Beckman Glucose Analyzer in his laboratory before HSPA acquired its own.

#### Summary

An enzymatic method for the determination of sucrose in molasses was tested. With this method, sucrose is determined from the amount of glucose released during inversion induced by invertase and glucose is determined with a glucose analyser. When compared with the modified Clerget method of sucrose analysis used in the Hawaiian sugar industry, it was found that, although the precision of the two methods appears comparable, the accuracy of the enzymatic method is significantly better. Also, the latter method was found to be significantly less tedious and time-consuming, to cost less, and to require only an extremely small amount of sample for each analysis. The method is thus considered reliable and convenient for research purposes. As for routine analysis, it also shows promise, but further investigation is recommended to determine the feasibility of adopting it as a supplement to, or a replacement of, the current industry method.

#### Determinación de sacarosa usando un analizador de glucosa

Se probó un método enzimático para la determinación de sacarosa en melazas. Con este método, la sacarosa se determinó a partir de la cantidad de glucosa liberada durante la inversión inducida por la invertasa y la glucosa se determinó con el analizador de glucosa. Cuando se comparó con el método

modificado de Clerget para el análisis de sacarosa usado en la industria azucarera de Hawaii, se encontró que, aunque la precisión de los dos métodos era parecida, la precisión del método enzimático era significativamente mejor. Al mismo tiempo, el método último se encontró que era significativamente menos tedioso y que consumía menos tiempo, cuesta menos y que requería solo una cantidad extremadamente pequeña de muestra para cada análisis. De esta manera el método es considerado digno de confianza y conveniente para propósitos de investigación. Para el análisis rutinario, también promete servir, pero se recomienda una mayor investigación para determinar la posibilidad de adoptarlo como un suplemento a, o como un reemplazo del método industrial corriente.

#### Détermination du saccharose à l'aide d'un analyseur de glucose

On a essayé une méthode enzymatique pour la détermination du saccharose dans les mélasses. Dans cette méthode le saccharose est mesuré aux dépens de la quantité de glucose mise en liberté au cours de l'inversion qui est induite par de l'invertase. Le glucose est déterminé avec un analyseur de glucose. Lorsqu'on compare cette méthode à la méthode modifiée Clerget qui, dans l'industrie sucrière hawaïenne, est utilisée pour la mesure du saccharose, on observe que l'exactitude de la méthode enzymatique est significativement meilleure, bien que pour les deux méthodes la précision soit comparable. La méthode enzymatique est aussi moins pénible, elle demande moins de temps, son coût est moindre et elle n'exige qu'une quantité fort réduite d'échantillon pour chaque analyse. On estime donc que la méthode est fiable et qu'elle convient pour des besoins de la recherche. Elle semble aussi prometteuse quant à son emploi pour le contrôle de routine. On recommande cependant de l'étudier davantage et de déterminer si ne peut pas l'adopter comme un supplément ou en remplacement de la méthode industrielle actuelle.

### Saccharosebestimmung mittels eines Glucose-Analysators

Geprüft wurde eine enzymatische Methode zur Bestimmung von Saccharose in Melassen. Mit dieser Methode wird die Saccharose aus der Menge von Glucose ermittelt, die während der durch Invertase induzierten Inversion freigemacht wird; die letztere wird mit einem Glucose-Analysator bestimmt. Vergleich mit einer zur Saccharose-Analyse in der hawaiischen Zuckerindustrie angewendeten Modifikation der Clerget-Methode zeigte, dass, obgleich die beiden Methoden anscheinend die gleiche Präzision hatten, war die Genauigkeit der enzymatischen Methode viel besser. Darüber hinaus, erwies sich die letztere Methode als viel weniger beschwerlich und zeitaufwendig, sie kostet weniger und benötigt nur geringe Menge Probe zur Analyse. Deshalb wird die Methode als zuverlässig und zweckmässig betrachtet. Sie ist auch hinsichtlich Routine-Analyse vielversprechend, dennoch es empfiehlt sich,

weitere Erforschung durchzuführen, um die Möglichkeit ihrer Anwendung zusätzlich zu oder anstelle von der gegenwärtigen industriellen Methode festzustellen.

#### Appendix I. Procedure for analysis of sucrose in molasses with a glucose analyser

**Apparatus:** Glucose Analyzer (Beckman Instruments Inc.), analytical balance, volumetric flasks, and appropriate (preferable capped) containers of approximately 5 - 10 ml volume.

#### Reagents

1. Glucosidase reagent (Beckman Instruments Inc.).
2. Buffer (15 g anhydrous  $\text{Na}_2\text{HPO}_4$ , 40 g anhydrous  $\text{NaH}_2\text{PO}_4$ , and 5 g sodium benzoate dissolved in distilled water to form 1 litre solution).
3. Invertase (0.2 g Sigma Chemical Company 14504 dissolved in buffer to form 1 ml).
4. Glucose standards (moisture-free glucose dissolved in buffer to form

100, 150, 200, 250, and 300 mg/dl solution).

#### Analysis

1. In two volumetric flasks of the same size, weigh two appropriate amounts of molasses, one being roughly 4 times the other. Mark the flasks No. 1 (less molasses) and No. 2 (more molasses). (For final molasses, if 100 ml volumetric flasks are used, approximately 1 g and 4 molasses should be weighed into flasks No. 1 and No. 2, respectively).
2. Make up flasks in step 1 to volume by adding buffer while mixing.
3. Transfer 3 - 5 ml of the contents of flask No. 1 to an appropriate container, add 0.005 ml invertase, and then set sample aside at room temperature for not less than 30 minutes.
4. Using the glucose analyser, measure the glucose contents in flask No. 2 and in the sample in step 3.
5. Calculate sucrose content in original molasses.

## Aconitic acid removal during cane juice clarification

*continued from page 220*

whereby aconitic acid is recovered from the anion exchanger regeneration eluent. The process of phosphatation by means of phosphoric acid and lime at pH between 5 and 5.5 appeared to be the best technique to use because it removes little aconitic acid (18.7%) while ensuring good clarification; in addition, it allows easy pH regulation. The sulphitation process removes less aconitic acid (16.3%) than phosphatation but does not ensure good clarification. The removal level of aconitic acid was 20.2% for phosphatation by means of calcium superphosphate, 73.8% for defecation and 78.8% for the carbonatation process. In the phosphatation process, the elimination of aconitic acid increases both with pH and the quantity of phosphoric acid used.

### Remoción de ácido aconítico durante la clarificación del jugo de caña

La variación del contenido en ácido aconítico ha sido estudiada de acuerdo con diferentes procedimientos de clarificación del jugo de caña usados en la obtención de azúcar empleando resina de intercambio iónico mediante la cual se recupera el ácido aconítico a partir del eluyente de regeneración del intercambiador aniónico. El proceso de fosfatación, el cual usa ácido fosfórico y cal a un pH entre 5 y 5.5 pareció ser la técnica mejor a usar debido a que remueve poco ácido aconítico (18.7%) al mismo tiempo que asegura una clarificación buena; además, permite una regulación fácil del pH. El proceso de sulfitación remueve menos ácido aconítico (16.3%) que la fosfatación pero no asegura una buena clarificación. El nivel de remoción del ácido aconítico fue de 20.2% en la fosfatación con el uso de superfosfato de calcio, 73.8% en la defecación y 78.8% en el proceso de carbonatación. En el proceso de

fosfatación, la eliminación del ácido aconítico aumenta con el pH y con la cantidad de ácido fosfórico usado.

### L'enlèvement de l'acide aconitique au cours de l'épuration du jus de canne

On a étudié la variation dans les teneurs en acide aconitique pour différents procédés d'épuration du jus de canne qui sont utilisés pour la production du sucre. A cet effet on employait des résines échangeuses d'ions qui permettent de récupérer l'acide aconitique dans l'éluat de régénération des échangeurs anioniques. On a observé que le procédé de phosphatation à l'aide d'acide phosphorique et de chaux à une valeur de pH entre 5 et 5.5 était la meilleure technique utilisable. Ce procédé enlève en effet peu d'acide aconitique (18.7%) et il assure une bonne épuration. En plus il permet un

*continued on page 238*



## Message from the President

Those who attended the 20th Session at Colorado Springs, USA, in June 1990 would surely agree with me that the United States National Committee hosted a very successful conference. The business sessions were productive and the decisions taken provide the Commission with guidance for the next Session and beyond. These decisions are embodied in the recommendations to Referees' Reports which are published below. Other decisions came from three Executive Committee meetings. Some of these which will be of interest to members who did not attend are discussed below.

*Publications Department:* Mr. John Dutton has been appointed Editor/Manager of the Publications Department and will be paid by the Commission to carry out much of the work whose cost was previously borne by British Sugar. John's first major task will be the production of the Proceedings which are expected to be available early in 1991. After that he will undertake the editing and publication of analytical methods in the newly approved ISO format. It is expected that a complete set of methods will be available towards the end of the current Session. Additionally, John will strengthen contacts with fraternal organizations like ISO, Codex Alimentarius, AOAC and OIML.

*Steering Committee:* The Executive Committee urged the President to maintain a Steering Committee to assist in the achievement of the 21st Session goals. This committee will comprise Dr. Albert Emmerich and the President as members of the previous committee, and Drs. Roger Wood and Brian Purchase as new members. We are grateful for the contributions of the two retiring members of the previous committee, Dr. Margaret Clarke and Mr. John Dutton. The four tasks requiring the immediate attention of the committee are:

- \* Rearrangement of some Subjects prior to the appointment of Referees,
- \* Consideration of priorities between Subject Recommendations,

- \* Management of the new methods book, and
- \* Continue implementation of the sugar scale.

*Collaborative Testing:* Probably the most important measure adopted was the acceptance of IUPAC guidelines for the conduct of collaborative tests on analytical methods. With the goal of having a new methods book completed this Session, we must attempt to test all those methods not yet meeting full international standards. This will be a significant undertaking requiring the dedication of Referees and their Associates who will carry out the work and the support of companies and research organizations who pay for the time and facilities needed for the work.

*Membership Goals:* During the 20th Session we admitted Portugal and Indonesia as new members. Mr. Luis Bento and Dr. H. M. Mochtar, the chairmen of their respective National Committees, are well known throughout the sugar world. We look forward to the participation of associates in those countries in the current work program.

The Executive Committee expressed concern at the number of countries who today are both inactive and in arrears with their dues. It was decided

that such countries would have one year to communicate with the Commission and show that a functioning National Committee existed and that a plan for meeting financial obligations would be implemented. The President will shortly be writing to the countries concerned but if those involved read this, it would be appreciated if dialogue was initiated by those committees.

*ICUMSA News:* A survey was conducted at Colorado Springs to determine if the format of ICUMSA News was satisfactory to members. The results will be used to give readers more of what they want. Any readers wishing to contribute articles, letters or comments should do so by contacting either the Editor, Dr. Robert Pieck, or the President.

**Murray Player**  
September 1990

### *Remark of the editor.*

In the January 1990 issue of ICUMSA News<sup>1</sup> we included an article on "Rapid microbiological tests" but inadvertently omitted the author's name, which was Dr. Ruth Strauss (Südzucker AG, Mannheim/Ochsenfurt, Germany). We apologise to Dr. Strauss for the omission.

## RECOMMENDATIONS

The following recommendations were adopted during the 20th Session of ICUMSA held in Colorado Springs from 4th to 8th June 1990. They will be reproduced, together with the Referees' Reports, discussions etc., in the bound volume of the 20th Session Proceedings, which will be available early in 1991 from ICUMSA Publications Department, c/o British Sugar plc., Technical Centre, Colney, Norwich, England NR4 7UB.

### General Subject 1 : Raw sugar

*Referee :* R. J. McCowage (Australia)

1. Any approaches which offer the potential to determine the sucrose content of raw sugar with similar

efficacy and precision to the polarization should be studied.

2. Studies on the effect of different clarification agents on raw sugar polarization should continue, with the ultimate objective of specifying an alternative clarifying agent to basic lead acetate. Alternatively, a method for determining raw sugar polarization without clarification should be sought.

3. The Official status of the ICUMSA gravimetric method for determining the sulphated ash of raw sugar, with incinerations at 550°C and 650°C, is confirmed.

4. Work should be initiated to determine suitable conditions for the per-

<sup>1</sup> I.S.J., 1990, 92, 30.

formance of the ICUMSA gravimetric ash method on raw sugar using one incineration only.

5. The Official status of the ICUMSA conductivity ash method, without addition of sucrose, is confirmed for raw sugar.

6. Further work should be carried out to select one classical method for the determination of the reducing sugars content of raw sugar, or at least one method for raw cane sugar and one for raw beet sugar.

7. Instrumental methods for the determination of reducing sugar in raw sugar should be further investigated, with the aim of recommending suitable methods for consideration as Official methods.

8. ICUMSA Method 2 for raw sugar colour is withdrawn.

9. The Official status of ICUMSA method 4 for raw sugar colour is confirmed. It should be re-named "The ICUMSA method for raw sugar colour".

10. Work should continue in an attempt to find a suitable buffer for pH adjustment in the ICUMSA method for raw sugar colour.

11. The filtration step in the ICUMSA method for raw sugar colour should be further studied, with the aim of specifying conditions under which faster filtration rates can be obtained.

12. The Official status of the ICUMSA method of determining the pH of raw sugar is confirmed.

13. Further work should be undertaken to specify a procedure to prepare raw sugar for grist analysis.

14. Work should continue in attempt to find a specific test for dextran in raw sugar. Approaches based on enzymatic or immuno-assay techniques should be pursued. The test should reflect the refinery performance of raw sugar.

15. Work should be initiated in attempt to define a standardized affination procedure for raw sugar. The procedure should reflect refinery performance and should be capable widespread application in factory laboratories.

16. Specifications should be developed for temperature measurement of raw sugar.

17. Work should be reinstated in an attempt to specify a test for the determination of starch in raw sugar.

### General Subject 2 : White sugar

*Referee: C. W. Harvey (UK)*

1. The assessment of visual appearance of white sugar using Braunschweig colour type series is confirmed as the Official ICUMSA method, with sufficient experimental data to validate the procedure to IUPAC standards. Work should continue within Subject 7 to develop an instrumental technique to measure visual appearance of white sugar.

2. The method described in Appendix 1 of the Referee's Report for the measurement of white sugar solution colour using TEA buffer for pH adjustment is Officially adopted and replaces the existing procedure adopted in the 17th Session (Subject 22, Appendix 1).

3. The Braunschweig method for polarization of white sugar without clarification and with correction of the volume by weighing is Officially adopted. This method is validated to IUPAC standards by collaborative tests.

4. For white sugar needing clarification, the "Raw Sugar Polarization" method description given in Subject 11 up to the 19th Session is recommended as Official. Procedures for determining polarization of white sugar using clarifying agents other than lead should be sought.

5. The conductivity ash method at 28 g/100g is confirmed as the Official method for determining the ash content of white sugar (See Referee's Report for Subject 16).

6. The method for the determination of particle size should contain all the instructions necessary to produce basic screening data, based on sieves conforming to National Standards, including a specification of the type of sieve-shaking equipment to be used. The evaluation of the data by the methods of Power, Rens and RRS should be included with their limitations clearly stated. The selection of the method for evalu-

ation does not require a Recommendation from ICUMSA but should remain at the option of the user.

7. The methods of Knight & Allen for low levels of reducing sugars in white sugar and the Berlin Institute or the Ofner method, or a method based on a combination of these methods, for higher levels should be collaboratively tested to IUPAC standards. The choice of the latter method is to be made on the recommendation of the Referee for Subject 15, Reducing Sugars.

8. Methods for the determination of arsenic, copper and lead should be collaboratively tested, following proposals for up-dating the methods from Referee for Subject 6.

9. Original data from collaborative tests on methods for loss on drying and insoluble matter should be assessed by the Referee for Subject 3. Those methods with sufficient data to meet IUPAC standards should be confirmed as Official ICUMSA methods. Methods that do not reach IUPAC standards are to be included in the collaborative program.

10. Work should continue on the validation of the Rosaniline method for the determination of sulphite in white sugar.

11. Work should continue on the validation of the loss on drying method for white sugar.

### General Subject 3 : Refined sugars other than white sugar

*Referee : R. W. Plews (UK)*

1. The various products which fall within the scope of this Subject should be classified as follows :

- (a) Powdered sugars.
- (b) Partly-refined sugars.
- (c) Brown sugars.
- (d) Very pure syrups.
- (e) Coloured syrups.

2. The status of procedures previously adopted (as listed in the Referee's Report) should be confirmed as applying to products within the scope of this Subject. With respect to the determination of sulphite, this shall apply to powdered sugars only.

3. Methods for the determination of

sulphite in products other than white sugars should be studied.

4. The established method for polarization of raw sugar should be extended to apply to products within the scope of this Subject.
5. Methods for the determination of colour of brown sugars should be studied.
6. Methods for the determination of anti-caking agents in powdered sugars should be studied.
7. HPLC, HPIC and enzymatic methods for the quantitative determination of specific sugars, including sucrose, glucose and fructose, should continue to be studied.
8. Methods for the determination of dry substance in the products within the scope of this Subject should be studied.
9. All proposed studies will be pursued with the co-operation of the appropriate Scientific Referees.

#### General Subject 4 : Molasses

*Referee:* D. S. Martin (UK)

1. Further studies of the correlation between the Luff Schoorl and Lane & Eynon constant volume methods should be pursued through the Referee for Subject 15.
2. The Karl Fischer method for water content and its correlation with vacuum oven drying should be pursued through the Referee for Subject 17.
3. A method for total fermentable compounds should be established and fermentation properties in general should be investigated.
4. Co-operation with the Referees for Subjects 8 and 9 on the applications of GLC/HPLC to the analysis of molasses should continue.

#### General Subject 5 : Cane

*Referee:* M. A. Brokensha (South Africa)

1. Core sampling is adopted as an ICUMSA Accepted method.
2. Full width hatch sampling is adopted as an ICUMSA Accepted method.
3. The hydraulic press method should be further studied, with a view to attaining Tentative or Accepted Status at the 21st Session.

4. The wet disintegrator method (as submitted at the 19th Session) is Officially adopted.

5. The Referee for Subject 9 is requested to recommend the most suitable HPLC method for cane juice analysis and collaborative studies should then be undertaken, subject to the development of a suitable cane juice preservation procedure.
6. Collaborative studies of the GLC method of Schäffler, with the goal of moving to Official status, should be undertaken as soon as a suitable cane juice preservation procedure has been developed.
7. The investigation of freeze-drying of cane juice samples as a means of preservation and thus facilitation of collaborative studies, should be pursued.
8. Investigations into the use of clarification agents or procedures which will eliminate the use of lead salts in cane juice analysis should be pursued with urgency.
9. Investigations into the application of near infra-red spectroscopy for cane analysis should be pursued.

#### General Subject 6 : Beet

*Referee:* W. Mauch (Germany)

1. In view of the importance of samples being both representative and homogeneous, proposals should be evolved for standardizing apparatus for the preparation of brei and of brei characteristics; consideration should also be given to the comminution and handling of beet cossettes.
2. In order to create the necessary requirements for inter-laboratory testing, methods for sample preservation of brei and cossettes should be selected and tested.
3. In view of the widespread use of digestion-filtration units, proposals for the standardization of relevant instrumental and analytical parameters should be evolved.
4. The Recommendation of the 19th Session (*Proceedings 19th Session ICUMSA*, 1986, 163, Rec. 3) that clarification with aluminium sulphate

solution be Tentatively adopted, should continue to be valid and the procedure should be collaboratively tested in order to obtain Official status.

5. The development and examination of alternative routine and standard methods for the determination of sucrose (e.g. HPLC, enzymatic and NIR spectrometric) should be encouraged in collaboration with the Referee for General Subject 8; in doing so, the individual steps of the method should also be checked for causes of systematic errors.

#### General Subject 7 : Cane sugar processing

*Referee:* W. S. C. Tsang (USA)

1. Owing to poor reproducibility, the cation-exchange HPLC method for sucrose in molasses, adopted Tentatively at the 19th Session, should be further studied, under the guidance of the Referee for Subject 9.
2. HPLC methods for the determination of fructose, glucose and sucrose in process syrups and liquors should be studied, under the guidance of the Referee for Subject 9.
3. Methods for the determination of sugars in bagasse and filter muds, as well as in process effluents, should be further studied.

#### General Subject 8 : Beet sugar processing

*Referee:* J. P. Lescure (France)

1. The method described by Ivin<sup>2</sup>, modified by Schäffler, is Tentatively adopted to correct direct polarization for the purpose of conducting a sugar balance.
2. The method described by Schäffler<sup>3</sup>, modified by Oikawa is Tentatively adopted for the GLC analysis of sucrose in brei or raw juice.
3. The analysis of pulp should be studied, with special attention to sodium, potassium, calcium, nitrogen, dry substance, total ash, total sugar and sand contents.

2 Ivin, P. C.: *Proceedings 19th Session ICUMSA*, 1986, 127 - 135; K. J. Schäffler, Referee's Report for Subject 9, Appendix 1.

3 Schäffler, K. J.: *Proceedings 19th Session ICUMSA*, 1986, 114 - 121; S. Oikawa, Referee's Report.

4. Process control methods should be studied with special attention to lactic acid, nitrogen and amide contents.
5. A standard approach to collaborative tests to assess the performance of process laboratories should be developed.
6. Special attention should be paid to developing procedures for checking on-line analytical techniques.
7. Methods to be used in determining sugar losses during extraction should be studied.

**General Subject 9 : Starch hydrolysis products**

*Referee:* D. B. Whitehouse (Belgium)

1. This Subject should be maintained by ICUMSA, so that contact may be continued with the International Federation of Glucose Manufacturers. In this way, methods of analysis developed by the starch industry with application to this Subject may be notified to ICUMSA from time to time; equally, ICUMSA methods of interest to the starch industry may be communicated.

\* \* \*

**Subject 1 : Constitution and By-Laws**

*Referee:* M. R. Player (Australia)

1. The text of the Constitution and By-Laws will be studied with a view to proposing revisions to eliminate ambiguities.

**Subject 2 : Laboratory apparatus and reagents**

*Referee:* S. A. Brooks (West Indies)

1. Referees for other Subjects are requested to identify and specify specialized sugar apparatus which would be suitable for international standardization and to co-operate in this respect with the Referee.
2. Referees for other Subjects are requested to identify and specify reagents specially formulated to meet the requirements of the sugar industry, and which could meet with internationally-agreed standards. This information should be passed to the Referee.

**Subject 3 : Method format, collaborative testing and statistical treatment of data**

*Referee:* M. A. Godshall (USA)

1. The IUPAC protocol for the design, conduct and interpretation of collaborative studies is Officially adopted and this will be incorporated into ICUMSA By-Laws.
2. A Methods Review Committee will be established within the province of the Referee for Subject 3 and it is highly desirable that this Committee review the format and procedures of collaborative studies proposed by ICUMSA Referees. This is to ensure that these are in accordance with IUPAC requirements and therefore eligible for acceptance by ISO and AOAC.
3. Note will be taken of the Horowitz guidelines for acceptable variability in a method, which are listed below:  
 "The RSD<sub>R</sub> (%) of any given test will not usually exceed twice the value given below for chemical analyses:"

Analyte concentration	RSD <sub>R</sub> (%)
1 ppb	45
0.01 ppm	32
0.1 ppm	23
1 ppm	16
10 ppm (0.001%)	11
100 ppm (0.01%)	8
1000 ppm (0.1%)	5.6
1%	4
10%	2.8
100%	2

4. The ISO method format and numbering system is adopted and the method description will include matrix/analyte/ concentration and present performance characteristics, individually for each sample.
5. Fundamental values and reference tables, which cannot be collaboratively tested, are to be recognised as Official if they have met the following criteria:
  - (a) the data are obtained with an accuracy significantly greater than that used in practical applications.
  - (b) The data are published in detail in a refereed journal.
  - (c) There are no substantial

objections to the data in 4 years.

(d) The data are obtained by a recognised standardizing laboratory.

It should also be noted that it is considered highly desirable that Referees intending to carry out work in this area should advise others, to give them the opportunity to provide input into the work at an early stage.

6. Recommendations "for further study" should indicate a plan of action.
7. Ways to shorten the interval required to take action on methods should be explored.
8. The following Recommendations are to serve as guidelines for Referees in their Reports of Collaborative Studies:
  - (a) Report each collaborative study separately.
  - (b) Give a summary of the study at the beginning of the report. This should include:
    - (i) The method tested (with references).
    - (ii) The type and number of samples tested.
    - (iii) The number of participating laboratories.
    - (iv) A summary of the performance characteristics.
    - (v) The recommendations.
  - (c) Include all raw data in a table. This allows to others also perform their own statistics on the data.
  - (d) Include test results, sample by sample, in a table, following the Recommended IUPAC format (Appendices 1 & 3 of the Referee's Report).
  - (e) Include any other information gained about the method, such as interferences, ruggedness, variations tried, comments from collaborators, etc., in the report at the end.
9. Accepted, Tentative and Official status, as described in the Addendum to the Referee's Report, are Officially adopted. Further work will be undertaken to define a means of developing microbiological tests to Official status.

**Subject 4 : Polarimetry**

*Referee:* A. Emmerich (Germany)

1. Recommendations 1 to 4 of Subject 5 at the 19th Session, regarding the

Official adoption of the new polarimetric scale (*Proceedings 19th Session of ICUMSA*, 1986, 66 - 68) are confirmed.

- Emmerich's formula for temperature corrections to polarimetric measurements, Officially adopted at the 18th Session (*Proceedings 18th Session of ICUMSA*, 1982, 63) is confirmed.
- The use of near infra-red wavelengths for saccharimetry should be further studied.

#### Subject 5 : Quartz plates

Referee: K. Zander (Germany)

- Conversion of rotation values at the basic wavelength of  $\lambda_{vac} = 546.2271$  nm to other wavelengths in the range of  $\lambda_{vac} = 650$  nm to  $\lambda_{vac} = 1000$  nm should provisionally be made using the equation given by Bünnagel (*Proceedings 14th Session of ICUMSA*, 1966, 32).
- For using the 100°Z point at wavelengths other than 546.2771 nm, the equation agreed at the 14th Session (*Proceedings 14th Session of ICUMSA*, 1966, 17), should also be provisionally used for the wavelength of 650 nm to 1000 nm.
- Further measurements of the rotary dispersions of quartz and sucrose should be carried out in the spectral region of approximately 850 nm.

#### Subject 6 : Spectrophotometry

Referee: G. Mantovani (Italy)

- A method for the standard preparation of white and other sugar samples for trace metal determination by atomic absorption and emission spectrophotometry should be chosen and submitted to a collaborative study.
- Work should be carried out to select a method for determining formaldehyde in white sugar. A collaborative study should then be carried out in cooperation with the Referees for General Subjects 2 (White sugar) and 8 (Beet sugar processing).
- Near infra-red spectrophotometric methods should be further studied.
- The Blue Number method and fluorimetric analysis for amino-nitrogen should be further studied.

#### Subject 7 : Colour, turbidity and reflectance measurements

Referee: S. Akoglu (Turkey)

- Studies on the use of TEA buffer for pH adjustment in the measurement of colour in sugars other than white sugar should be continued, particularly to obtain data on a wide range of sugars.
- The determination of turbidity in sugar solutions should be further studied. The effect of instrument design should be taken into account in these studies.
- Studies on the use of probe-type colorimeters for sugar colour measurement should be continued and the results compared with those obtained from standard spectrophotometers.
- Investigation of reflectance measurement for the evaluation of the colour of white sugars should be continued, giving special consideration to the CIE L\* a\* b\* system.
- Studies on the application of reflection measurements, visual evaluations and solution colour measurements to the evaluation of brown sugars should be continued.
- The Imperial Sugar method for reflectance measurement of brown sugars should be studied internationally.
- Studies on the influence of crystal size, gloss and surface structure on visual grading and on reflectance measurements should be continued at a lower level of priority.
- In the measurement of solution colour of sugars other than white sugars by the ICUMSA method, the data presented in Table 2 of the Referee's Report should be adopted as guidelines for solution concentration and cell length.

#### Subject 8 : GLC methods

Referee: N. Kubadinow (Austria)

- In co-operation with the Referees for the General Subjects, a method should be sought which allows for the simultaneous determination of sucrose, glucose, fructose and trisaccharides in cane and/or beet products.

#### Subject 9 : HPLC methods

Referee: K. J. Schäffler (South Africa)

- Further testing of the cation-exchange HPLC method for sucrose, glucose and fructose in cane molasses is required. The reason that over-estimation is occurring on occasions should be established. Cations other than sodium and calcium should be tested. A new method with tighter control of the major variables should be drawn up.
- If Recommendation 1 is successful, then the cation-exchange HPLC method should be re-evaluated in an inter-laboratory study.
- An alternative HPLC method using anion-exchange and pulsed-amperometric detection should be drawn up for inter-laboratory studies.
- An HPLC procedure for betaine in beet molasses should be developed and tested.

#### Subject 10 : Enzymatic and immunological methods

Referee: D. F. Day (USA)

- A series of criteria should be established for the standardization of enzymatic reagents used in Recommended methods.
- A series of criteria should be established for the standardization of immunological reagents used in new methods.
- Inter-laboratory tests should be carried out in co-operation with the appropriate Referees, in order to assess the validity of existing enzymatic methods (e.g. SO<sub>2</sub>, glucose, fructose, sucrose, D- and L-lactic acid).
- New enzymatic methods should be developed with the collaboration of interested laboratories (e.g. pectin, glutamine, ammonia, etc.)

#### Subject 11 : Density

Referee: F. Spieweck (Germany)

- Polynomial (1) is Officially adopted in place of Plato's density table for the determination of the density of aqueous sucrose solutions as a function of mass concentration and temperature.
- For technical applications, polynomial (2) is Officially adopted for the deter-

mination of the density of aqueous sucrose solutions as a function of mass fraction and temperature.

3. Confirmation of the results obtained with polynomials (1) and (2) should be sought by approaching other standardizing laboratories to carry out independent checks.

**Subject 12 : Rheology**

*Referee:* R. Broadfoot (Australia)

1. The Power Law model is adopted for the presentation of rheological data for massecuites and molasses. The range of shear rates used for the measurements should be nominated in addition to the values of consistency and flow index. It is also recommended that the geometry of the measuring systems should be clearly defined.

2. Studies should continue to investigate those factors which influence the rheological properties of molasses and massecuites. This work should include problems of crystal migration which may occur in the field of shear, while measuring the viscosity of massecuite.

3. In using the Official ICUMSA rotating cylinder method for determining the rheological properties of molasses, the geometry of the measuring system, particularly the ratio of the diameter of the spindle to the diameter of the coaxial cylinder reservoir, should continue to be defined, until an assessment is made to determine whether the geometry of the measuring system influence the values of the Power Law parameters.

4. The falling-ball viscometer should be deleted as a suitable alternative to the rotating cylinder method for measuring the viscosity of molasses.

5. Further studies should be conducted into the use of the pipeflow method for both massecuite and molasses. Emphasis should be given to specifying the appropriate tube dimensions or defining a procedure to correct for pressure losses at the tube ends and for wall effects.

6. The pipeflow method should not be offered as an alternative to the rotating cylinder method, for determining the rheological properties of molasses, until

the procedures to correct the pipeflow data for tube and effects are defined and further comparisons of data from the two methods are made with molasses showing non-Newtonian behaviour.

7. Investigations of the elastic flow properties, tack, interfacial tension and surface tension of molasses and massecuites should be continued.

8. The work towards establishing standard measurement procedures for the rheological properties of massecuites should be confined to the rotating cylinder and pipeflow techniques. The interest in other viscometric methods should be of a general nature and not directed to developing them as standard techniques.

**Subject 13 : Refractive index**

*Referee:* K. J. Rosenbruch (Germany)

1. Equation (1) and the associated coefficients described in Table 1 of the Referee's Report for the refractive index in standard air of aqueous solutions of D-glucose, D-fructose and invert sugar at concentrations of 0 to 85%, temperatures of 15°C to 30°C and wavelengths from 546.1 nm to 589.3 nm are Officially adopted.

2. Equation (2) and the associated coefficients described in the Referee's Report for correcting refractometric dry substance to true dry substance for mixtures of aqueous solutions of sucrose and invert sugar at concentrations from 0 to 85%, a wavelength of 589.3 nm and a temperature of 20°C, are Officially adopted.

3. Studies of the accuracy achievable for refractive index measurements of technical sugar solutions, with commercially available refractometers, should be continued.

4. The equation given in Subject 12 of the 17th Session (*Proceedings 17th Session of ICUMSA, 1978, 166*) for the refractive index in standard air of aqueous solutions of sucrose at concentrations from 0 to 85%, temperatures from 15°C to 30°C and wavelengths from 546.1 nm to 589.3 nm is Officially adopted.

**Subject 14 : Microbiological tests**

*Referee:* R. Strauss (Germany)

1. As a result of many collaborative tests by internationally recognised institutes, the method for the enumeration of mesophilic bacteria in sugar by concentrating the sample on membrane filters (pore size between 0.2 and 0.45 µm) and using plate count medium or nutrient medium at 30°C incubation temperature should be confirmed.

2. With regard to slime-forming bacteria:

(a) It is recommended that the development of a test for slime forming bacteria which indicates the quality of a sugar, should be confirmed.

(b) Both Weman (B1) and McCleskey (B2) (*Proceedings 19th Session of ICUMSA, 1986, 372*) media should be further investigated and compared with other media for the detection of lactic acid-forming bacteria.

3. For yeasts and moulds:

(a) The nutrient media Wort agar (C1), Yeast glucose (C2), Mycophilic (C3) (*Proceedings 19th Session of ICUMSA, 1986, 373*) should be investigated in further tests for yeasts and moulds.

(b) The following modified medium of de Whalley should be further investigated in testing for osmotolerant yeasts:

	Per litre
Yeast extract	5g
Peptone	2g
Soluble starch	2g
Glycerol	2g
Ammonium chloride	1g
Glucose	20g
Sucrose	400g
Agar	16g
pH	5.7

*Fractionated Sterilization:* 3 × 20 min, at 100°C

*Incubation Temperature:* 30°C or 25°C

*Incubation Time:* 72hr to 6 days

(c) The incubation temperature for yeasts and moulds should be subjected to further collaborative studies, as the temperature specified in the new ISO standard differs from that recommended

by ICUMSA.

4. In the case of thermophilic spore-forming bacteria:

(a) Because of improvements in eliminating vegetative organisms in tests for thermophilic spore-forming bacteria, a modification of the AOAC method for the detection of spore-forming bacteria in sugar should be developed.

(b) Moreover the comparative tests should include the method using phenol red as acid indicator.

5. Developments in the area of rapid microbiological testing should be monitored.

#### Subject 15 : Reducing sugars

Referee: J. Laursen (Denmark)

1. The Luff School method, as described in Appendix 2 of the Referee's Report, with the modifications described under point 2.1.5, is Tentatively adopted for the determination of total sugars in molasses.

2. The reasons for the difference in total sugars in samples of beet molasses by the Luff School method in comparison with the Lane & Eynon method should be further studied.

3. The determination of total sugars in syrups and feedstuffs, other than molasses, by the Luff School method, as described in Appendix 2 of the Referee's Report, with modifications described under point 2.1.5, should be further studied.

4. The sucrose correction factors for the Constant Volume modification of the Lane & Eynon method, as shown in Table 6 of the Referee's Report, are Officially adopted. The correction factors adopted at the 17th Session (*Proceedings 17th Sessions of ICUMSA*, 1978, 194) are withdrawn.

#### Subject 16 : Ash

Referee: J. P. Ducatillon (France)

No Recommendations were adopted under this Subject.

#### Subject 17 : Dry substance

Referee: G. Vaccari (Italy)

1. The method for the determination of

moisture in raw sugar, as specified in Appendix 2 of Subject 13 of the 19th Session (*Proceedings 19th Session of ICUMSA*, 1986, 225) is Officially adopted when the moisture content of the raw sugar is greater than 0.15%. In place of aluminium dishes, glass dishes may be used. It is recognised that this method is most reliable above 0.5% moisture.

2. For raw sugar having a moisture content of less than 0.5%, the Official method for determining "Loss on Drying" of white sugar ("Sugar Analysis - ICUMSA methods" by F. Schneider, 1979, 113, as modified in *Proceedings 18th Session of ICUMSA* 1982, 330 and *Proceedings 19th Sessions of ICUMSA*, 1986, 348) should be taken into consideration. A collaborative test should be carried out, comparing this procedure with the procedure Recommended in 1 above.

3. The formula developed by Morton & Muller for correcting refractometric solids contents of recovery products, which is used by Tate & Lyle, should be taken into consideration. The Associate Referees should evaluate its applicability on their local sugar products, with the aim of its possible adoption as an Official correction.

4. Equation 2 of Subject 13 "Refractive Index" for correction of the refractometric dry substance of white sugar syrups which contain defined amounts of invert sugar, is Officially adopted.

5. New measurement techniques, such as NIR-spectroscopy, should be further studied if the results which they give are comparable with those obtained by traditional ICUMSA methods.

6. The Karl Fischer and vacuum oven drying methods should be collaboratively tested before being considered for Official adoption.

#### Subject 18 : Sucrose

Referee: S. E. Bichsel (USA)

1. It is Recommended that Subject 18 be discontinued and the work accommodated in other Subjects.

#### Subject 19 : Oligosaccharides & polysaccharides

Referee: K. Thielecke (Germany)

1. Specific enzymatic methods for dextran, such as that of Sayama & Kamada (*Sugar Industry Abstracts*, 88 - 1632) should be further developed. The type of enzyme used and its purity should be defined.

2. Immunological methods for dextran determination involving the standardization of antiserum and the development of test kits should be further studied.

3. Following the recommendations of the Review of Methods in Subject 3, the CSR method for estimation of dextran in raw sugar should be renamed the "haze method" (see Subject 3, Appendix 2). Since the variability is known, the method could qualify for Official status under the auspices of General Subject 1. The method is no longer a matter for Subject 19. (*Editor's Note: The method was not adopted under General Subject 1*).

4. Following the adoption of the method by the AOAC, the Roberts method for dextran in raw cane sugar could qualify for adoption under the auspices of General Subject 1. The method is no longer a matter for Subject 19. (*Editor's Note: The method was not adopted under General Subject 1*).

5. Pectic acid methods based upon enzymatic hydrolysis followed by HPLC or enzymatic quantification, should be developed further and compared with photometric methods, such as that of Reinefeld & Schneider (*Sugar Industry Abstracts*, 83 - 1235).

6. The Referee for Subject 3 should evaluate the Schiweck & Büsching method for raffinose in molasses and other juices. The procedure using Carrez clarification, if necessary, as outlined in the Braunschweig modification (*Sugar Industry Abstracts* 88 - 1618) should be Tentatively adopted for total  $\alpha$ -galactosides.

7. For technical purposes, a 20% reduction of the enzymatic total  $\alpha$ -galactoside result may be used to account for the galactinol in impure beet syrups, until suitably accurate enzymatic or other

methods for raffinose become available.

8. The Official Schiweck & Büsching enzymatic method for raffinose in white sugar should be re-evaluated under General Subject 2 "White Sugar". The method is no longer a matter for Subject 19.

9. The use of the I.R.I.S GLC method (Appendix 2 of the Referee's Report), as a reference method for raffinose in beet molasses, should be further studied.

10. HPLC and HPIC (HPAEC) methods for technically-important oligosaccharides and galactinol should be further studied.

11. Work aimed at preparing analytical standards, especially for galactinol and the kestoses, should be encouraged.

12. Following the recommendations made in the Report on the Review of Methods (Subject 3 Report, Appendix 2), the methods below should be retained, until more advanced methods presently being assessed are considered for adoption:

(a) Reinefeld, Thielecke & Lücker TLC method for dextran, levan and araban in technical juices (Tentative adoption, 1982).

(b) Carruthers & Oldfield photometric method for pectic acid in beet

raw juice (Tentative adoption, 1978).

(c) Reinefeld, Thielecke & Lücker photometric method for pectic acid in beet raw juice (Tentative adoption, 1982).

(d) British Sugar TLC method for raffinose (Tentative adoption, 1974).

(e) Schiweck HPLC method for oligosaccharides and galactinol (Tentative adoption, 1982).

These methods are considered to be Accepted methods.

13. Analytical methods for oligo- and polysaccharides and preparation procedures for standards under consideration should be extracted from the literature and tested.

14. Following the recommendations made in the Report on the Review of Methods (Subject 3 Report, Appendix 2), the following obsolete, or seldom-used methods, should be retired:

(a) Albon & Gross paper chromatography method for raffinose and kestoses.

(b) Braunschweig paper chromatography method for raffinose.

(c) Paine & Balch enzymatic-polarimetric method for raffinose.

(d) McCready enzymatic-photometric

method for raffinose.

(e) Böttger & Steinmetzer enzymatic method for raffinose.

(f) Serro & Brown paper chromatography method for galactinol.

(g) Atterson *et al.* TLC method for dextran.

(h) Atterson *et al.* TLC method for levan.

(i) British Sugar TLC method for araban.

### Subject 20 : Ion selective electrodes and ion chromatography

Referee: P. Bourlet (France)

1. Studies of the analyses of the inorganic anions, chloride, sulphate and sulphite in white sugar, refined sugar products and raw sugar should be carried out.
2. Analysis of the inorganic anions, chloride, sulphate, nitrate and nitrite in molasses should be further studied.
3. A list of organic acids for which characterization would be interesting should be drawn up, in order to define the proper mode of analysis.
4. Monovalent and divalent cation analysis should be further studied, in order to determine the limits of this application of ion chromatography.

## Facts and figures

### China sugar exports, 1989<sup>1</sup>

Exports of sugar from China in 1989 rose to 465,000 tonnes, raw value, from 270,000 tonnes in 1988. Hong Kong received 69,000 tonnes (83,000 tonnes in 1988), Mexico 13,000 tonnes (0), and the USSR 13,000 tonnes (0), while the destinations of the remaining 370,000 tonnes are unknown. In 1988 Pakistan received 12,000 tonnes and Singapore

4000 tonnes, while unknown destination received 171,000 tonnes.

### Reduced Mexican sugar crop forecast<sup>2</sup>

Mexico's 1991 sugar crop is forecast at 3,150,000 tonnes, raw value, down from the 3,425,000 tonnes forecast earlier but up from the 3,000,000 estimated 1990 crop, according to the US agricultural

attache. Plantings and harvested area were reduced as a result of low cane prices, higher production costs and reduced subsidies for the domestic sugar industry. As a consequence, sugar imports are expected to continue at 100,000 tonnes, nearly the record levels of 1989/90. Private sources believe that imports will in fact double, to 200,000 tonnes. Uncertainty remains over how privatization of the state owned sugar factories will affect production, but it is expected that Mexico will continue to supply its sugar quota to the United States.

### Aconitic acid removal during cane juice clarification

*continued from page 230*

réglage facile du pH. Le procédé de sulfitation enlève moins d'acide aconitique (16.3%) que la phosphatation mais il ne réalise pas une bonne épuration. La phosphatation avec du superphosphate de chaux enlevait 20.2% d'acide aconitique.

Ce taux était de 73.8% pour la défécation et de 78.8% pour le procédé de carbonatation. Dans le procédé de phosphatation, l'enlèvement d'acide aconitique augmente avec le pH et avec la quantité d'acide phosphorique utilisée.

### ACTALAC conference, 1990

The 1990 Congress of ACTALAC (the

<sup>1</sup> *I.S.O. Stat. Bull.*, 1990, 49, (7), 2.4.  
<sup>2</sup> *Reuters News*, July 13, 1990.



Latin American and Caribbean Association of Sugar Technologists) was scheduled to be held during September 9 - 14 in Cali, Colombia, within the framework of the 3rd Congress of the Colombian Society of Sugar Cane Technologists, TECNICAÑA. ACTA-LAC was established in 1979 during the XI GEPLACEA Assembly as a regional group to provide self-help against the difficulties faced by the sugar industry during the 1980's.

#### Algeria sugar imports, 1989<sup>3</sup>

	1989	1988
	<i>tonnes, raw value</i>	
Brazil	26,000	15,000
Cuba	189,000	163,000
Dominican Republic	0	20,000
EEC	405,000	460,000
Germany, East	26,000	67,000
Guatemala	14,000	0
Korea, South	13,000	0
Mexico	0	26,000
Sweden	12,000	0
Thailand	12,000	0
	697,000	751,000

#### Reduction in Thailand sugar exports estimate<sup>4</sup>

Thailand's sugar exports are expected to fall to 2,720,000 tonnes in calendar 1990 from 2,920,000 tonnes in 1989, according to an agency of the Ministry of Commerce, which attributed the expected fall to a reduced cane crop and the fact that Thailand's domestic sugar consumption is expected to rise to 980,000 tonnes this year from 907,500 tonnes in 1989.

#### Mexico sugar factory sales to Coca-Cola<sup>5</sup>

Negotiations were being completed in July for the sale of Ingenio La Abeja by the State-owned organization Azúcar S.A. to the Scorpio Group, which represents the Coca-Cola company. The Group has already bought Ingenio Atencingo. A successful agreement was expected to permit an immediate start to repairs which would allow increased production in the crop commencing in December.

#### Egypt sugar imports, 1989<sup>6</sup>

	1989	1988
	<i>tonnes, raw value</i>	
Australia	26,000	0
Brazil	158,000	133,000
Cuba	38,000	50,000
EEC	328,000	528,000
Germany, East	26,000	36,000
Guatemala	15,000	13,000
Mexico	0	15,000
Poland	0	42,000
Sweden	4,000	10,000
Thailand	22,000	0
USSR	0	24,000
	617,000	851,000

#### Brazil sugar factory co-generation<sup>7</sup>

As from February 1990, the União sugar factory in Pernambuco has been furnishing 340 MW electricity to the public grid; this amount is sufficient to supply the city of Recife, where the factory is located, and the cost \$18/MWh.

#### Guatemala sugar exports, 1989<sup>8</sup>

	1989	1988
	<i>tonnes, raw value</i>	
Algeria	14,074	0
China	0	38,000
Ecuador	69,735	29,661
Egypt	15,156	13,209
Guyana	15,006	3,500
Haiti	2,950	6,000
Jamaica	0	26,463
Mexico	103,403	0
Morocco	0	10,820
Sri Lanka	0	14,069
Trinidad	10,108	29,826
USA	78,682	30,912
USSR	99,476	179,096
Venezuela	0	9,200
	408,590	390,756

#### Iran sugar expansion plans<sup>9</sup>

The head of an Iranian government department set up to encourage foreign investment in Iran has said that the country's Central Bank is committed to providing US\$ 1500 million for the construction of seven cane sugar factories. Any foreign investment in these plants will start to be repaid within

one year of the start of operation of the relevant plant.

#### New Bangladesh sugar factories

The Bangladesh Vice-President in charge of the Ministry of Industry has said that two new sugar factories in the public sector are to be set up in the less developed north-east of the country to help achieve self-sufficiency. The official news agency BSS quoted him as saying that the new factories will be set up in the Pavana and Nilphamary districts, with financial support from Pakistan and China.

#### Peru sugar imports, 1989<sup>10</sup>

	1989	1988
	<i>tonnes, raw value</i>	
Brazil	12,000	48,600
Cuba	30,000	0
EEC	90,000	158,701
USA	20,000	0
	152,000	207,301

#### Sri Lanka sugar factory expansion

The Sri Lanka Sugar Co. Ltd is to call for the tenders for the expansion of the Sevanagala sugar factory from its present capacity of 1250 t.c.d. to 2000 t.c.d., as well as increasing production capacity of the distillery section from 150 to 250 hl/day of potable, industrial or anhydrous alcohol from cane molasses. The cost will be met by a loan from the Asian Development Bank<sup>11</sup>.

#### West German activity in the East German sugar industry<sup>12</sup>

A joint enterprise was established in August between the West German sugar company Pfeifer & Langen and East German sugar beet growers. The new company, Pfeifer & Langen Langenbogen GmbH & Co. KG, has been guar-

3 *I.S.O. Stat. Bull.*, 1990, 49, (7), 2.1.

4 *Reuters News*, July 5, 1990.

5 *GEPLACEA Bull.*, 1990, 7, (8), Sugar Inf.-2.

6 *I.S.O. Stat. Bull.*, 1990, 49, (7), 2.5 - 2.6.

7 *Alcool & Açúcar*, 1990, (52).

8 *I.S.O. Stat. Bull.*, 1990, 49, (7), 2.5 - 2.6.

9 *Islamic Republic News Agency report*, August 9, 1990.

10 *I.S.O. Stat. Bull.*, 1990, 49, (7), 2.13 - 2.14.

11 *See I.S.J.*, 1990, 92, 192.

12 *Zuckerindustrie*, 1990, 115, 621.

anteed a large enough quota to process 15,000 tonnes of beet per day and the cornerstone has been laid of a new sugar factory at Langenbogen, near Halle, which is to begin operation in 1992 with the initial capacity of 8000 tonnes/day. Süddeutsche Zucker AG, the biggest West German sugar company, has submitted plans for the modernization of five sugar factories in the southern part of East Germany. These activities put into question the future of Deutsche Ostzucker AG which was supposed to be the umbrella organization of the East German sugar industry<sup>13</sup>.

#### Portugal sugar imports, 1989<sup>14</sup>

	1989	1988
	tonnes, raw value	
Argentina	131	11,686
Belgium	7,065	31
Brazil	33,536	66,484
Egypt	11,167	5,236
France	19,686	46,217
Germany, West	422	278
Holland	318	1,546
Italy	9,648	2,702
Spain	4,336	4,111
UK	500	389
USA	7,005	83
Other countries	231,186	215,237
	325,000	354,000

#### New Madagascar sugar factories

All-India Radio reported on August 20 that India has agreed to cooperate in the construction of three new sugar factories in Madagascar, according to reports from that country's capital, Antananarivo. It is not known when construction will start.

#### South African cane alcohol project rejected<sup>15</sup>

The South African government has rejected the sugar industry proposal for a plant to produce alcohol from sugar cane<sup>16</sup>. After investigation by a special committee appointed last year to examine the 80 million rand proposal for a facility to produce 150 million litres/year, the cabinet decided it would need too much government support to make it worthwhile. When it was proposed, the

project was said to offer a prospect of up to 20,000 jobs and reduce South Africa's dependence on oil imports.

#### Rwanda sugar expansion plans<sup>17</sup>

Owing to increased sugar demand, expected to rise from the current 16,000 tonnes/year to 23,000 tonnes/year by 2000, production capacity is to be increased. This will involve raising of cane yields by extension of fertilizer use and improved methods, while the factory equipment is to be rehabilitated and the processing capacity doubled. This will cost 63 million French francs and the foreign exchange costs of 36.5 million francs will be financed by a loan from the French Central Bank for Economic Cooperation.

#### Yugoslavia beet crop reduction

A brief report from the Tanjung news agency in Belgrade on September 12 said that Yugoslavia had lost 1.4 million tonnes of sugar beet during the summer harvest as a result of a severe drought in the country.

#### Guyana sugar industry management agreement

Having secured funds from a number of financial institutions, the Guyana government reached an agreement on October 1, 1990 with Guyana Sugar Corporation, Booker plc and Booker Tate whereby the last will take over management of the Guyana sugar industry, operating both the cane estates and eight sugar factories. It is hoped that sugar production will thereby be increased; in 1989 it fell to 170,497 tonnes, raw value, less than half of output before nationalization.

#### Indonesia sugar industry development

According to an official of the Indonesian Ministry of Agriculture, there will be no more sugar factories built in Java and all new plants will be set up in other parts of the country. A number of factories are being rehabilitated and

productivity of the cane plantations is also being increased. The Secretary of the Indonesian Sugar Council said recently that several Australian and Taiwanese investors had expressed interest in establishing sugar factories in Indonesia; the government will provide incentives for investors if the sugar plants are set up outside Java and considers such incentives to be reasonable as it now costs at least \$130 million to build a sugar factory.

#### Danisco A/S 1989/90 report

In 1989/90, Danisco A/S, which includes A/S De Danske Sukkerfabrikker (The Danish Sugar Corporation) acquired Sukkerfabriken Nykobing, thereby becoming the sole Danish sugar producer. A rationalization program was thus made possible and Stege sugar factory was closed, while production is being extended at the remaining five factories. A large program of modernization and automation has been initiated, to cost 1000 million Danish kroner over the coming ten years, which will include a reduction of water consumption and waste water discharge, as well as increasing efficiency and reducing production costs. Total sugar production in 1989/90 amounted to 488,000 tonnes, while domestic sales amounted to 224,000 tonnes. Exports in 1989, mostly to the UK, Norway, the Faroe Islands and Iceland, amounted to 253,000 tonnes.

#### Bagasse board manufacture in Pakistan<sup>18</sup>

Al-Noor Sugar Mills Ltd. in Pakistan has put into operation a plant for the manufacture of medium-density bagasse fibre boards with a capacity of 30,000 cubic feet per annum. The plant includes a refiner especially designed for bagasse, a steam-heat dryer, scale, fibre recipient, press and finishing equipment.

13 *I.S.J.*, 1990, 92, 174.

14 F. O. Licht, *Int. Sugar Rpt.*, 1990, 122, S.353.

15 *Reuters News*, August 24, 1990

16 *I.S.J.*, 1989, 91, 215.

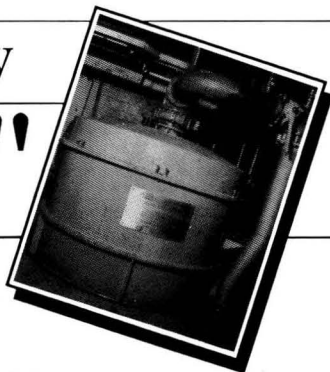
17 F. O. Licht, *Int. Sugar Rpt.*, 1990, 122, 439.

18 *O. Papei*, 1989, 50, (10); through *GEPLACEA Bull.*, 1990, 7, (9), Sugar Inf. 2.

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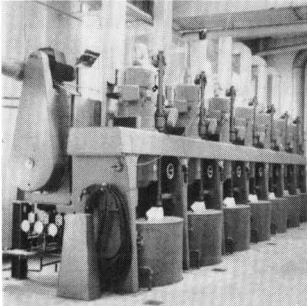
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- (2) Landsverk 48" dia. auto batch centrifugals
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- (2) Putsch beet slicers
- (1) 8" x 42" Louvre-type sugar dryer/cooler - 2-drums
- (2) 3,000 kVA Turbo generators, AEG
- (1) 308 kVA Diesel generator
- (1) 200 HP Ingersoll-Rand LLE air compressor
- (1) 75 kW Siemens vacuum pump
- (3) Kahl KW pellet mills
- (3) Dorr-Oliver 8' x 12 rot. vac. mud filters, steel, w/spray wash.



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- Crystallizer - Vertical, 3500 cu. ft. - New 1988!  
Filters - (10) Pronto 512 sq.ft., S.S.; (3) Anker 200 sq.ft. S.S.  
Stord Bartz MS-64 pulp press  
(2) Sihi CO<sub>2</sub> gas pumps, 430 HP

### 2,000 TCD at Barbados, W.I. ... Shut down May 1988

- Mill tandem with (5) 3-roller mills:  
(3) Fulton 27.5" x 48", inclined headstocks ...  
(2) new 1984/85  
(4) Farrel 28.5" x 48", 9" dia. rams  
Evaporation station, crystallizers, vacuum pans, etc.  
(1) 1,400 sq.ft. vacuum pan, stainless steel

### EUROPE

- (2) Putsch 2200 mm beet slicers  
(3) Evaporator bodies: 15,000; 22,000; 26,000 sq.ft. with S.S. tubes  
Diffuser 2000 TPD Vertical  
Diffuser 4000 TPD DDS  
Evaporator 7500 sq.ft. stainless steel  
Generator 6623 DVA Non-Cond.  
Beet sugar factory 4000 TPD

### Centrifugals

- (37) 48" x 30" ASEA-LANDSVERK auto batch  
(3) BMA K850 continuous  
(8) 49" x 44" ASEA Weibull auto batch  
(4) 40" x 30" Western States  
(2) 54" x 40" Western States auto batch  
(2) 34" Western States continuous  
**Other items**  
(1) Worthington turbo-generator, 2500 kW, recently overhauled, w/controls.  
(1) 1,400 cu.ft. Vacuum pan, stainless steel, with agitator and drive  
(1) Blaw Knox 3,000 sq.ft. falling film evaporator, nickel/T316 S.S.  
(5) Sihi vacuum pumps, 75, 120, 400 HP  
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## **Index to Advertisers**

British Rema Mfg. Co. Ltd. ... ..	iv
Thomas Broadbent & Sons Ltd. ... ..	Cover II
Contra-Shear Engineering Ltd. ... ..	iv
ManExec Inc. ... ..	v
Nadler Inc. ... ..	vi
NQEA Australia Pty. Ltd. ... ..	v
John H. Payne Inc. ... ..	v
Perry Equipment Co. Inc. ... ..	vi
H. Putsch GmbH & Co. ... ..	Cover IV
Rohm & Haas Separation Technologies ... ..	iii
Sugarequip Pty. Ltd. ... ..	Cover III
Wabash Power Equipment Co. ... ..	Cover III

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One 84" x 43" MIRRLEES WATSON Four Roller Mill  
Two 72" x 38" MIRRLEES WATSON Four Roller Mills  
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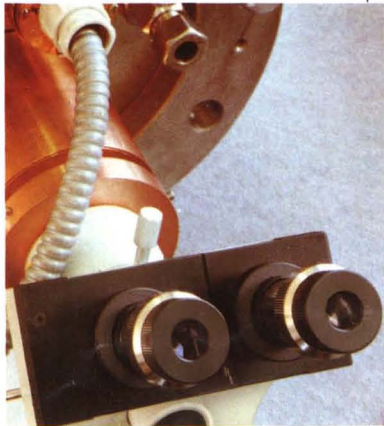
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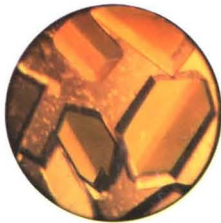
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