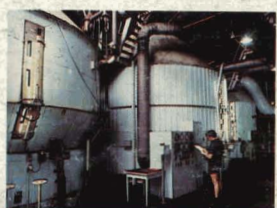
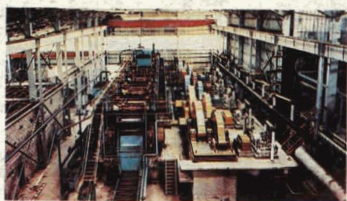


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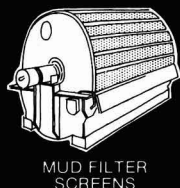
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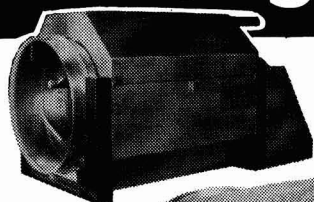
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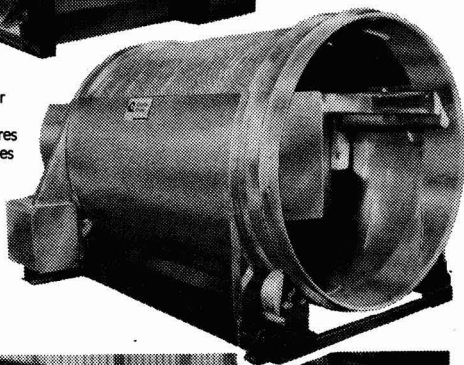
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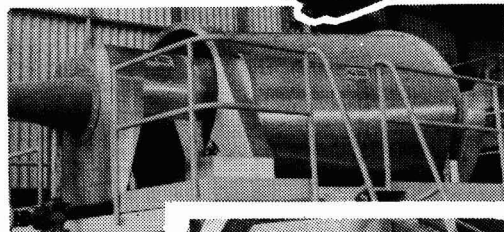


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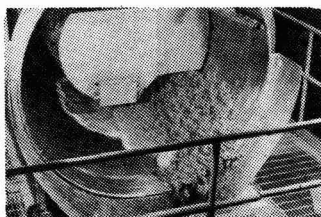
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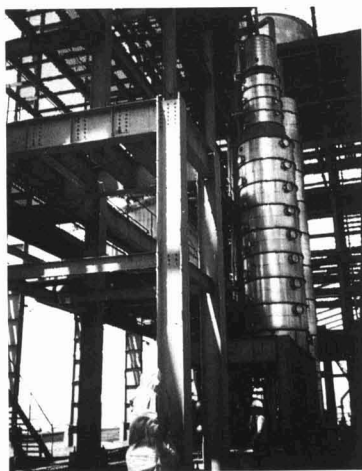
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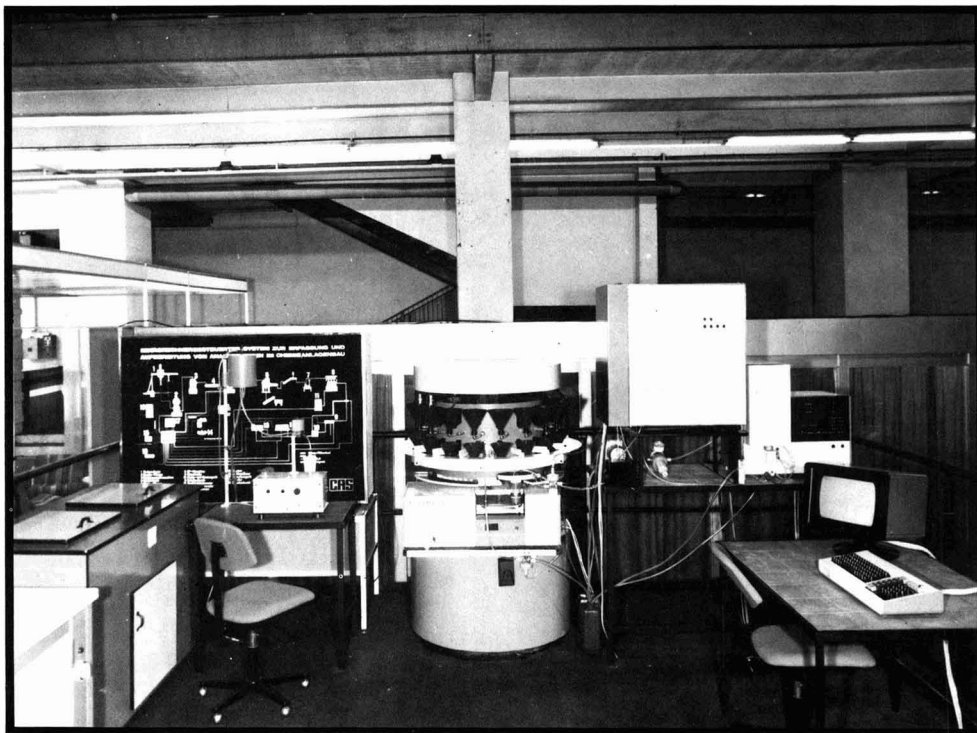
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# NOTES AND COMMENTS

## International Sugar Agreement talks

Delegates will have only one more chance to try and break down the barriers which stand in the way of a new International Sugar Agreement when they meet in Geneva on June 12. That appeared to be the only concrete decision to emerge from the informal round of talks in London in early April. Sr. Jorge Zorreguieta, who has chaired all the previous attempts at compromise, told reporters that many leading nations wanted the June session to be the last. Many of them need agreement by the beginning of July at the latest for it to come into force in 1985 for legal, constitutional and administrative reasons. His remarks were backed by the ISO Executive Director, William Miller, who admitted that he is not anticipating failure and will only think about it if and when it happens. It is presumed that there will be at least an administrative pact to continue from the current ISA but Miller said that another negotiating conference would not occur for a long time.

There appear to be two major areas of disagreement where the various delegations appear entrenched: the first concerns the proposed Reference Export Availabilities (REA) which some countries want to be based on actual performance over recent years, while Cuba and India want enhanced tonnages to reflect their planned and actual expansions of production. The EEC has claimed an REA based on recent exports but availabilities and potential markets have since shrunk and, in any case, the high EEC exports were achieved when ISA exporting members were restricting their sales under the terms of the Agreement.

The second major area is in the special arrangements, particularly between Cuba and the Comecon countries and China. Cuba has been adamant that these should not be the concern of a new ISA, yet it would seem to be a problem to work out an equitable REA for Cuba if these were not taken into account and, furthermore, sales by other exporters to China and the USSR could be displaced by Cuban sugar if sales of the latter were unrestricted. It has been suggested that Cuban sales to the socialist bloc could be unrestricted while also subjecting that country's total world sales to the same sort of limitation by REA as would apply to all the other major sugar exporters.

Certainly, compromises seem only to emerge in the closing stages of negotiating conferences and it is to be hoped that intransigence does not mark the June 1984 meetings since it seems likely that in the absence of a willingness to make concessions an effective ISA will recede from view for a long time.

## No Federal aid for the Australian sugar industry<sup>1</sup>

Australia's depressed sugar industry cannot look to the Federal Government for large amounts of aid either through an income transfer to producers from consumers or budget payments, according to the Federal Primary Industry Minister. Opening the annual conference of the Queensland Cane Growers Council, he said that the

government believes aid should only be provided as part of a package dealing with the fundamental structural problems of the industry. He dismissed as too expensive for the government an industry proposal for a price underwriting scheme and another proposal for setting domestic sugar prices to the average industry cost of production.

He added that the industry's structural problems must be dealt with in any aid package included in a new five-year sugar agreement between the Federal and Queensland governments, effective from July 1. The agreement will cover the marketing of domestic sugar, which is priced well above export sugar and accounts for about 25% of Australia's annual production, which reached 3.17 million tonnes in 1983. On the industry's structural deficiencies, the Minister said that the current system of assigning sugar land has created land-locked sugar factories unable to share in any expansion and has limited the scope for shifting production into areas best suited for cane.

## EEC first step to CAP reform<sup>2</sup>

The EEC has taken the most important step in its history towards limiting farm spending which has pushed the Community to the edge of bankruptcy. On March 31, 1984, the EEC ministers agreed on EEC support prices for 1984/85 as well as on measures in the currency sector which represent, in principle, a major first step towards a long-overdue reform of the ruinously expensive Common Agricultural Policy (CAP). Moreover, arguments over the farm policy have been a serious obstacle to a settlement of other contentious issues.

The two most crucial achievements of the deal are largely philosophical ones; first, it acknowledges for the first time in the 20-odd years of the operation of the CAP that demand must play a part in the equation balancing farmers, consumers and Community interests. The second hard reality for the farmers is that real cuts in product prices, the first on any scale since the foundation of the CAP, have now been agreed by the Ten.

Concerning sugar the ministers agreed to freeze prices for 1984/85 while generally farm prices have been reduced by 1%. For most major commodities this will have little impact on prices paid to farmers in their own national currencies, owing to adjustments in the "green" rates used for conversion from the Community's own currency, the European Currency Unit (ECU) to marks, pounds, etc. Producers in West Germany, Holland and the UK will see a minimal price cut of less than 1% while there will actually be price rises in the other countries, reaching 5% for payments in francs to French farmers. Monetary Compensation Amounts are to be phased out, ending a long-running dispute between France and West Germany.

The provisional agreement between the member states emphasizes that farm spending must in future grow more slowly than the rest of the Community budget without specifying how much more slowly. For most observers this does not seem to be tough enough but the principle that finance must determine expenditure, rather than the other way about, is a major change in Community doctrine, a belated recognition of a reality long accepted by member states in the national budgets.

As was to be expected, the ministers were criticized heavily by farmers' organizations throughout the EEC. The strongest attacks came from dairy producers who claim that the average 7% cut-back in output, together

<sup>1</sup> *Reuter Sugar Newsletter*, February 27, 1984.

<sup>2</sup> F. O. Licht, *International Sugar Rpt.*, 1984, 116, 195-197.

with a 1% rise in the cost-responsibility tax, will bankrupt thousands of dairy producers. However, the dairy sector is the most expensive part of the CAP and, by setting a ceiling for milk production significantly higher than that proposed by the Commission, and by making special concessions to the Irish milk producers, the ministers have enabled EEC dairy farmers to continue to produce substantially more than the Community can consume, so that the disposal of the surplus will continue to impose a major burden on the Community budget.

### World sugar prices

Little variation in the London Daily Price occurred during April and there was little physical trading. From its high point of £115 (\$166) on April 2, the LDP fluctuated in a range down to £110 (\$157) during the month, ending at £112 (\$156.50) on April 30. The LDP(W) similarly fell from \$180 per tonne on April 2 and reached \$175.50 on April 30, with drops to as low as \$170 during the month. The announcement that Brazilian sugar production was to be reduced to 8.5 million tonnes against 9.1 million tonnes in 1983/84 was a helpful factor, but E.D. & F. Man note<sup>3</sup> that, in spite of current price levels, there are still no signs of dramatic cut-backs in production. "One explanation is that so many producers have the advantage of protected prices, either in their domestic market or through special arrangements such as the US sugar quota, ACP and Comecon trade, that marginal costing enables them to continue to supply sugar to the world market. Of the larger producers only two sell more than 50% of their production at world market prices, a further five countries sell between 40 and 50% and the remaining majority less than 40% at world market prices".

It has been pointed out<sup>4</sup> that the root of the problem is that consumers simply have no need to build-up their stock levels and therefore live from hand to mouth. Since demand holds the key to sugar price prospects in the medium-to-long term they are in a strong position to dictate what they are paying. Large-scale buying has not been a feature of the sugar market for some considerable time and, with all the signs pointing downwards, consumers run little risk of being caught out with poor stocks should prices suddenly rise.

### US sugar import quota

The sugar imports quota for the fiscal year October 1983/September 1984 was originally set by the US Dept. of Agriculture at 2.95 million short tons, raw value. Subsequently, however, it has been urged that the quota be raised because of lower domestic sugar production (the mainland cane areas were badly affected by a severe frost at Christmas) and prices had risen on the New York market to above the 21.17 cents/lb stabilization price. Some officials argued that US domestic consumption was likely to fall in the period owing to increased use of HFS, while others considered that an increase of 200-300,000 tons in the quota would help the developing countries who supply sugar under the quota.

An inter-agency meeting took place in Washington on March 15 and it was announced that the overall quota would be raised by 100,000 tons. Of this amount 8000 was awarded to Malawi and the balance was shared among the other suppliers in proportion to their shares of the original quota (with the exception of Nicaragua, which was excluded from a share in the increase). Because the smallest suppliers had a minimum quota of 16,500 tons, the total supplies under the quota

reached 3,075,150 tons rather than 2,950,000 tons, and with the increases, the new overall supplies reach 3,175,150 tons. The quota, calculated by C. Czarnikow Ltd.<sup>5</sup>, appear below:

	Share	Quotas at 2.95 m short tons, raw value	Increase of 0.1 m	Quotas at 3.05 m
Dominican Republic	17.6	519,200	16,192	535,392
Brazil	14.5	427,750	13,340	441,090
Philippines	13.5	398,250	12,420	410,670
Australia	8.3	244,850	7,636	252,486
Guatemala	4.8	141,600	4,416	146,016
Argentina	4.3	126,850	3,956	130,806
Peru	4.1	120,950	3,772	124,722
Panama	2.9	85,550	2,668	88,218
El Salvador	2.6	86,771	2,392	89,163
Colombia	2.4	70,800	2,208	73,008
South Africa	2.3	67,850	2,116	69,966
Nicaragua	2.1	6,000	—	6,000
Swaziland	1.6	47,200	1,472	48,672
Costa Rica	1.5	61,035	1,380	62,415
Thailand	1.4	41,300	1,288	42,588
Mozambique	1.3	38,350	1,196	39,546
Taiwan	1.2	35,400	1,104	36,504
Guyana	1.2	35,400	1,104	36,504
Zimbabwe	1.2	35,400	1,104	36,504
Mauritius	1.1	32,450	1,012	33,462
Ecuador	1.1	32,450	1,012	33,462
Jamaica	1.1	32,450	1,012	33,462
Belize	1.1	32,450	1,012	33,462
Canada	1.1	32,450	1,012	33,462
Honduras	1.0	58,594	920	59,514
Bolivia	0.8	23,600	736	24,336
India	0.8	23,600	736	24,336
Barbados	0.7	20,650	644	21,294
Fiji	0.7	20,650	644	21,294
Malawi	0.7	20,650	644	21,294
Trinidad & Tobago	0.7	20,650	644	21,294
St. Kitts (a)	0.3	16,500	276	16,776
Malagasy (a)	0.3	16,500	276	16,776
Haiti (a)	0.3	16,500	276	16,776
Paraguay (a)	0.3	16,500	276	16,776
Mexico (a)	0.3	16,500	276	16,776
Ivory Coast (a)	0.3	16,500	276	16,776
Uruguay (a)	0.3	16,500	276	16,776
Congo (a)	0.3	16,500	276	16,776
Specialities	—	2,000	—	2,000
	102.1	3,075,150	100,000	3,175,150

(a) These countries are entitled to a minimum quota of 16,500 short tons, raw value, regardless of the amount derived from calculating their proportionate share from the 0.3 shown.

(b) Consists of an outright award of 8,000 tons in addition to a proportionate share in the remaining 92,000 tons.

### World sugar production, 1983/84

Sugar production jumped sharply between 1980/81 and 1981/82 from 87.7 to 100.4 million tonnes and remained over 100 million tonnes in 1982/83. Bad weather in Europe and a number of cane-growing countries brought about a reduction in 1983/84 and F. O. Licht GmbH originally forecast in October 1983 an output of less than 95 million tonnes. This was later raised to 95.1 million tonnes and a new estimate has now been published<sup>6</sup> which totals 96.2 million tonnes.

By the time of the second estimate in January last, most of the information on beet sugar production was reasonably definite and the current figure is only about 200,000 tonnes higher. The cane sugar sector has been increased by 800,000 tonnes, however, to 60,688,000 tonnes and results from larger crops than previously expected in Australia, Brazil, China, Cuba and Thailand, partly offset by lower crops in India, the Philippines and South Africa.

<sup>3</sup> *The Sugar Situation*, 1984, (396).

<sup>4</sup> *Public Ledger's Commodity Week*, April 14, 1984.

<sup>5</sup> *Sugar Review*, 1984, (1693), 56.

<sup>6</sup> *International Sugar Rpt.*, 1984, 116, 207-214.



# Experiences with DDS vertical crystallizers

By P. KLEIN\*

In the mid-seventies the production capacity of some of the DDS sugar factories was increased substantially, especially in the case of Assens, where the daily slicing rate was increased from approx. 6000 tonnes in 1974/75 to approx. 9000 tonnes in 1978/79. This increase in capacity involved extension of the beet silo, installation of an additional beet washer and beet elevator, increase of the diffusion capacity through installation of an additional 3000 tonnes/day DDS diffuser, and increase of the capacity of the juice purification, evaporation and pan station.

However, the consequent increased quantity of after-product massecuite could not be sufficiently exhausted in the existing horizontal cooling crystallizers of conventional design because of their insufficient cooling surface.

The DDS-Engineering Division in Copenhagen, in collaboration with the DDS Technological Research Laboratories in Nakskov, set out to solve this problem. Through a thorough study of relevant literature and subsequent calculations combined with pilot-scale trials, the influence of the technological parameters on after-product sugar recovery was investigated.

These initial investigations led to the conclusion that the most effective exhaustion of the massecuite is obtained by rapid cooling — over approx. 24 hours, with an exponential drop in temperature — from the discharge temperature of the pans of approx. 80°C to 40°C. Almost the same result is obtained with an exponential drop from 80°C to 60°C, followed by a linear drop to approx. 40°C. The pilot-scale trials showed that it is possible to cool the massecuite at a rate of 10°C/hr without deterioration of the quality of the massecuite, i.e. without formation of fine grain.

Concurrently with the investigations concerning the design of a new vertical cooling crystallizer, we carried out similar investigations for the experimental demonstration of the fundamental principles of a new type of vertical massecuite reheater.

The conclusion of the investigations was that, owing to the fact that heat is transmitted by conduction in media of high viscosity and with laminar flow, it is

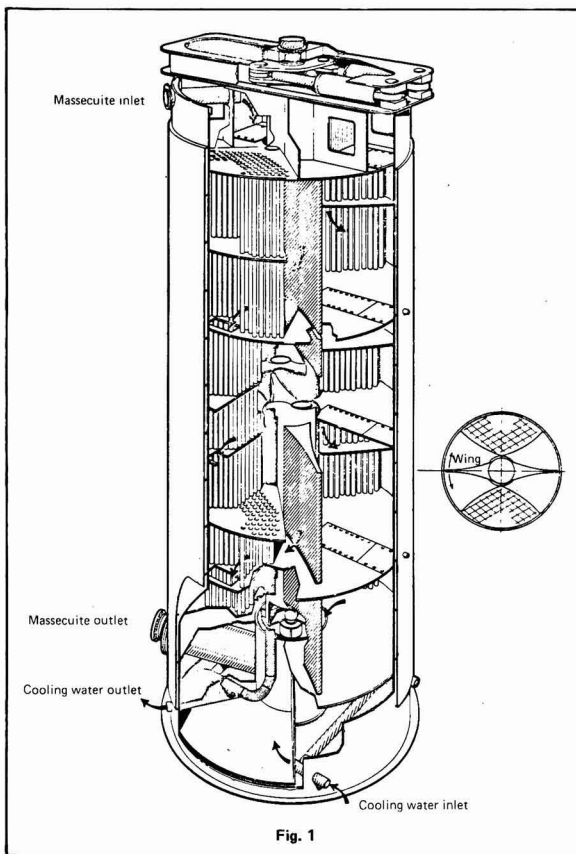


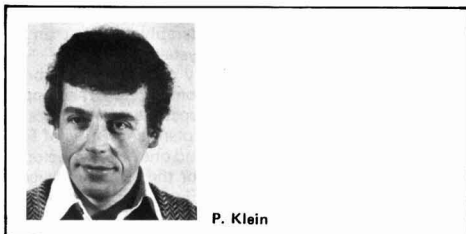
Fig. 1

possible to heat the massecuite without any substantial increase of purity in the mother liquor by using relatively thin massecuite layers and a relatively large heating surface with a small temperature difference between heating medium and massecuite.

On the basis of the above investigations the first DDS vertical crystallizer was installed at Assens for the 1977 campaign — Appendix 1 gives a general description of this DDS vertical crystallizer. At the same time a massecuite heater of the new design was also installed on a factory scale with the purpose of confirming the experimental results for massecuite heating. Figs. 1 and 2 show section and flow in the cooling crystallizer and Fig. 3 shows the design of the massecuite heater.

The first DDS vertical crystallizer plant installed consisted of one cooling crystallizer and one massecuite heater and was connected in parallel with the existing plant to treat approximately one-third of the massecuite, i.e. 8-10 tonnes/hr. Fig. 4 shows the flow diagram.

After minor adjustments, the cooling crystallizer



P. Klein

Paper presented to 17th Gen. Assembly C.I.T.S., 1983.

\* DDS-Engineering Division.

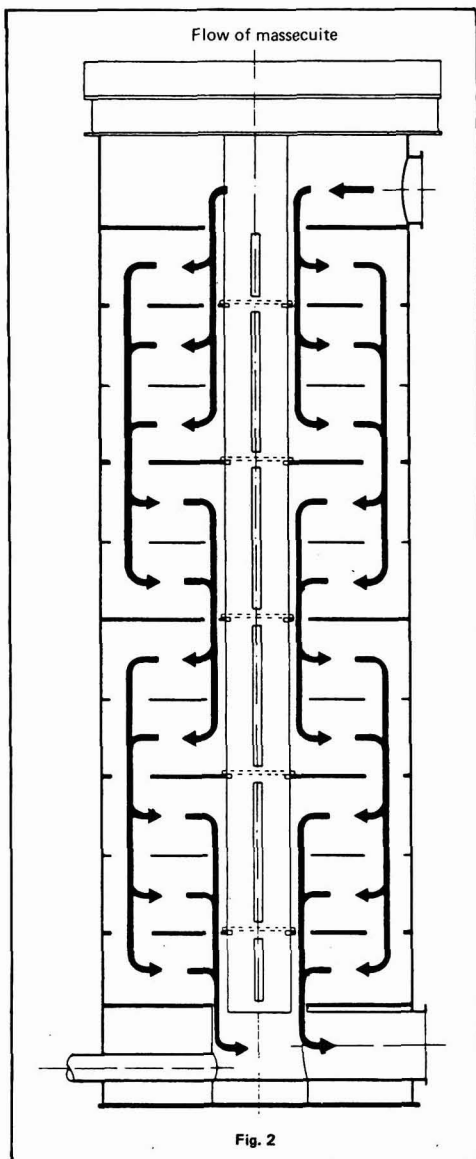


Fig. 2

of this plant certainly fulfilled expectations as regards cooling capacity and massecuite quality, whereas the massecuite heater caused too high an increase in purity.

For the 1978 campaign the new cooling crystallizer was connected in series with the existing crystallizer plant, as shown in Fig. 5, in order to test the crystallizer with full flow, approx. 30 tonnes/hr. The vertical massecuite heater installed in 1977 was dismantled and new investigations and experiments were carried out at our Technological Research Laboratories with a view to developing an improved type of massecuite heater.

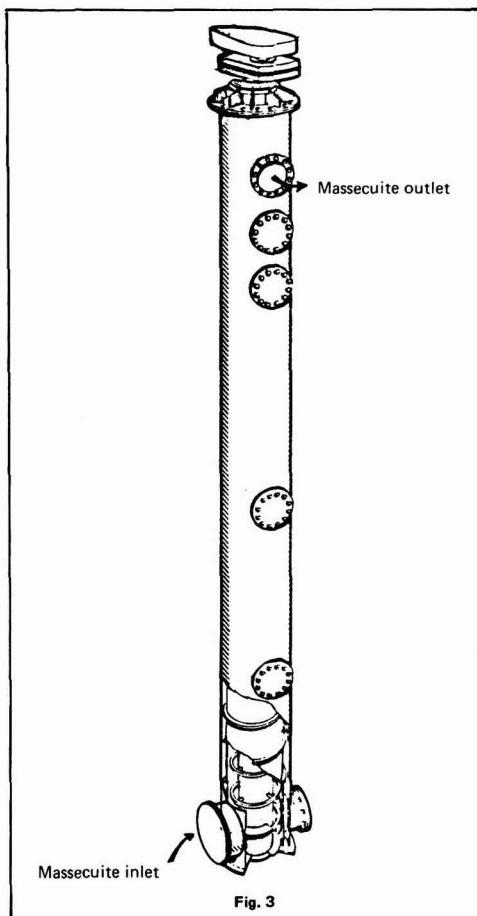


Fig. 3

During the 1978 campaign the operation results of the plant connected in series were good as regards exhaustion of the massecuite, the crystallizer cooling the massecuite from the pans in a short retention time of approx. 4 hours, from approx. 80°C to approx. 60°C at the point of entry to the old crystallizer plant. In approx. 15 hours in the old crystallizer plant the massecuite was then cooled to 42°C — in view of the drives of the old crystallizers — and then finally reheated in the existing heater to 54-56°C before being centrifugalled.

As mentioned before, the operation results of this plant were good, and it was in fact concluded that it is a sensible solution to install the DDS cooling crystallizer as pre-cooler to an existing crystallizer plant whose capacity has gradually proved inadequate. This is because the investment required is considerably smaller than the investment in a new, complete crystallizer plant.

However, as it was necessary for DDS to obtain experience in operation with a complete plant equipped with vertical crystallizers and as space was getting scarce at Assens, a complete crystallizer plant consisting of four DDS vertical cooling crystallizers and one newly-developed massecuite reheater was erected for the 1979 campaign.

This new plant, as shown in Fig. 6, consisted of two DDS cooling crystallizers, type A, i.e. for cooling the massecuite from approx. 80°C to approx. 55°C, each

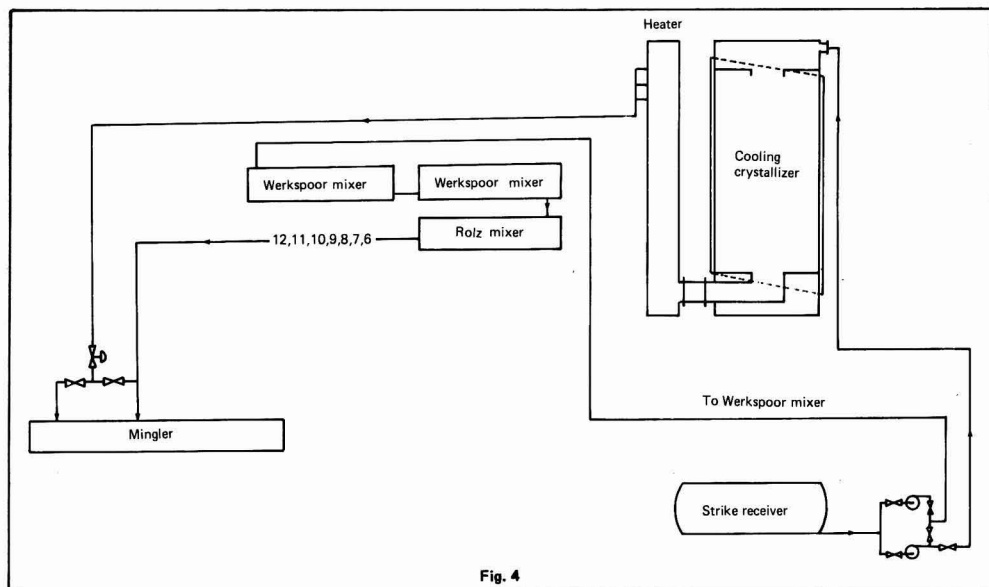


Fig. 4

with tubes of 356 m<sup>2</sup> cooling surface, and two DDS cooling crystallizers, type B, for cooling in the high-viscosity range from approx. 55°C to approx. 38°C, each with a cooling surface of 252 m<sup>2</sup>.

Experience with this plant showed that, as desired, the cooling was effected through an exponential drop in temperature, from 85°C to approx. 40°C, with a consequent very satisfactory exhaustion of the massecuite.

The retention time in the crystallizer system was in this case 18-20 hours. Unfortunately, the results of the

new massecuite heater were not entirely satisfactory, as it caused an increase in purity in the mother liquor of 0.5-1 purity unit with heating from approx. 40°C to 54-56°C. Experiments were therefore conducted on heating of massecuite in the last of the DDS crystallizers, and the values obtained showed that it was possible to produce the necessary heating with no purity increase whatsoever in the mother liquor.

In our opinion, this is due to the fact that the cooling crystallizer had a larger heating surface than the tested

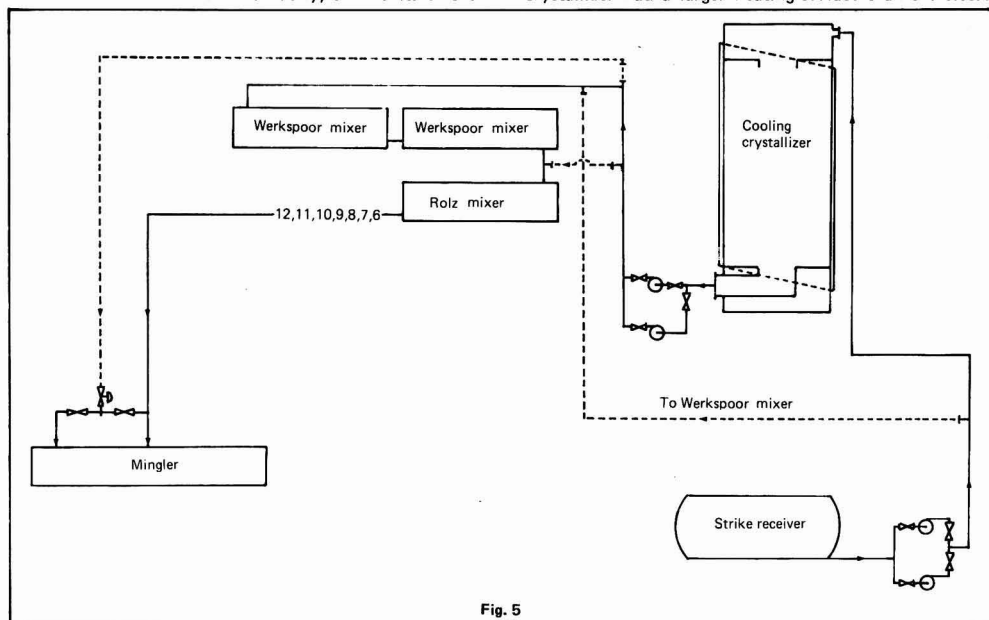


Fig. 5



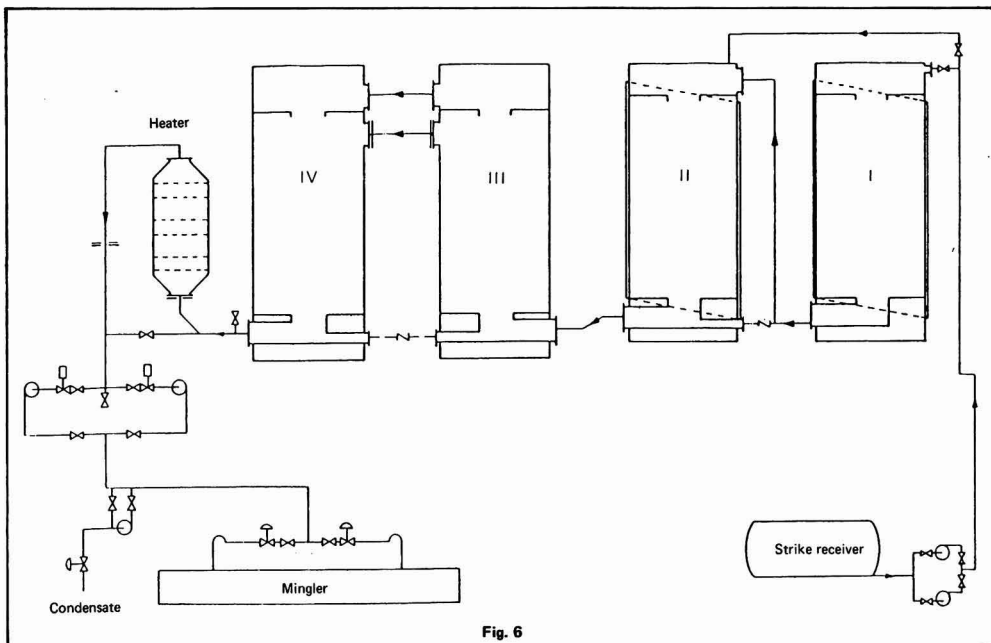


Fig. 6

massecuite heater and that the design of the crystallizer, with its effective contact between massecuite and cooling tubes, permitted a high heat transfer rate. Consequently, it became possible to heat the massecuite by means of a medium with a temperature only a few degrees higher than that of the surrounding massecuite and so avoid local under-saturation. Therefore, for the 1982 campaign, an additional DDS crystallizer of type B was erected for heating of the massecuite before centrifugalling. Fig. 7

shows the plant as it is today.

In order to ensure reliable operation also in the case of unusually high massecuite viscosity, pump stations have been mounted between crystallizer No. 3 and 4 as well as between crystallizer No. 5 and the centrifugal station. As the experience gained during the 1982 campaign did not involve any modifications of the plant, we now regard the development and extension of the crystallizer plant in Assens as completed.

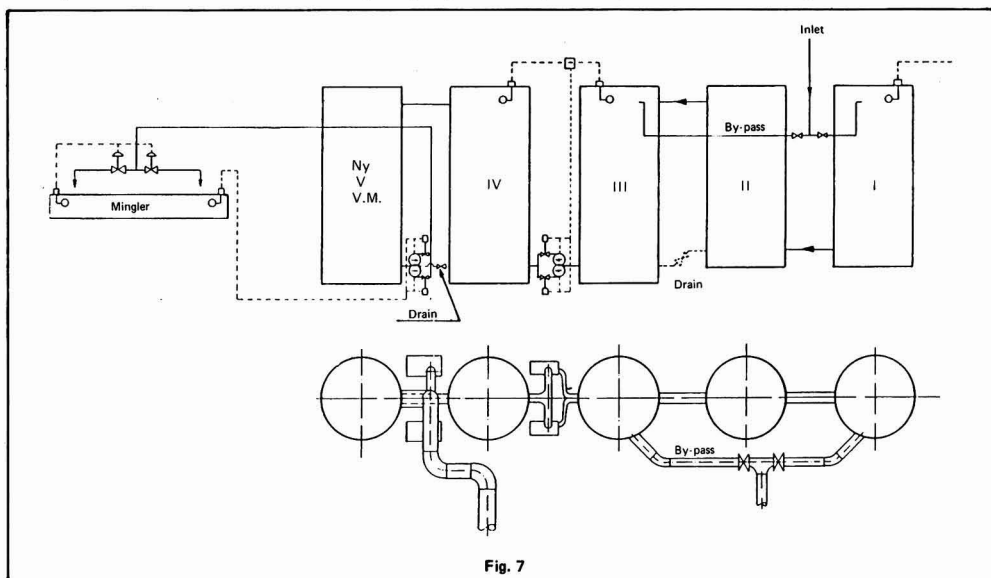
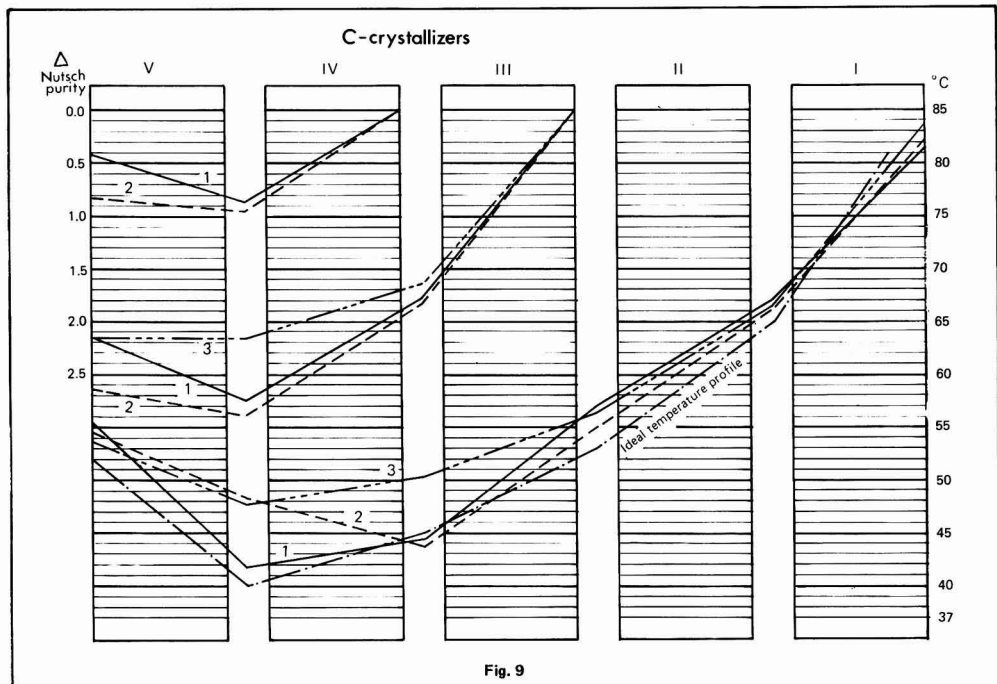
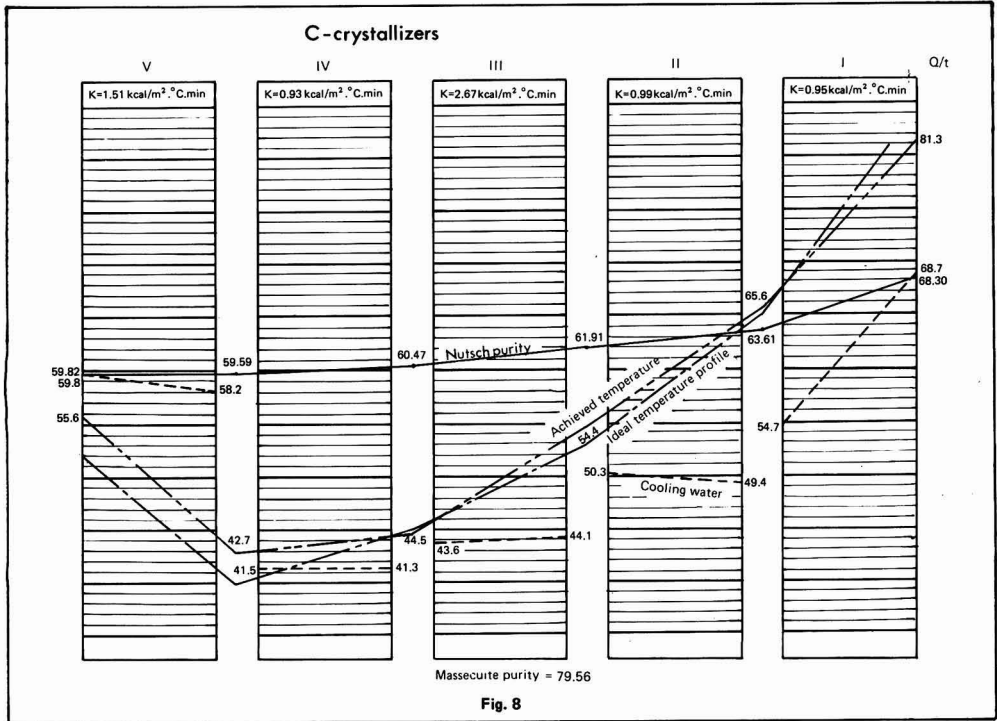
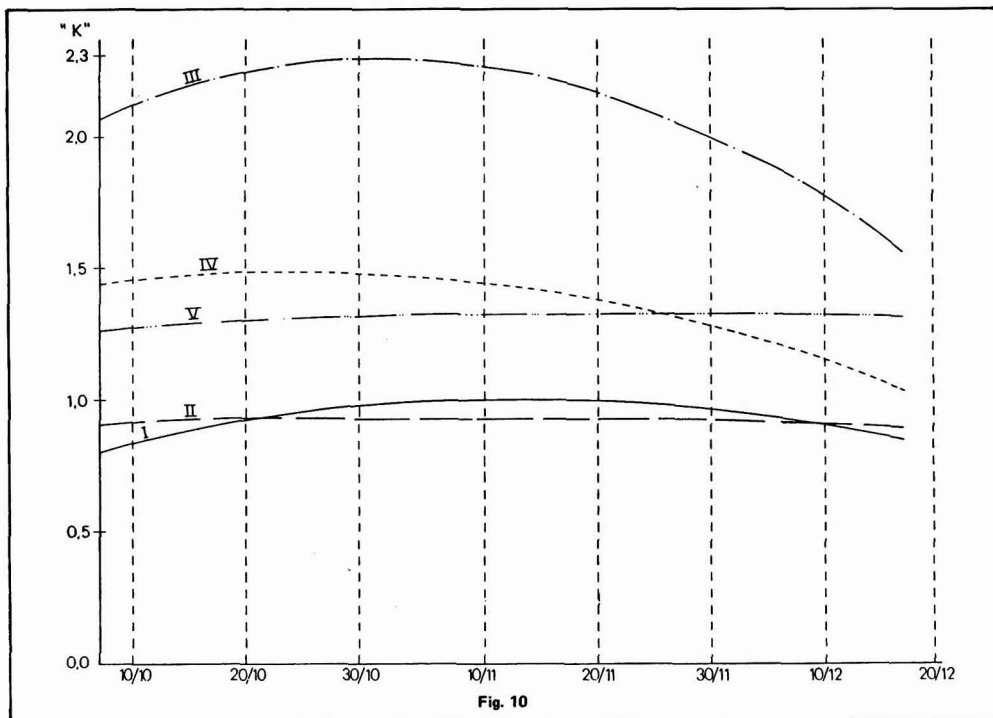


Fig. 7



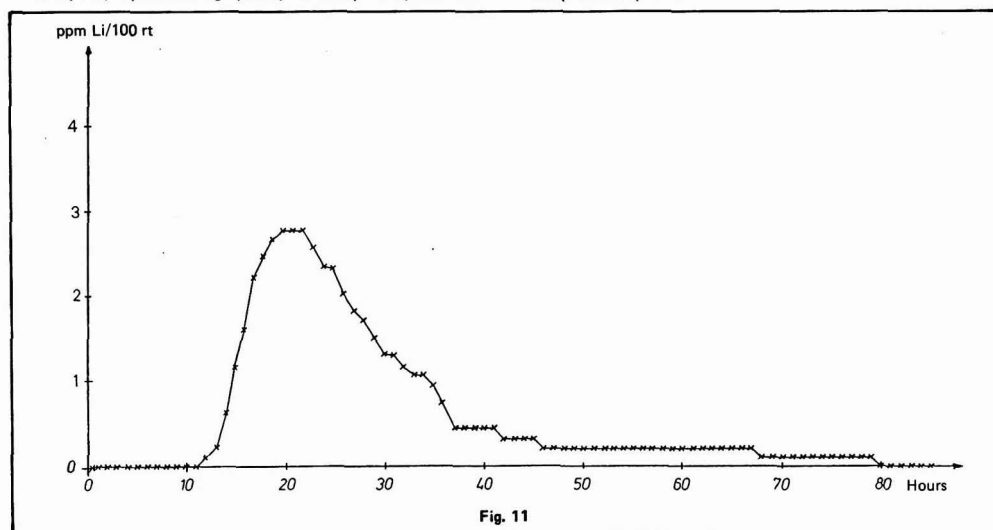


During the 1982 campaign a heavy measuring program was carried through in connexion with the crystallizer plant, and some of the values recorded, illustrative of the good results obtained, are worth mentioning, while at the same time the fundamental principle of the plant should be emphasized.

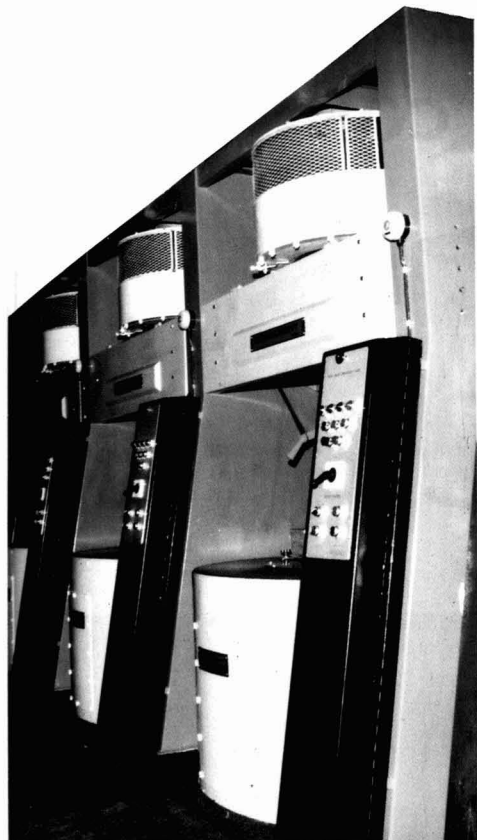
Rapid cooling and short retention time has resulted in a compact, space-saving plant, entirely adapted for

installation outdoors.

Fig. 8 shows the temperature profile in the crystallizer plant for a period of 3 x 24 hours in the campaign 1982 — both the temperature profile aimed at and that obtained. The same diagram shows the purity of mother liquor. It is worth noting that there is a slight increase in purity during heating which led us to try out other temperature profiles.







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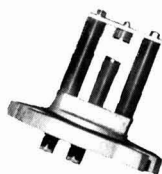
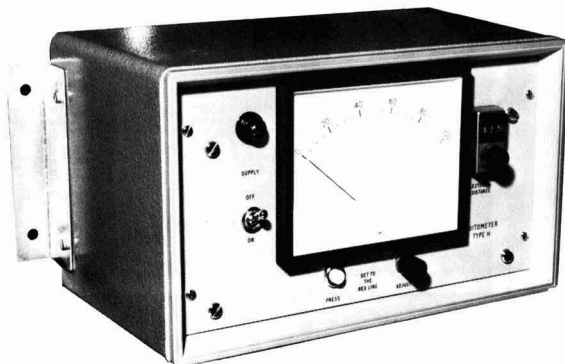


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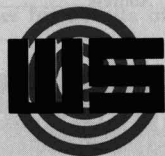
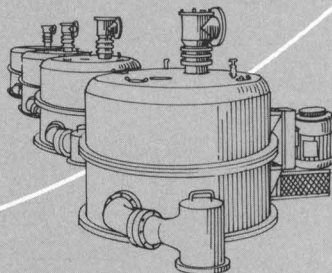
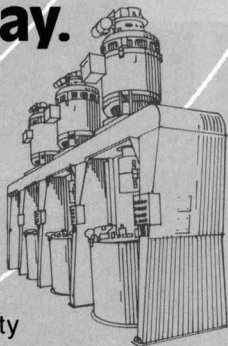
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Fig. 9 makes a comparison between three such temperature profiles, the temperature conditions in crystallizer I and II being unchanged. The result is that the best exhaustion is obtained with the temperature profile, marked 2. In this case the crystallizers I, II and III are used as cooling crystallizers and the crystallizers IV and V as massecuite heaters. It is noteworthy that during heating in crystallizer IV there is still a considerable drop of purity in the mother liquor.

During the 1982 campaign also the heat transfer coefficients for all the crystallizers were recorded as indicated in Fig. 10. The lowest values are found in crystallizers I and II, where the heat transfer coefficient is approx.  $0.8 \text{ kcal/min/}^\circ\text{C/m}^2$ , whereas in crystallizers III and IV the value recorded is  $1.5\text{--}2 \text{ kcal/min/}^\circ\text{C/m}^2$ .

Finally, Fig. 11 shows the result of investigation of the retention time in the entire system by means of lithium chloride. The graph shows that the flow in the crystallizers is approximate plug-flow with a recorded retention time of  $24\frac{1}{2}$  hours, compared with the calculated retention time of 25 hours.

*(Since this paper was presented at the 17th General Assembly of C.I.T.S., the 1983/84 campaign has been concluded and its is reported by Assens sugar factory that, in spite of unfavourable sugar beet condition owing to unseasonable weather prior to lifting, the crystallizers have achieved a performance equal to that of the previous campaign.)*

#### Appendix 1. General description of the DDS vertical crystallizer

The crystallizer is shown in Fig. 1. It is built as a cylindrical, vertical container with a diameter of 3.9 m and a height of 12.9 m. The total massecuite volume is  $100 \text{ m}^3$ . The vertical cooling tubes, arranged diametrically opposite each other in two separate sections, are 10 m in length and 50 mm in diameter.

A lower and an upper tube plate at a distance of 10 m form the limits for the massecuite volume. This volume is divided by supporting plates and intermediate tube plates into a number of sections. The plates secure a controlled flow of massecuite through the crystallizer. For further support of the tubes, a set of plates are built into each section.

An agitator wing, activated by two heavy hydraulic cylinders fixed to the top frame, oscillates through  $\pm 52.5^\circ$  in the massecuite compartment between the two cooling sections. The agitator is located in the top frame in a combined thrust and radial bearing and supported by a radial bearing in the bottom. The agitator wing goes all along the axis of the crystallizer, only broken at the baffle plates. This gives a very efficient stirring effect because the wing positively displaces the massecuite along the outer surface of each tube in the tube bundles.

Below the lower tube plate, two distribution reservoirs for the cooling water to the two cooling sections are built in. Corresponding reservoirs are built into the top. The crystallizer has a separate recycling cooling water system with a flow of approx.  $120 \text{ m}^3/\text{hr}$  through the tubes. In order to keep the recycling water at the desired inlet temperature, the system is supplied with make-up water from a common cooling water system and the excess water is removed from the recycling system through overflow in the top back to the common system.

#### Summary

After a thorough study of relevant literature and after having considered mathematically the influence of technological parameters on the after-product sugar recovery,

DDS designed and tested a new type of vertical crystallizer, and a full-scale plant was installed at the Assens factory. The vertical crystallizer was installed for both cooling and heating after-product massecuite and it has met expectations for a crystallizer plant, characterized by large heating/cooling surfaces and short retention time, giving satisfactory and effective exhaustion of the massecuite.

#### Expérience avec les malaxeurs verticaux DDS

Après une étude approfondie de la littérature sur le sujet et après un examen mathématique de l'influence des paramètres technologiques sur l'épuisement des arrière-produits, la DDS a conçu et mis à l'essai un nouveau type de cristalliseur vertical et monté une installation en vraie grandeur à la Sucrerie d'Assens. Le cristalliseur vertical a été prévu à la fois pour le refroidissement et le réchauffage des masses cuites arrière-produits et a répondu complètement à l'attente d'une installation de malaxage caractérisée par de grandes surfaces de chauffe et de refroidissement, avec un temps de séjour court, tout en assurant un épuisement de la masse cuite efficace et satisfaisant.

#### Erfahrungen mit vertikalen DDS-Kühlmaischen

Nach eingehendem Studium der relevanten Literatur und mathematischer Berücksichtigung des Einflusses der zuckertechnologischen Parameter auf die Nachprodukt-ausbeute hat die DDS eine neue vertikale Kühlmaische entwickelt und getestet. Eine komplette Anlage wurde in der Fabrik Assens installiert. Die vertikale Kühlmaische wurde zum Kühlen und Anwärmen der Nachprodukt-Füllmasse installiert und erfüllte alle Anforderungen an eine Kühlungs-Kristallisationsanlage, die charakterisiert ist durch große Heiz-/Kühlflächen und kurze Retentionszeit, bei zufriedenstellender und effektiver Erschöpfung der Füllmasse.

#### Experiencia con cristalizadores verticales DDS

Después de un estudio minucioso de la literatura relevante y después de un examen matemático de la influencia de parámetros tecnológicos sobre el rendimiento de azúcar de recuperación, la sociedad DDS ha diseñado y experimentado con un nuevo tipo de cristallizador vertical, y ha hecho una instalación de escala comercial en la fábrica de Assens. El cristallizador vertical se ha instalado para enfriamiento tanto como recalentamiento de la masa cocida de recuperación, y ha cumplido con las expectativas de un instalación de cristalización caracterizada por grandes superficies de enfriamiento/recalentamiento y corto tiempo de retención, logrando un agotamiento satisfactorio y efectivo de la masa cocida.

#### Fiji sugar exports, 1983<sup>1</sup>

	1983	1982
	tonnes, raw value	
Canada	0	16,936
China	44,082	43,708
EEC	181,032	168,961
Japan	0	14,092
Malaysia	53,068	36,547
New Zealand	23,177	71,763
Singapore	24,792	30,972
US	18,987	31,746
	345,138	414,725

<sup>1</sup> I.S.O. Stat. Bull., 1984, 43, (2), 17.



# Cane sugar invert analysis by HPLC utilizing a post-column derivatization reaction

By MARK WNUKOWSKI

(Operations Laboratory, American Sugar Division,  
Amstar Corporation, New York)

## Introduction

The loss of sucrose through chemical inversion and decomposition reactions has always been of primary concern to the cane sugar refiner. A pathway of this chemical destruction of sucrose can be traced from the time of harvest and throughout the entire processing operation. In the refining process alone, loss due to sucrose decomposition has been estimated to be as high as 0.5%<sup>1</sup>.

In addition to the direct loss of sucrose, the resulting decomposition products play havoc in the refinery by increasing the load on the recovery house, and the amount of molasses formed, and therefore the amount of sucrose lost to the molasses<sup>1</sup>.

In general, there are two major classifications of these sucrose chemical decomposition reactions: (a) those occurring in an acid solution and (b) those occurring in a basic or alkaline solution. The decomposition reactions of sucrose in an acid matrix or inversion are the ones by far most familiar to and scrutinized by the sugar refiner.

In the sugar industry, the terms inversion and invert sugars are generally applied to the decomposition of sucrose in an acid solution and its products. It should be noted that dextrose and levulose can and do enter into a series of reactions to form other non-sugar compounds<sup>1</sup>.

It is normal practice in a cane sugar refinery laboratory to carry out much invert sugar analysis of the refinery process streams and products. The data generated from these tests are important because they provide the refiner with information essential in determining the refining quality of raw sugars (invert to ash ratios), and the efficiency of his process in regard to sucrose losses through inversion (invert and invert to chloride ratios)<sup>2</sup>.

To date the most widely used and accepted method of reducing sugar analysis is by a copper reducing titration. The following four methods: (1) the Lane & Eynon method, (2) the Berlin Institute method, (3) the Öfner method, and (4) the Knight & Allen method, are all based upon the reduction of the copper (II) complex with tartaric acid in alkaline solutions which differ in composition, especially in their varying degrees of alkalinity<sup>3</sup>.

These methods present several problems, the major one being that they analyse for all reducing substances in solution, and not specifically for dextrose and levulose, so that one value is obtained which represents all reducing substances. Another problem is that invert sugars undergo further reactions quickly and may not be accounted for by a copper titration analysis<sup>1</sup>.

Additionally, these methods do not provide any information as to the nature of the ratio of dextrose to levulose concentration. Knowledge of this ratio is important, for a high D/L ratio in a cane sugar sample will give a false sugar content when determined by polarimetry.

Enzymatic reactions catalysed by hexokinase can be specific for the determination of dextrose and levulose<sup>4</sup>; however they are costly and too time-consuming to be performed on a routine basis.

Gas-liquid chromatographic methods requiring prior sample derivatization have been tried<sup>5</sup>; however, considerations of cost and/or time per sample have deferred acceptance of these methods by the cane sugar industries.

Today, high-performance liquid chromatography offers an alternative to these methods. High efficiency packings, sensitive detectors, and precise solvent delivery systems afford this technique a new level of efficiency. Multiple sugars can now be identified and quantitatively measured in a single analysis.

This paper represents part of the findings of our investigation into two different HPLC systems and their applicability to invert analysis in a cane sugar refinery. System I employed the use of a strong cation exchange column, a water mobile phase and a refractive index detector. System II, a modified version of a technique reported by Boykins & Liu<sup>6</sup>, involved a normal phase column, an absorbance detector and a post-column derivatization reaction.

## Principles of the HPLC systems evaluated

**System I:** This chromatographic system utilizes what now seems to be the preferred method of mono-, di- and trisaccharide separation<sup>7</sup>, namely an isocratic elution mode is used with water in conjunction with a column packed with cation exchange resin in the calcium form, and a refractive index detector. The column is operated at an elevated temperature, approximately 90°C, controlled by a thermostated heater compartment, to facilitate shorter retention times and better resolution of the chromatographic peaks. A small amount of calcium propionate is added to the mobile phase to inhibit stripping of the Ca<sup>++</sup> from the resin by exchange with hydrogen ions. Separations of sucrose, dextrose and levulose can be achieved in one analysis in about fifteen minutes on this system. Applicability and use of this system can be traced to Scobell *et al.*<sup>8</sup> and Wong-Chong & Martin<sup>9,10</sup> in the corn and cane sugar industries, respectively.

**System II:** This system is a modified version of the one reported by Boykins & Liu<sup>6</sup> and more recently Wright & Van Niekerk<sup>11,12</sup>, employing a post-column derivatization reaction with tetrazolium blue.

## Paper presented to Sugar Industry Technologists, 1983.

<sup>1</sup> Clarke *et al.*: *Tech. Rpt. Cane Sugar Refining Research Project*, 1978, (43).

<sup>2</sup> Meade-Chen: "Cane Sugar Handbook", 10th edn. (Wiley, New York), 1977.

<sup>3</sup> "Sugar Analysis ICUMSA Methods", Ed. Schneider, (ICUMSA, Peterborough), 1979.

<sup>4</sup> "Enzymatic Analysis for Food Chemistry", Technical Brochure (Boehringer, Mannheim).

<sup>5</sup> Clarke & Brannan: *Tech. Rpt. Cane Sugar Refining Research Project*, 1979, (45).

<sup>6</sup> "Reverse-Phase Chromatography of Neutral Sugars and Disaccharides", (Div. of Bacterial Products, Bureau of Biologies, Food & Drug Administration, USA).

<sup>7</sup> Ivie: *Sugar y Azúcar*, 1982, 77, (2), 44-53.

<sup>8</sup> *Cereal Chem.*, 1977, 54, 905-917.

<sup>9</sup> *J. Agric. Food Chem.*, 1979, 27, 929-932.

<sup>10</sup> *Proc. 17th Congr. ISSCT*, 1980, 2373-2388.

<sup>11</sup> *Food Chem.*, 1983, 10, 211-224.

<sup>12</sup> *J. Agric. Food Chem.*, 1983, 31, 282-285.

The system consists of a normal-phase Micro-Bondapak-NH<sub>2</sub> column, a polar mobile phase (80:20 acetonitrile:water) and an absorbance detector.

Chromatographic systems for the separation of carbohydrates and sugars using columns packed with porous micro particles derivatized with amino groups have been in use for some time<sup>13,14</sup>. However, the unique portion of this system is the use of the post-column derivatizing reagent tetrazolium blue to increase the sensitivity and the detection limits of the separated compounds.

Tetrazolium blue reacts specifically in the presence of reducing agents to yield a dark blue diformazan pigment<sup>14</sup>. By utilizing an additional pump to the solvent delivery system, this reagent can be added to the mobile phase upon exit from the chromatographic column. The reagent can then combine with the separated reducing components (sugars) by means of a network of mixing and reaction coils maintained at the temperature of 90°C to ensure the reaction reaches completion. The derivatized sugars then enter the absorbance detector with their newly acquired sensitivity for detection.

Separations of invert sugar (dextrose and levulose) can be accomplished in one analysis in fifteen to twenty minutes with this equipment, depending upon the polarity of the solvent. Sucrose, however, having no reducing group, is not derivatized and therefore remains undetected.

## Materials and methods

### Apparatus

**System I:** (1) Waters Associates' Sugar Analyser I liquid chromatograph model SA1 equipped with (a) a model 401 refractive index detector, (b) a M45 solvent delivery system with reference valve, (c) Eldex column heater, (2) a model 710B Wisp Waters automatic sampler, (3) a model 730 Waters data module with LC calculation capability, and (4) a Bio-Rad Aminex Carbohydrate HPX-87 column of 300 mm x 7.8 mm.

**System II:** In addition to the instrumentation mentioned for System I, the following pieces of equipment were added: (1) a model 441 Waters absorbance detector, (2) a model A-30-S post-column reaction pump, (3) a Waters NH<sub>2</sub> Micro-Bondapak column, and (4) two lengths of stainless steel reaction/mixing coil supplied by Waters Associates.

### Solvents and reagents

**System I:** Water originating from a Millipore Milli — R/Q water purification system, containing 0.02 g/l added (CH<sub>3</sub>CH<sub>2</sub>COO)<sub>2</sub>Ca, was the only solvent used. The water was filtered using a pyrex filter holder assembly fitted with 0.45 micron pore size membrane type HA, and degassed by stirring under vacuum. The invert sugar standards (dextrose and levulose) were prepared from J. T. Baker Analysed Reagents. All standards and samples were diluted to working concentrations with Milli — R/Q purified water. Filtration of the standards and cane sugar samples was carried out using Waters Associates' sample clarification kit (0.45 micron pore size, aqueous solvent filtration disc).

**System II:** Acetonitrile and water from a Milli — R/Q system were separately filtered with an appropriate type Millipore 0.45 micron pore size filter fitted in a pyrex filter holder assembly. The acetonitrile and water were then mixed to the desired 80:20 ratio, and degassed by stirring under vacuum. The tetrazolium blue derivatizing reagent (Sigma Chemical Co.) was prepared by dissolving 2 grams of the reagent and diluting to one litre with 0.1 N NaOH, and filtering the solution through a 0.45

micron filter. The invert sugar standards were again prepared from J. T. Baker Analysed Reagents. The standards were diluted to volume as above with Milli — R/Q purified water.

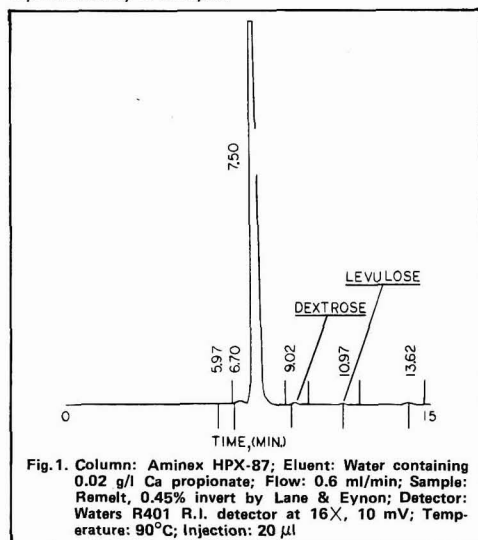
All standards and cane sugar samples were additionally filtered through a Waters Associates' Sep Pak C<sub>18</sub> cartridge before injection into the LC system.

## Results and discussions

In view of the large amount of literature published on the subject recently, the first chromatographic system to be investigated for possible application to invert analysis in our cane sugar refineries was System I employing the strong cation exchange resin column. Although this system has the obvious advantage of being able to separate sucrose as well as dextrose and levulose in a single analysis, its major inadequacy became evident rather quickly.

The detection limits of the refractive index detector used in this system for dextrose and levulose are considerably lower than those needed to measure invert sugar quantitatively in most of our process samples. These samples consist mainly of those high purity streams and products which contain invert sugar of the order of less than 1.0% as measured by the conventional Lane & Eynon method.

A prime example of this shortcoming is illustrated by the chromatogram, shown in Figure 1, of a 2.0% solution (w/v) of remelt liquor containing 0.45% invert as determined by the Lane & Eynon method. Note the low magnitude of the recorded detector signal for the dextrose and levulose peaks by comparison with that of sucrose. This obviously poor sensitivity for detection of invert sugar invariably leads to poor precision and reproducibility of analysis.

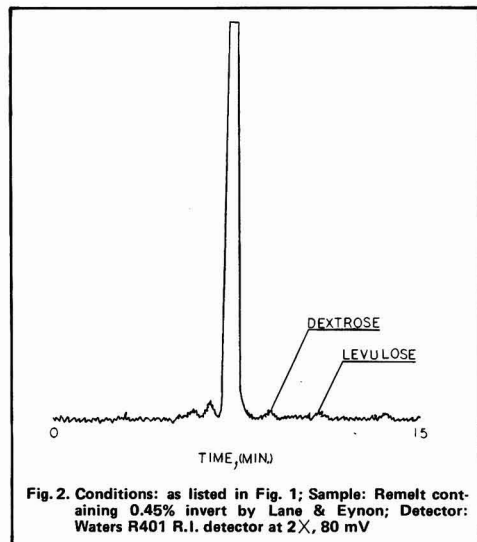


A further illustration of the severity of this problem is depicted in Figure 2. The chromatogram shown is exactly the same as that in Figure 1, only run at an increased detector sensitivity setting of 2X, 80 mV,

<sup>13</sup> Yang *et al.*: *J. Chromatography*, 1981, (209), 316-322.

<sup>14</sup> Ghernati *et al.*: *J. Liquid Chromatography*, 1982, 5, 1725-1748.

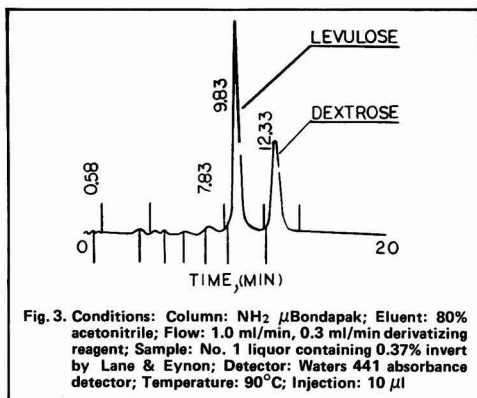
by virtue of the Waters refractive index signal splitter supplied with the HPLC instrumentation. Even at this increased signal magnification the eluted peaks for dextrose and levulose are barely visible on the recorded chromatogram. In addition, the detected signals for the eluted invert sugar peaks are approaching the noise level of the instrumentation.



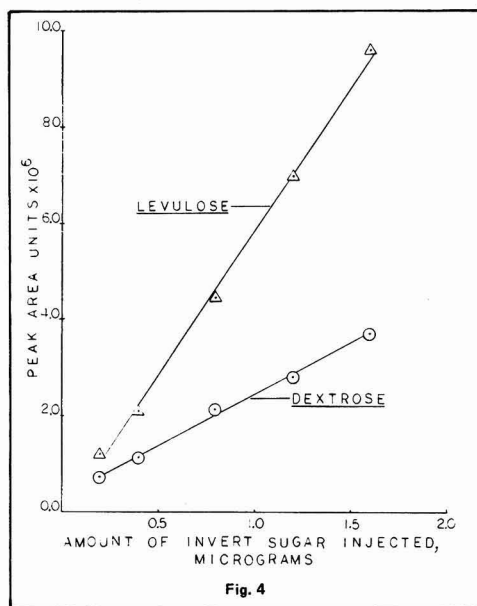
Since the detection limit is usually defined as the minimum sample size needed to produce a signal equal to twice the short-term noise<sup>15</sup>, only an increased sample concentration could possibly rectify this problem. However, increasing the sample size injected on to the cation exchange column led only to a shorter column lifetime and a decrease in overall resolution owing to column overload and the disparity in the concentration of sucrose *versus* that of invert sugar.

Because of the difficulties encountered with the cation exchange column (System I), the alternative chromatographic System II, employing a post-column derivatization reaction with tetrazolium blue, was suggested by the manufacturer as a possible solution. Favourable results with good separations and increased sensitivity were obtained immediately. Illustrated in Figure 3 is a chromatogram of a 3.0% solution (w/v) of a liquor similar in sucrose and invert concentrations to that of the sample shown previously in Figs. 1 and 2. The gain in sensitivity of detection for the eluted dextrose and levulose peaks is apparent, by comparison with that of System I. This increased sensitivity is attributed primarily to the derivatization of the invert sugar as well as the substitution of the solute (absorbance) detector for the bulk property (refractive index) detector. Generally speaking, refractive index detectors have a maximum sensitivity of about a few micrograms, whereas absorbance detectors are roughly 1000 times more sensitive giving a detectable signal for a few nanograms of sample<sup>15</sup>.

Encouraged by the preliminary results, the chromatographic System II was further investigated. First a series of dextrose and levulose standards were prepared and analysed using the System II. Plots of the resulting peak areas *versus* invert sugar concentrations, displaying linear



relationships, appear in Figure 4. Linear regression analysis applied to the data gathered in this experiment yielded correlation coefficients of 0.999 and 0.996 for the levulose and dextrose curves respectively. It can be seen from these data that levulose has an apparently higher affinity for the derivatizing reagent tetrazolium blue than has dextrose. The exact reason for this is unknown; however, similar cases illustrating a difference between the reactivity of dextrose and levulose have been previously reported. For example, in terms of the reduction of standard Fehlings solution, dextrose has a higher relative reducing power (1.000) than that of levulose (0.924)<sup>16</sup>.



The next step of this study involved the injection of a series of dextrose and levulose standards into the system, and measuring their relative recoveries. These results are listed in Table I.

<sup>15</sup> "Practical High Performance Liquid Chromatography", Ed. Simpson, (Heyden, London), 1976.

<sup>16</sup> Brown & Zeman: "Sugar Analysis", 3rd edn. (Wiley, London), 1941.

Table I. Recovery of dextrose and levulose standards by HPLC System II

Sugar	Amount injected ( $\mu$ g)	Amount recovered ( $\mu$ g)	% Recovery
Dextrose	0.20	0.25	125
"	0.40	0.45	112
"	0.80	0.87	108
"	1.20	1.13	95
"	1.60	1.53	96
Levulose	0.20	0.25	125
"	0.40	0.44	110
"	0.80	0.90	113
"	1.20	1.29	108
"	1.60	1.90	118

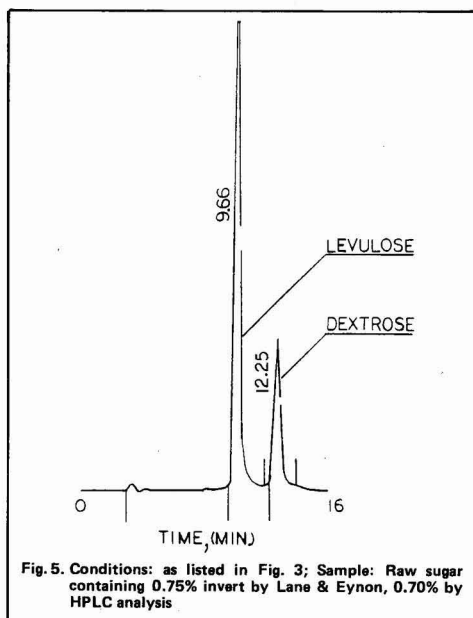
Deviations between the expected and actual recoveries of the sugars are probably due to a combination of inherent method errors in the dilution, weighing, and completeness of the derivatizing reaction. However, since only one standard concentration of 0.80 micrograms of each sugar was used to quantify the range of unknowns, the deviations reported from actual values may be minimized if the samples are re-analysed using external standards equivalent in concentration.

Having reasonably-well ascertained the sensitivity, detection limits and accuracy of System II, it was next applied to the analysis of some selected cane refinery process samples. The invert results determined for these samples chromatographically were then compared with those obtained by the conventional Lane & Eynon titration method. Table II includes the results, from which it will be seen that, as the sample analysed approaches the lower purity portions of the refinery process, the % invert determined by HPLC deviates significantly more (to a lower value) from the conventional Lane & Eynon results.

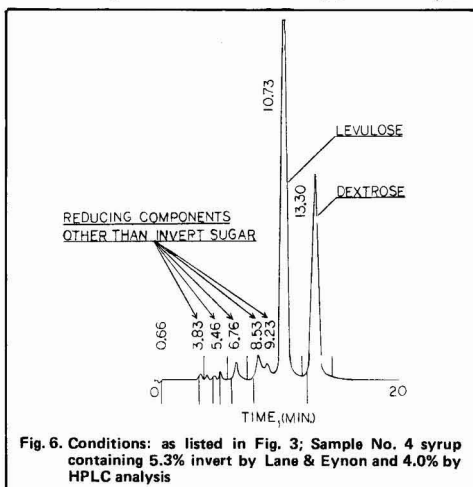
Table II. HPLC analysis of some selected refinery process samples

Sample	% Invert by Lane & Eynon	% Invert by HPLC Test 1	% Invert by HPLC Test 2
Raw sugar-A	0.50	0.42	0.47
Raw sugar-B	0.75	0.77	0.65
W.S.L.-A	0.15	0.14	0.15
W.S.L.-B	0.20	0.19	0.16
W.S.L.-C	0.10	0.11	—
Liquor 1-A	0.10	0.12	—
Liquor 1-B	0.30	0.27	0.27
Liquor 1-C	0.20	0.24	—
Liquor 1-D	0.20	0.23	—
Remelt-A	0.60	0.40	—
Remelt-B	1.15	1.18	0.92
Syrup 1-A	0.70	0.70	0.64
Syrup 1-B	0.55	0.59	—
Syrup 2-A	0.80	0.53	—
Syrup 2-B	1.90	1.55	1.57
Syrup 3-A	3.85	2.97	3.03
Syrup 3-B	2.50	1.96	—
Syrup 4-A	5.50	4.03	—
Soft liquor-A	4.50	3.78	3.84
Soft liquor-B	2.60	1.89	—
Soft Liquor-C	2.80	2.02	—
Low sweet water	3.00	2.04	—

The explanation lies in the chromatograms illustrated in Figures 5 and 6; that in Fig. 5 is of a 10 microlitre



injection of a 4.0% solution (w/v) of a raw sugar sample containing 0.75% invert by Lane & Eynon analysis, which is in good agreement with the 0.70% invert value obtained by HPLC analysis. This chromatogram shows no peaks other than those expected for dextrose and levulose; however, in the chromatogram illustrated in Fig. 6, of a 0.5% solution (w/v) of a syrup 4, the presence of an appreciable number of peaks before the appearance of the levulose can be observed. These peaks represent chemical components other than dextrose and levulose which reacted with the derivatizing reagent tetrazolium blue, and consequently contain reducing groups. Whether these components are present in minute amounts in the raw sugar received by the refiner, or are the result of a series of degradation reactions taking place during the



refining process, has yet to be determined. However, the net effect of the presence of such constituents in the cane sugar refiner's process would be for them to react with the Lane & Eynon reagents so introducing an error into invert sugar measurements.

A further example of these deviations in invert sugar values determined by HPLC from those obtained by Lane & Eynon, can be seen vividly by comparing the results collected during the automated HPLC run of the technical process control samples from one of our cane sugar refineries (Table III). Invert analysis data for a series of four samples beginning with the syrup 1 sample show relatively good agreement between HPLC and the titration method for the first sample. However, as the impurities become successively more concentrated across the multiple crystallizations (syrup 2, syrup 3) the HPLC values tend to be lower than those expected by the Lane & Eynon method to a more pronounced extent. The above explains why HPLC results reported in the literature for invert sugar analysis of cane sugar products tend to be lower than expected, especially for low purity samples.

**Table III. HPLC automated analysis of a refinery's process control samples**

Sample	% Invert by		% Invert by HPLC
	Lane & Eynon	Test 1	Test 2
Affination syrup	6.51	5.32	5.53
Washed sugar liquor	0.24	0.27	0.28
Remelt	0.89	0.95	1.03
Thin liquor 1	0.37	0.42	0.40
Thick liquor 1	0.32	0.28	0.27
Syrup 1	0.70	0.65	0.65
Syrup 2	1.51	1.18	1.17
Syrup 3	3.05	2.42	2.38
Syrup 4	8.53	7.28	6.97
Soft liquor	5.96	5.75	5.87
LSW	20.49	23.92	23.73
Soft liquor to evaporator	5.69	5.21	5.19
Soft liquor from evaporator	5.89	5.30	—
HSW	1.63	1.46	—
Final remelt	6.98	6.30	—
Raw sugar	0.79	0.70	—
Blackstrap molasses	25.92	23.84	—

Finally, in addition to the testing previously mentioned, some attempts were made to determine the reproducibility of analysis and possible degradation of samples over a period of time. As a measure of the reproducibility of analysis and consistency of the derivatization reaction technique used, an experiment was conducted in which three samples of brown sugar of cane origin were chosen and analysed for invert content by HPLC System II on three successive days. Solid samples were chosen in order to minimize any chances of sample deterioration. The results listed in Table IV show good reproducibility and no apparent change in the nature of the derivatization reaction.

In addition, since sample preparation for this analysis requires its dilution to approximately a 0.5 to 6.0% solution, we determined whether a sample would degrade over the period of an automated analysis. A time interval of an automated sample analysis could last 10 to 20 hours, depending on the sample load. In order to resolve the possibility of decomposition, two samples were chosen (1) a raw sugar and (2) a soft liquor. These

**Table IV. HPLC analysis of sugar samples over three consecutive days**

Sample	% Invert by HPLC		
	Day 1	Day 2	Day 3
Brown sugar 1	1.92	1.92	1.96
Brown sugar 2	1.98	2.00	1.95
Brown sugar 3	1.68	1.82	1.76

samples were analysed for % invert by HPLC System II and the same diluted samples re-analysed twenty four hours later. The results appear in Table V. It can be concluded from the data that no significant degradation of the samples was detected.

**Table V. Effect of time on possible degradation of sample**

Sample	% Invert by HPLC	% Invert by HPLC 24 hours later
Raw sugar	0.56	0.56
Soft liquor	1.71	1.80

### Summary

A modified version of a high pressure liquid chromatography analytical system employing a post-column derivatization reaction and its applicability to cane sugar invert analysis are described. The advantages gained in sensitivity by using such a system *versus* others currently used for invert sugar analysis are discussed. Invert measurements made by this system for some selected cane sugar refinery samples are compared with values obtained by the conventional Lane & Eynon method.

### Analyse de l'inverti du sucre de canne par HPLC utilisant la formation de dérivé après colonne

L'auteur décrit une version modifiée du système d'analyse par chromatographie liquide à haute pression utilisant, après colonne, une réaction de formation de dérivé ainsi que son applicabilité à l'analyse de l'inverti du sucre de canne. Il discute des avantages d'une plus grande sensibilité, obtenue grâce à ce système, par rapport à d'autres systèmes couramment utilisés pour l'analyse du sucre inverti. Les mesures d'inverti réalisées avec ce système sur quelques échantillons choisis de sucre de canne, en raffinerie, sont comparées avec les résultats obtenus avec la méthode conventionnelle de Lane & Eynon.

### Rohrzucker-Invertzucker-Analyse mit HPLC unter Verwendung einer Nach-Säulen-Derivatisierungsreaktion

Eine modifizierte Version der Hochdruckflüssigkeitschromatographie unter Verwendung einer Nach-Säulen-Derivatisierungsreaktion und deren Anwendbarkeit auf Rohrzucker-Invertzuckeranalysen wird beschrieben. Die Vorteile in Bezug auf Sensibilität bei Verwendung eines solchen Systems im Vergleich zu anderen momentan für die Invertzuckeranalysen verwendeten werden diskutiert. Invertzuckerbestimmungen, die mit diesem System für einige ausgewählte Rohrzuckerrefinerieproben gemacht wurden, werden mit Werten verglichen, die mit der konventionellen Lane & Eynon-Methode erhalten wurden.

### Análisis de azúcar invertido en azúcar de caña por HPLC utilizando la formación del derivado después de la columna

Se describen un versión modificada del sistema analítico por cromatografía a presión alta, empleando la



formacion, después de la columna, del derivado, tanto como su aplicabilidad al análisis del invertido en azúcar de caña. Los ventajas obtenido en sensibilidad por uso de un tal sistema *versus* los otros empleados actualmente para análisis de azúcar invertido se discuten. Medidas del

Cane sugar invert analysis by HPLC

invertido obtenido por este sistema sobre algunas muestras escogidas de una refinería de azúcar de caña se comparan con valores obtenido por el método convencional de Lane & Eynon.

# Microprocessor control of sugar crystallization

By JOUKO VIRTANEN  
(Finnish Sugar Co. Ltd.)

## Introduction

The rapid increase in energy prices during the last ten years has put new demands on the efficiency of sugar production. Also, at the crystallization stage new trends can be seen; new designs for continuous crystallization equipment have been studied and the degree of automation of batch boiling pans has increased.

In the crystallization of any material, supersaturation of the mother liquor around the crystals is the most important variable: crystallization velocity is proportional to supersaturation and nucleation rate is also a function of supersaturation. In the sugar industry the following methods have been used to measure the concentration of the mother liquor during boiling:

**Boiling point elevation:** This is particularly applicable to pure sucrose solutions being crystallized. Its disadvantage is, however, that no information can be obtained about the crystal content of the massecuite. With impure solutions the boiling point elevation only measures the dry substance, not the sucrose concentration of the mother liquor, so that supersaturation cannot be defined accurately.

**Refractive index measurement:** This can be applied but suffers the same limitations as boiling point elevation.

**Viscosity:** This is synonymous with consistency measurement; it has gained a strong foothold in control systems and can be applied for pure and impure sucrose solutions. In this case the greatest disadvantage is the interfering effect of the crystals upon the reading: different mother liquor concentration — crystal content combinations can give the same result.

**Electrical conductivity:** This method has also been applied widely, especially in beet and cane sugar factories. In refineries with low salt concentrations in the solutions the method is not applicable. A more sophisticated method, which is based on dielectric constant, has also been developed recently<sup>1</sup>. This method can also be used in refineries.

With all these methods the whole strike from seeding to tightening cannot be controlled properly with one measurement alone. This problem has been overcome in the latest automatic systems by combining two of the above mentioned measurements, for instance boiling

point elevation and consistency measurement<sup>2</sup>. Even then, in these systems supersaturation cannot be exactly defined under all boiling conditions. Usually the process is forced to follow a predetermined pathway, which may not necessarily define the correct supersaturation.

In the Finnish Sugar Co. Ltd. we have tried to develop a method by which the exact supersaturation can be determined during the whole strike, and which can be used both with pure solutions and with low purity syrups. A characteristic of the method is also that the process controls itself, with the crystallization velocity at the desired supersaturation level determining the rate of progress.

## Background

In Finland we started the automation of the crystallization process in cooling crystallizers. The cooling rate of fructose crystallizers has now been controlled successfully for five years with a system which is based on the measurement of mother liquor concentration by refractometer. Encouraged by this achievement we started to consider how the same principles could be used in the crystallization of sucrose. What we needed was an additional method to measure continuously the total dry substance concentration of the massecuite.

Different possibilities, for instance measuring the amount of feed dry substance and of secondary condensate, were investigated. The solution was found at one of our beet sugar factories, where a density meter based on radioactivity measurement was tested on juice concentration after extraction. We installed the density meter in a sugar pan and found a good correlation between the total dry substance concentration and density of the massecuite.

## Calculation of supersaturation from the measured data

The supersaturation of the mother liquor can be determined at any moment during the strike, when the mother liquor concentration and total dry substance concentration of the massecuite are measured. Crystal yield is calculated from these two measured values by the equation (1).

$$Y_c = \frac{10,000 (C_{TDS} - C_{ML})}{C_{TDS} (100 - C_{ML})} \quad (1)$$

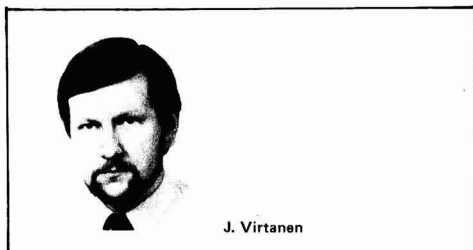
where  $Y_c$  = crystal yield (% of dry substance amount),  $C_{TDS}$  = measured total dry substance concentration (% w/w), and  $C_{ML}$  = measured mother liquor concentration (% w/w).

When the purity of the feed syrup is known from laboratory analysis, the purity of the mother liquor can be calculated by equation (2).

Paper presented to Sugar Industry Technologists, 1983.

<sup>1</sup> US Patent 4,196,385.

<sup>2</sup> Sullivan: *ibid.*, 1979, 81, 259-263.



J. Virtanen

$$Q_{ML} = 100 \times \frac{Q_{FL} - Y_C}{100 - Y_C} \quad (2)$$

where  $Q_{ML}$  = mother liquor purity (% w/w of dry substance) and  $Q_{FL}$  = feed syrup purity (% w/w of dry substance).

When the purity of the mother liquor is known and the temperature of the massecuite is measured, we can define the saturation concentration of the mother liquor at that particular temperature and purity as function (3):

$$C_{ML}^{SAT} = f(Q_{ML}, t_M) \quad (3)$$

where  $C_{ML}^{SAT}$  = saturation concentration of the mother liquor and  $t_M$  = temperature of the massecuite.

Finally, the supersaturation can be calculated from equation (4):

$$SS = \frac{C_{ML} (100 - C_{ML}^{SAT})}{C_{ML}^{SAT} (100 - C_{ML})} \quad (4)$$

### Instrumentation

The instrumentation diagram of the pan is shown in Figure 1. The measuring instruments, refractometer (QI 189) and density meter (DT 190), are installed at the bottom of the pan to eliminate disturbances due to steam bubbles. Temperatures are measured at four points with Pt-100 probes, of which the measurement closest to the refractometer and density meter is used in the calculation of the supersaturation. In addition the pan is equipped with a liquid level transmitter (LT 191) and vacuum meter (PT 188). The steam and feed flow rates and vacuum are regulated through control valves. The water feed pipe is equipped with on-off valves and a pulse counter. All other pipe-lines are equipped with on-off valves. The seed funnel is also furnished with an agitator and level sensors. All the valves and instruments, which are marked with the relevant  $\oplus$ -sign in the diagram, have a LED light in the instrument panel to indicate their operational state.

The measuring signals are taken to a microprocessor unit, which calculates supersaturation and controls the operation of the pan.

A parts list of the devices used is given in Appendix I.

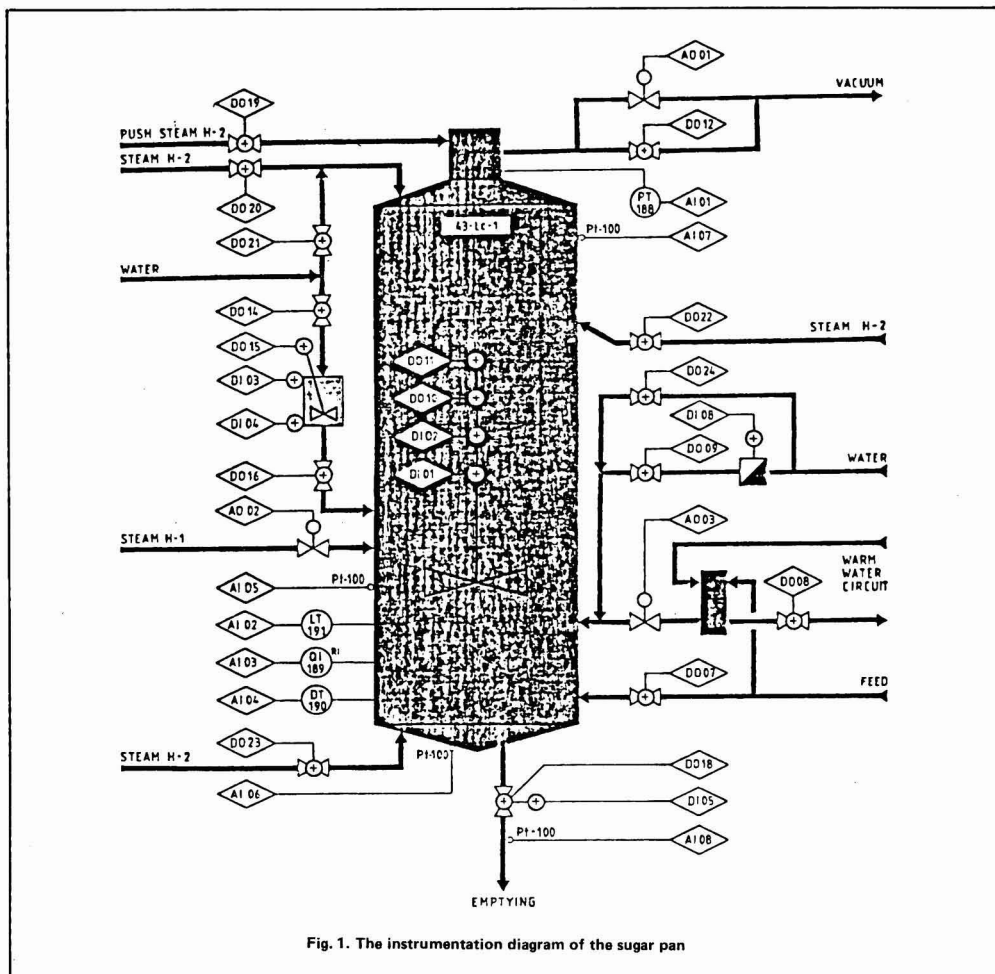


Fig. 1. The instrumentation diagram of the sugar pan

The program of the microprocessor has been constructed in sequence form. At the beginning all valves of the pan are closed and the pan can be under normal pressure or vacuum. When the operator gives a command to start the crystallization, the pressure is at first checked and vacuum applied if necessary. When a pre-set vacuum is reached, the feed control valve and feed by-pass valve are opened. When the footing level is reached, both valves are closed and the steam valve opened slowly. During concentration of the footing, the level is controlled by the feed valve. At this point in the sequence a signal is given to the operator to fill the seed funnel. The concentration of the solution is measured continuously by refractometer and density meter and the microprocessor calculates the supersaturation continuously from equations (1)-(4). When the supersaturation is close to the seeding point, the feed valve is closed and the steam valve is set at "seed setting". At the pre-determined supersaturation the bottom valve of the seed funnel is opened and the seed slurry sucked into the pan. After seeding the settings of the valves are kept constant for a while to give the seeds time to grow visible, then the extra supersaturation is diluted by the addition of water drinks. The steam valve is opened to "growing setting" and the supersaturation is controlled with the feed valve. Theoretically, at this point in the process, steam should be used carefully to remain in step with the deposition of sugar on the seed crystals which is low, owing to the small crystal surface area. However, good agitation is vital to keep the growth velocity of single crystals at its optimum and good agitation can be maintained only by efficient boiling. In the case of low purity strikes, water drinks can also be added in this sequence to keep the supersaturation at the desired value and to avoid the level rising too rapidly.

When the total crystal mass reaches a certain value,

the control strategy is changed to keep the circulation of the massecuite at its optimum: the crystal content is controlled by the feed valve and supersaturation is controlled by the steam valve. This sequence continues until the pan is full, when the feed valve is closed. Supersaturation is controlled during tightening by the steam valve. The crystallization is ended when the power uptake of the motor of the impeller reaches a set value. The steam and vacuum valves are closed and the vacuum is decreased to a predetermined value, when the bottom valve may be opened and the pan emptied. The pan is then washed with steam, all valves are closed and the program awaits a new command to start the next cycle. The vacuum is controlled during the whole cycle by a vacuum control valve.

In Figure 2 a typical recorder chart is shown, in which the concentration measurements and supersaturation during high purity crystallization are presented.

To be profitable in practice, automation must improve the process. In sucrose crystallization we had as an objective the shortening of boiling time while obtaining uniform crystal size distribution, reducing labour costs and saving energy consumption.

#### Accuracy of the measurements

At first we can have a look at the accuracy requirements of the measurements to produce reliable supersaturation readings. If we can accept an error of 0.02 units in the supersaturation value, the error in mother liquor concentration measurement must be smaller than  $\pm 0.2\%$  w/w, the error in total dry substance concentration smaller than  $\pm 0.2\%$  w/w and the accuracy of temperature measurement within  $\pm 1.5^\circ\text{C}$ .

The accuracy of temperature measurement with a Pt-100 sensor is of the order of  $\pm 0.3^\circ\text{C}$ , which satisfies

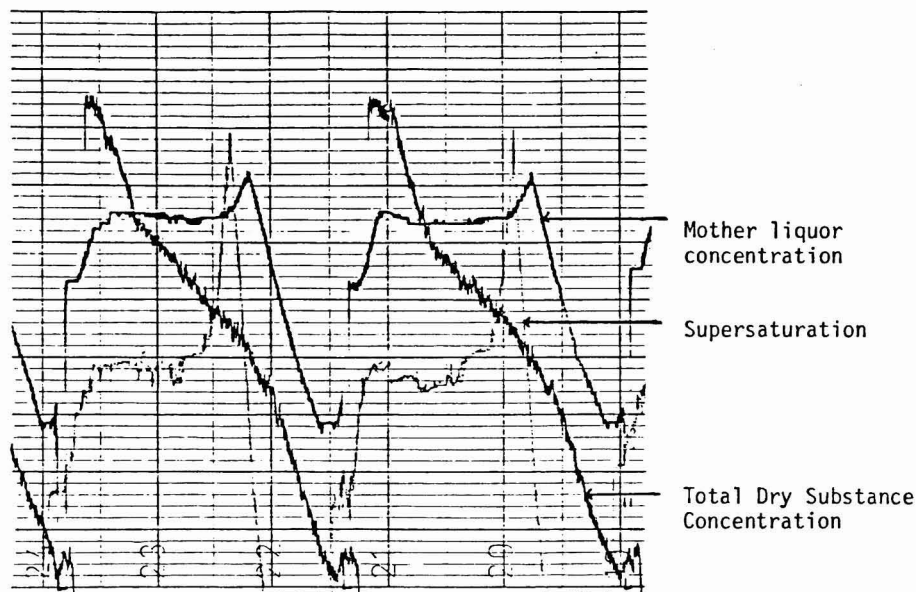


Fig. 2. A typical recorder chart of a high purity crystallization

the above requirement. The accuracy of total dry substance measurement by the radioactivity-based density meter has been found to be  $\pm 0.3\%$  w/w, which is completely satisfactory for high purity massecuites. In the case of feed purities below 80% at the end of crystallization, this inaccuracy slightly limits the value of supersaturation. The density meter has worked reliably all the time with no disturbances.

We faced the biggest problem with refractive index measurement. During the trial runs the instrument first used behaved faultlessly until the crystal content reached a certain critical area. At this point the signal suddenly became unstable and could travel outside the measuring range. The reason for this behaviour was obviously deposition of sugar crystals on the prism owing to the use of cooling water on the measuring head. The final solution to this problem was a new refractometer of totally different construction. This new instrument has worked well throughout the whole strike, and the accuracy has been of the order of  $\pm 0.1\%$  w/w.

The inaccuracy of each measurement is well below the required limit and it is clear that the total uncertainty in the supersaturation lies around  $\pm 0.02$  units.

#### The effect of pan construction

In addition to the errors in the measurements, this control method suffers from the same disadvantage as all other systems: supersaturation is measured at one point only. It is well known that sugar pans are by no means perfectly mixed vessels. Concentrations can vary considerably in different parts of the pan and the situation becomes especially difficult when the crystal content rises during tightening. We also know that when the level in the pan increases, hydrostatic pressure increases the temperature at the bottom of the pan, if the circulation is not good enough. This means higher supersaturation on the surface of the massecuite.

We have overcome these problems by measuring temperature at several points in the pan. In this way we have obtained valuable information about circulation inside the pan. This has helped us to find the optimum crystal content, where circulation is still efficient. The properties of the microprocessor can also be exploited by changing the supersaturation set-value to take account of the differences in conditions at the surface and at the bottom.

#### Crystal size distribution

Having discussed the reliability of the method, we can now return to the final targets we set for the control system. In Table I the crystal size characteristics (mean aperture and coefficient of variation) are shown for automatic and manually controlled boilings which were run simultaneously with pure sucrose solutions. The results are relatively similar; the coefficients of variation are equal, but the reproducibility of the mean size is better in the automatic boilings.

#### Crystallization time

The boiling time with high purity syrups was equal for both manual and automatic operation. However, as mentioned in the previous paragraph, better reproducibility is obtained within minimum crystallization time by automatic operation, as the different personal habits of the operators are eliminated. The demand for correct supersaturation is much more obvious with low purity

Table I. Crystal size characteristics for high purity sugars

Automatic boiling		Manually controlled boiling	
M.A.	C.V.	M.A.	C.V.
0.51	24.3	0.49	25.1
0.47	39.8	0.41	47.6
0.53	26.4	0.55	32.4
0.51	27.8	0.49	26.3
0.50	27.6	0.51	21.2
0.51	38.8	0.49	28.0
0.54	27.8	0.50	27.8
0.52	27.3	0.45	29.3
0.49	28.2	0.50	32.6
0.48	27.7	0.54	28.1
0.47	27.7	0.51	25.5
0.53	26.0		
0.51	27.5		
0.48	29.4		
0.49	42.0		
0.54	26.1		
0.47	30.0		
0.50	28.2		
0.50 $\pm$ 0.02		0.50 $\pm$ 0.04	

boilings in beet and cane sugar factories, where we expect to see big advantages from our automatic process.

#### Labour costs

A boiling house with up to ten pans can easily be run with only one operator, if all the pans are equipped with this control system. The only manual work which is left is grinding of the seed sugar, filling the seed funnel and giving the microprocessor a command to start the cycle and permission to empty the pan. The crystal growth can naturally be followed with a projecting microscope.

#### Energy considerations

Even loading of the power plant is necessary to keep the efficiency in steam generation at optimum. In manual operation of the boiling station the steam consumption can vary considerably depending on the momentary needs of different pans. With the control system described the progress of the boiling can be adjusted by giving each pan different values of supersaturation. This can be done by connecting the individual microprocessors to a central unit, which monitors the steam consumption in different pans, makes optimization calculations and adjusts the set value for supersaturation accordingly.

#### Conclusions

As we have seen, this new control method gives a product of quality equal to that of the traditional manual operation and with the same throughput. The main improvements are achieved in reductions of labour costs and improved power plant economy. The investment costs of this control system depend on the existing equipment, such as control valves etc. The price of the main instruments (refractometer, density meter and the microprocessor) is altogether approximately \$16,000 in Finland.

#### Future

We are co-operating with a Finnish university, Abo Akademi, where a simulation model of sugar crystallization has been developed<sup>3,4</sup>. Combining this simulation

<sup>3</sup> Gros: *Kemia-Kemi*, 1979, 6, 448.

<sup>4</sup> Gros & Nurmi: *ibid.*, 534.

model with our present method makes totally new control algorithms possible. For instance we can apply feed-forward loops in addition to traditional feed-back loops.

The use of this new control method is not limited to sucrose crystallization, but can be applied in any batch crystallization. Also in continuous crystallization the measurement of both crystal yield (magma density) and supersaturation make control of the process easier.

#### Appendix I. List of measuring instruments

##### Refractometers:

- A. Electron Machine Corp., model SSR-72
- B. K-patents, model PR-0.1 (Ekström, Helsinki)

##### Radioactivity-based density meter:

Berthold, model LB371, with Cs-137  $\gamma$ -ray source

##### Microprocessor:

Labko, model LMPS, with INTEL SBC 80/10 CPU CARD

#### Summary

A combination of refractive index, massecuite density measurements and temperatures at different locations within a pan is used in conjunction with a microcomputer programmed to control automatically the whole of the boiling process. Comparative trials have shown sugar crystal uniformity to be better under automatic control than with manual regulation, while the computer can also adjust the progress of each strike by adjusting the supersaturation in individual pans so as to ensure the optimum regularity of steam demand for energy economy.

#### Microprocesseur pour contrôle de la cristallisation du sucre

Une combinaison de l'index de réfraction, de mesure de densité et de température de masse cuite à différentes

localisations dans la cuite sont utilisés en liaison avec un microordinateur pour contrôler automatiquement le process complet de cuisson. Des essais comparatifs ont montré qu'une meilleure uniformité du cristal de sucre est obtenue avec un contrôle automatique qu'avec une régulation manuelle. Le mini-ordinateur ajuste la progression de chaque cuite en ajustant la supersaturation dans les cuites individuelles de façon à assurer une régularité optimum de la demande en vapeur et ainsi en résulte une économie d'énergie.

#### Microprozessor-Regelung der Zuckerkrystallisation

Eine Kombination der Messungen von Brechungsindex, Kochmassendichte und Temperaturen an verschiedenen Punkten innerhalb eines Kochapparates wird in Verbindung mit einem Microcomputer verwendet, der programmiert ist, den gesamten Kochprozeß automatisch zu regeln. Vergleichsuntersuchungen zeigten, daß die Einheitlichkeit der Zuckerkrystalle bei automatischer Kontrolle besser war als bei Handregelung. Der Rechner kann auch die Geschwindigkeit jedes Sudes durch Anpassung der Übersättigung in den einzelnen Kochapparaten regeln, um die optimale Gleichmäßigkeit der Dampfabnahme für die Energiewirtschaft zu sichern.

#### Control por micro-procesor de cristalización de azúcar

Se usa una combinación de índice de refracción, medidas de densidad de masa cocida y temperaturas a varios lugares en un tacho, junto con un micro-computador, programado para control automático del proceso completo de cocción. Ensayos comparativos han demostrado que la uniformidad de cristales de azúcar es mejor sobre control automático que con regulación manual, mientras que el computador puede ajustar el progreso de cada templa por regulación del sobresaturación en tachos individuales para asegurar la regularidad óptima del consumo de vapor para economía de energía.

## Polarimetric study of the basic lead acetate-fructose complex

By AMANDA RODRIGUEZ\*, ARMANDO PERDOMO\*, FERNANDO FERNANDEZ\* and Dr. JOSE FERNANDEZ BERTRAN†

#### Introduction

The effect of lead ions on the optical rotatory power of sucrose, glucose and fructose solutions is of importance owing to the use of basic lead acetate as clarifying agent in polarimetric determination of sucrose in juices<sup>1-5</sup>. The effect is due to complex formation and depends on the spatial arrangement of the hydroxyl groups in the carbohydrate molecules<sup>6,7</sup>. Glucose forms an insoluble complex with basic lead acetate, while sucrose interacts weakly in neutral solutions<sup>8</sup>.

The optical rotation of fructose solutions is affected by lead ions<sup>2,5</sup>. In this paper we present a polarimetric study of fructose-basic lead acetate solutions which confirms the formation of a 1:1 complex of fructose and lead acetate.

#### Experimental method

A standard solution of basic lead acetate (Riedel de Haen) was prepared and filtered, the lead concentration being determined gravimetrically as sulphate. A standard fructose solution was prepared and its concentration was determined polarimetrically after mutarotation.

Mixed solutions of different fructose (F) and lead (L) concentrations were prepared by mixing of the standard solutions. Polarimetric readings were performed in a Polartronic I polarimeter, using sodium light and 200 mm tubes at 20°C.

The difference  $\Delta$  between the polarization of complexed and uncomplexed fructose solutions are precise to  $\pm 0.02^\circ\text{S}$  and reported in Table I.

#### Results and discussion

The values of  $\Delta$  for solutions of fixed fructose concentration (F) and varying lead concentration (L) are linearly correlated to L for  $(L) < (F)$ . The slopes T and

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<sup>1</sup> Mesley: *J.S.J.*, 1962, **64**, 75.

<sup>2</sup> Gaskin & Mesley: *ibid.*, 1958, **60**, 65.

<sup>3</sup> Parker: *ibid.*, 197.

<sup>4</sup> Douwes Dekker: *Comm. Sugar Milling Research Inst. (Natal)*, 1957, 36.

<sup>5</sup> Bates & Blade: *Bull Nat. Bur. Standards*, 1907, (3) 105.

<sup>6</sup> Rendleman: *Adv. Carbohydr. Chem.*, 1966, (21), 209.

<sup>7</sup> Vavrinecz: *Zeitsch. Zuckerind.*, 1969, **94**, 607.

<sup>8</sup> Ramaiah & Vishnu: *Sharkara*, 1959, (2), 3.

Table I. Values of  $\Delta$  for fructose and basic lead acetate solutions at 20°C in 200-mm tubes (Concentrations in Moles/litre;  $\Delta$  in saccharimetric degrees)

F	L	$\Delta$
0.055	0	0.00
	0.022	0.50
	0.044	1.05
	0.066	1.45
	0.088	1.97
	0.110	2.35
0.110	0	0.00
	0.022	0.67
	0.044	1.24
	0.066	1.75
	0.088	2.43
	0.110	2.86
0.220	0	0.00
	0.022	0.66
	0.044	1.30
	0.066	2.21
	0.088	2.75
	0.110	3.32
0.440	0	0.00
	0.022	0.80
	0.044	1.59
	0.066	2.54
	0.088	3.25
	0.110	3.90

correlation coefficient  $r$  for four series of solutions are given in Table II. The excellent correlation proves that, in the concentration range studied, the complex contains only one lead acetate molecule. For this stoichiometry the following relation should be obeyed between the slopes  $T$  and the concentration  $F$  for the four series of fructose solutions ( $F = 0.055; 0.11; 0.22$  and  $0.44$  moles/litre)<sup>9</sup>:

$$(F/T) = \frac{1}{(c-f)} (F) + \frac{1}{K(c-f)} \quad (1)$$

where  $c$  and  $f$  are the optical rotations of 1 Molar solutions of the complex and free fructose in 200 mm tubes. The constant  $K$  is the equilibrium constant of the 1:1 complex. The values of  $c$  and  $K$  can be obtained from the slope and intercept of equation 1.

Table II. Correlation parameters of  $\Delta$  vs. (L) plots, at constant fructose concentration and at 20°C

(F), m/l	Slope T	Correlation coefficient, $r$	No. of experimental points
0.055	21.5	0.999	6
0.110	26.1	0.999	6
0.220	30.8	0.998	6
0.440	36.1	0.999	6

The observed dependence of  $(F/T)$  on  $F$  is:

$$(F/T) = 24.8 \times 10^{-3} (F) + 1.41 \times 10^{-3} \quad (2)$$

The correlation coefficient is 0.999 which corresponds to 99% confidence level for 4 experimental points. The excellent linearity confirms that the predominant species is the 1:1 fructose-lead acetate complex.

The value of  $K$  is 17.5 l/m at 20°C which corresponds to a weak complex. The value of  $c$  is  $-55.0^\circ$ , nearly half that of fructose which is  $-95.33^\circ$ .

The change in optical rotatory power can be due to different causes: (a) changes in conformation of the carbohydrate molecule, (b) changes in the equilibria between  $\alpha$ ,  $\beta$ , pyranose, furanose forms of fructose on complex formation<sup>10</sup>, or (c) creation of a new centre of a symmetry.

In polysaccharides, changes in conformation of the polymer with solvation or complex formation can produce drastic changes in optical rotatory power. However, such large changes are not possible for monosaccharides, particularly in the case of a weak complex.

The shift of equilibrium between the different molecular species of fructose can also be discarded since no mutarotation is observed on addition of the complexing agent to the equilibrated fructose solution. The two predominant species,  $\beta$ -fructopyranose (57%) and  $\beta$ -fructofuranose (31%) both have suitably oriented hydroxyl groups for chelating the lead cation but the marginal species,  $\alpha$ -fructopyranose (3%) and  $\alpha$ -fructofuranose (9%) do not have properly oriented hydroxyl groups.

The value of  $c$  is possibly determined by the induced asymmetry of the lead ion by the fructose molecule. Lead has a large numbers of electrons compared with C, O and H atoms and small induced asymmetries can produce large changes in rotatory power as in the well-studied  $\text{Cu}^{++}$  complexes<sup>11</sup>.

### Summary

A polarimetric study of fructose-basic lead acetate solutions at 20°C reveals the formation of a weak 1:1 complex with a stability constant of 17.5 l/m and a rotatory power half of that of fructose.

### Etude polarimétrique du complexe fructose-acétate basique de plomb

Une étude polarimétrique des solutions de fructose-acétate basique de plomb, à 20°C, montre la formation d'un complexe 1:1 faible, avec une constante de stabilité de 17,5 l/m et un pouvoir rotatoire réduit de moitié par rapport à celui du fructose.

### Polarimetrische Untersuchung eines basischen Bleiacetat-Fructose-Komplexes

Eine polarimetrische Untersuchung von Fructose-Bleiacetat-Lösungen bei 20°C ergab die Bildung eines schwachen 1:1-Komplexes mit einer Stabilitätskonstanten von 17,5 l/m und einer Rotation, die halb so groß ist, wie die von Fructose.

### Estudio polarimétrico del complejo fructosa-acetato básico de plomo

Un estudio polarimétrico de soluciones de fructosa-acetato básico de plomo a 20°C revela la formación de un complejo 1:1 débil con un constante de estabilidad de 17,5 l/m y un poder rotatorio reducido un mitad por relación al poder de fructosa.

<sup>9</sup> Fernández Bertrán & Marinello: *Cuba-Azúcar*, 1966 (May-June), 1.

<sup>10</sup> Dodrell & Allerhand: *J. Amer. Chem. Soc.*, 1971, (93), 2777.

<sup>11</sup> Reeves: *Adv. Carbohydr. Chem.*, 1951, (6), 108.



## South Africa sugar imports and exports, 1983<sup>1</sup>

	1983	1982
	tonnes, raw value	
<b>Imports</b>		
Argentina	14,553	0
Brazil	70,920	0
Korea, South	13,682	0
Swaziland	79,878	0
US	12,827	0
	<u>191,860</u>	<u>0</u>
<b>Exports</b>		
Canada	158,983	212,285
Israel	65,212	16,846
Japan	140,631	452,020
Korea, South	91,434	136,348
Portugal	39,389	27,218
US	59,787	34,832
Other countries	8,893	3,106
	<u>564,329</u>	<u>882,655</u>

**French-Soviet sugar trade<sup>2</sup>.** — It was announced in early February that a long-term economic agreement had been signed between the USSR and France entailing the supply of agricultural products from France in exchange for natural gas, among other things. During 1984, France is scheduled to supply 600,000 tonnes of sugar to the Soviet Union, some 65% more than the 361,000 tonnes delivered in 1983.

**Burundi cane sugar factory tender<sup>3</sup>.** — It is intended to call an international tender for the delivery on a turnkey basis of a new sugar factory with a guaranteed crushing capacity of 1000 t.c.d. with the possibility of extension to 8500 t.c.d., to be located at Kihovi, near Rutana, in the south-eastern part of Burundi. The project will be financed by the African Development Bank and a number of Arab funds.

**Ivory Coast sugar factory closures<sup>4</sup>.** — After only four years in operation, two of the Ivory Coast's six sugar complexes are to close. The complexes — Ferkessedougou 2 and Katiola — each cost about \$150 million and have been operating at a considerable loss. The complexes have never run efficiently, partly owing to poor management and lack of skills. The closures follow pressure from the I.M.F. to cut public expenditure, and will facilitate agreement on a SDR 50 million (\$51.7 million) one-year standby arrangement in 1984. This would replace the three-year extended fund facility that ran out at the end of February.

**New Thailand sugar factory<sup>5</sup>.** — A sugar factory belonging to Wang Kanai Sugar Company in Kanchanaburi, opened in February, has a crushing capacity of 12,000-15,000 t.c.d. and is claimed to be the largest in Asia.

**EEC Commission proposals for an alcohol regime<sup>6</sup>.** — The revised proposals by the EEC Commission for an alcohol regime provide for the following: (i) the creation of a market support system regime for ethyl alcohol of agricultural origin; (ii) the definition of alcohol-producing agricultural products to include fruit and vegetables, cereals, sugar beet and sugar cane; (iii) a flat rate subsidy to be paid per hectolitre of alcohol produced from beet, cane, potatoes, fruit and cereals, the amount to vary according to the raw material used; (iv) the subsidy to be available only for a limited quantity of alcohol to be set on the basis of an average of the previous five years' production and possible production outlets (this could lead to conflict as the quantity of alcohol produced from these raw materials has been too large for the outlets available); (v) the alcohol quantities eligible for subsidy to be apportioned among distillers; (vi) specific provision to prevent disruption in the market for alcohol from molasses will include the payment of a "temporary aid" to alcohol from molasses; and (vii) national provisions allowing monopolies, or exclusive rights, for alcohol of agricultural origin to be banned. The regime would operate by management committee procedure.

## Norway sugar imports, 1983<sup>7</sup>

	1983	1982	1981
	tonnes, raw value		
Austria	10,036	17,764	11,270
Belgium	1,726	2,361	3,672
Denmark	73,990	61,854	94,792
Finland	28,514	13,040	13,702
France	1,045	0	1,241
Germany, West	17,516	22,001	35,144
Sweden	5,792	113	78
UK	18,884	26,341	29,745
Other countries	273	34	367
	<u>157,776</u>	<u>143,508</u>	<u>190,011</u>

**US corn sweetener company plans<sup>8</sup>.** — Corn Products Company has announced plans for a program of expansion and productivity improvement. Production capacity at the company's corn wet-milling plant in Winston-Salem, NC, will be expanded by 50%, primarily to increase production of second generation (55%) high fructose syrup. At Stockton, CA, CPC will add a channel for production of second generation HFS, shifting some of its grind capacity from starch and first generation (42%) HFS which have been produced there since the plant was completed two years ago. The company will also rebuild its plant in Argo, IL, which will take several years and cost more than \$300 million to complete. In the spring of 1984, CPC's new subsidiary, Enzyme Bio-Systems Ltd., will begin construction of a new plant in Beloit, WI. The two-storey structure will contain approximately 30,000 square feet of work area and will be completed in 1985. Enzymes for the production of carbohydrate sweeteners, alcohol, baking products, etc., will be produced at the plant.

**Thailand sugar factory expansion<sup>9</sup>.** — Eighteen foreign firms, most of them Japanese, are reported to have bought tender documents for an expansion project whereby the daily crushing capacity of the Supanburi sugar factory is to be increased from 3000 to 13,000 tonnes.

**Madagascar sugar project<sup>10</sup>.** — Madagascar has invited bids for the building of infrastructure for the Analaiwa-Morondava sugar scheme on the western shore of the island state. Financing is being provided by the African Development Bank which, since the ADB is well-known for its conservative policies, is seen as an indication of its potential profitability.

**EEC sugar carry-forward.** — A total of 215,000 tonnes of white sugar from the 1983/84 crop is to be carried forward to the 1984/85 marketing year<sup>11</sup>. Italy will carry forward 1000 tonnes and Greece 7000 tonnes of B-quota sugar, which receives partial price support. The remainder is C- or non-quota sugar, including 40,000 tonnes in West Germany, 147,000 tonnes in France, and 20,000 tonnes in Ireland. The total compares with 1.1 million tonnes set aside in 1982/83 and 1.0 million tonnes in 1981/82, more than a quarter of C-sugar produced in each year; when carried forward it became part of the next year's A- or B-quota sugar. Prospects are for a rise in production in the 1984/85 crop, however, with a beet area larger by some 3% and better weather conditions during sowing than for the 1983 crop. Consequently carried-forward C-sugar from the 1983/84 crop is likely to result in a larger amount of C-sugar from the 1984/85 crop, and producers have thus decided to sell rather than store it.

**New Turkish sugar factory<sup>12</sup>.** — The new Bor-Nigde sugar factory in Turkey began operations in November 1983 for a trial campaign. The 3000 tonnes/day plant was built in Turkey except for a few pieces of equipment (turbines, centrifugal drives, etc.). As the boiling house was not completed, the factory operated at a beet slice of only 1800 tonnes per day in 1983.

<sup>1</sup> F. O. Licht, *International Sugar Rpt.*, 1984, 116, S57.

<sup>2</sup> C. Czarnikow Ltd., *Sugar Review*, 1984, (1687), 27.

<sup>3</sup> F. O. Licht, *International Sugar Rpt.*, 1984, 116, 129.

<sup>4</sup> *World Sugar J.*, 1984, 6, (8), 40.

<sup>5</sup> F. O. Licht, *International Sugar Rpt.*, 1984, 116, 131.

<sup>6</sup> *World Sugar J.*, 1984, 6, (9), 41.

<sup>7</sup> F. O. Licht, *International Sugar Rpt.*, 1984, 116, S66.

<sup>8</sup> *Amerop-Westway Newsletter*, 1984, (124), 13.

<sup>9</sup> F. O. Licht, *International Sugar Rpt.*, 1984, 116, 131.

<sup>10</sup> *African Business*, 1984, (66).

<sup>11</sup> F. O. Licht, *International Sugar Rpt.*, 1984, 116, 139.

<sup>12</sup> *Zuckerind.*, 1984, 109, 259.

## Dominican Republic sugar exports, 1983<sup>1</sup>

	1983	1982
	tonnes, raw value	
Algeria	0	11,330
Cuba	0	73,491
EEC	19,724	0
Haiti	24,337	4,922
Jamaica	10,815	0
Japan	14,913	0
Mexico	19,157	0
Morocco	53,597	15,449
Philippines	30,345	0
Portugal	0	46,098
Senegal	24,462	25,692
Trinidad	2,626	0
Tunisia	8,652	0
US	536,095	362,920
USSR	193,320	234,736
Venezuela	15,593	66,787
Other Central America	2,537	8,753
	<u>956,173</u>	<u>850,178</u>

**Revere Sugar contraction<sup>2</sup>.** — Revere Sugar Corporation, which owns three refineries in the US, is to be restructured; operations at the Charlestown (Boston, MA) refinery and the Chicago liquid sucrose and invert syrup plant are to be suspended, while the corporate offices are to be relocated from Lyndhurst, NJ, to the Brooklyn, NY, refinery which will continue operations. A program of capital and other improvements to increase efficiency at the Brooklyn refinery is in progress and while this will compensate for some of the lost capacity, there will be an effective reduction for the company. Revere will maintain sales offices and inventories in the Boston and Chicago markets. The company said the consolidation of operations was necessitated by the severe reduction in the market for refined sugar in recent years resulting largely from the increased use of high fructose syrup by the US food and beverage industries. During this period the US government's sugar program has shifted most of the burden of that reduced demand for refined sugar to the cane sugar refineries. By raising domestic sugar prices through strict quotas, the government has provided a price umbrella for HFS and guaranteed a market for domestic beet and cane sugar producers at more than triple current world prices, the company said.

**USSR sugar beet production<sup>3</sup>.** — While total beet purchases in 1983 were below the level set in the five-year plan, they were 22% up on the 1982 figure. Planned beet procurements for 1984 are set at 86.5 million tonnes.

**Turkey campaign results, 1983/84<sup>4</sup>.** — It has been officially reported that, from a total crop of 12,142,200 tonnes of sugar beet, Turkey produced 1,628,660 tonnes of white sugar in the 1983/84 campaign. Exports amounted to 308,900 tonnes. The 1984/85 beet crop is expected to be about the same as in 1983/84.

## Philippines sugar exports, 1983<sup>5</sup>

	1983	1982	1981
	tonnes, raw value		
Algeria	11,383	20,685	0
Bulgaria	12,622	0	0
China	0	198,776	92,719
Comoros	542	0	0
Fiji	13,655	0	0
Hong Kong	8,132	4,468	0
India	0	0	8,648
Indonesia	4,337	86,321	217,583
Iran	0	0	8,648
Iraq	0	75,725	23,050
Japan	207,622	315,102	121,227
Korea, South	140,714	98,725	157,519
Malaysia	15,693	15,413	19,899
Mexico	86,561	13,696	0
Morocco	0	0	26,930
Persian Gulf	8,123	0	0
Portugal	0	0	22,059
Saudi Arabia	0	0	15,033
Sri Lanka	31,191	39,988	33,104
US	290,260	222,893	188,558
USSR	167,955	209,306	336,836
	<u>998,790</u>	<u>1,301,098</u>	<u>1,277,593</u>

## Japan sugar imports, 1983<sup>6</sup>

	1983	1982	1981
	tonnes, tel quel		
Australia	464,810	563,765	595,299
Brazil	0	17,918	0
Colombia	35,712	0	0
Cuba	318,091	304,190	245,106
Dominican Republic	14,398	0	0
Fiji	0	14,766	52,990
Philippines	234,161	270,541	137,507
South Africa	147,668	494,947	365,710
Taiwan	65,946	130,644	97,624
Thailand	528,090	370,966	95,649
Other countries	107	1,130	1,663
	<u>1,808,983</u>	<u>2,168,867</u>	<u>1,591,548</u>

**HFS plant in Egypt<sup>7</sup>.** — A Belgian-Egyptian consortium has begun construction in Egypt on a high fructose syrup plant. From 120,000 tonnes of maize the plant will produce 50,000 tonnes of 42% HFS and 46,000 tonnes of 55% HFS as well as 3000 tonnes of corn oil and animal fodder. The HFS will be used in the soft drink industry. Operations are due to start in the second half of 1984.

**Florida sugar production, 1983/84<sup>8</sup>.** — During the 1983/84 season in Florida, 12 million short tons of cane were crushed to yield 1,220,000 tons of raw sugar, down 8% from the 1.3 million tons crop of 1982/83 and 100,000 tons below the pre-season estimate. The shortfall was due to the freeze at Christmas and to a lower sucrose content in the cane than had been expected. The 1983/84 season lasted 144 days compared with 171 days in 1982/83.

**Philippines sugar imports possibility<sup>9</sup>.** — Customs sources in the Philippines report that four ships carrying nearly 80,000 tonnes of sugar are still unloading at a central port in the country, despite denials in the past from the state-owned sugar body that the Philippines, traditionally a major exporter, is importing or buying sugar. The ships are said to be carrying Brazilian and Thai sugar which, after being refined at a nearby mill, will subsequently be re-exported, according to the chairman of the Philippines Sugar Commission. Some cane planters said they believe the sugar will be exported to the US as lower sugar production forecast for the year will not allow the Philippines to meet its quota. The Commission chairman denied this and said that there is already enough sugar for the American market. In January there were about 17 ships with sugar from Thailand and several South American countries destined for Philippine mills.

**New Sierra Leone sugar project<sup>10</sup>.** — Conditions for the erection of a second sugar factory in Sierra Leone have recently been studied by a delegation from the Commonwealth Secretariat. A cane plantation of 10,000 acres is planned in Komrabai Mamila, in the Tonkolili district, and a factory to produce 12,000 tonnes of sugar in its first year, 15,000 tonnes in the second and 18,000 tonnes from the third year.

**GATT and the US/Nicaragua dispute<sup>11</sup>.** — A GATT arbitration panel has issued a report stating that the reduction in Nicaragua's US quota for the 1983/84 fiscal year to 6000 short tons, raw value<sup>12</sup>, contravenes GATT rules. The US has refrained from blocking the adoption of this panel report by the GATT Council but has pointed out that wider issues are involved and, until these differences are resolved, the US quota action will not be revoked.

**New Moroccan cane sugar factory<sup>12</sup>.** — A new cane sugar factory, built by a Japanese consortium, has recently come on stream in Morocco. The factory, which has a processing capacity of 3500 tonnes of cane per day, will be able to produce 45,000 tonnes annually of white sugar. The sugar factories in Morocco, beet and cane, now total 12 and will be able to cover 70% of domestic consumption, which amounted to 646,000 tonnes in 1983.

<sup>1</sup> I.S.O. Stat. Bull., 1984, 43, (2), 12.

<sup>2</sup> F. O. Licht, *International Sugar Rpt.*, 1984, 116, 183-184.

<sup>3</sup> Sakhar. Prom., 1984, (3), 2-5.

<sup>4</sup> C. Czarnikow Ltd., *Sugar Review*, 1984, (1692), 52.

<sup>5</sup> I.S.O. Stat. Bull., 1984, 43, (2), 32-33.

<sup>6</sup> C. Czarnikow Ltd., *Sugar Review*, 1984, (1689), 38.

<sup>7</sup> *Starch/Stärke*, 1984, 36, 103.

<sup>8</sup> F. O. Licht, *International Sugar Rpt.*, 1984, 116, 183.

<sup>9</sup> *Public Ledger's Commodity Week*, March 31, 1984.

<sup>10</sup> F. O. Licht, *International Sugar Rpt.*, 1984, 116, 161.

<sup>11</sup> C. Czarnikow Ltd., *Sugar Review*, 1984, (1692), 52.

<sup>12</sup> I.S.J., 1983, 85, 194, 290.

<sup>13</sup> F. O. Licht, *International Sugar Rpt.*, 1984, 116, 160.

# CANE SUGAR MANUFACTURE

**Cane yard evaporation from short billet chopper harvested/loaded cane in bins.** W. R. van Duyker, H. Lung Kit and P. M. Schorn. *Proc. 57th Ann. Congr. S. African Sugar Tech. Assoc.*, 1983, 64-66. — About half of the cane crushed at Simunye in Swaziland is chopper-harvested and the rest hand-cut, laid in windrows and picked up by chopper/loaders for transfer to bins. After the cane (all in billets 200-300 mm long) is weighed at the factory, it is either crushed directly or stored. Evaporation after weighing poses serious problems for factory control, since the cane pol figure obtained by direct analysis is applied to the measured weight. Investigations on bins containing about 10 tonnes of cane showed that, between 0.89% and 2.46% of the total cane weight was lost during 12 hours (most of which were daylight hours). Apart from the reduction in moisture loss at night, the loss was also affected by the physical state of the cane, and there was a progressive reduction in loss during daylight hours from the first to the third day, probably a result of some protection provided by dried, exposed tissues to the underlying tissues. It is shown that, although a 0.80% evaporation loss means a correction of -0.80% to the cane pol as found by direct analysis, it is equivalent to a change of only -0.02% in the tonnes of pol in cane as determined from the mill balance, and this is the basis for cane payment. The corresponding changes in the pol factor and Brix factor are + 0.79% and + 0.77%, respectively.

**Mhlume Rota-Tipper cane off-loading system.** D. S. Robinson. *Proc. 57th Ann. Congr. S. African Sugar Tech. Assoc.*, 1983, 67-70. — During 1979, increase in chopper-harvested cane delivered to the expanded sugar factory at Mhlume necessitated extension to the cane off-loading station. However, the existing Cameco Port-A-Box system was not able to meet two of the requirements of a new system, viz. ability to cater for growers who preferred different sized containers (because of soil compaction, etc.) and those who wished to handle their own containers, and adjustability for any type of vehicle. Moreover, duplicating the existing system would have entailed encroachment on the storage area for gantry-bundled cane. As a result, a new system was designed which comprises a main support frame, a lifting frame (of 20 tonnes capacity) and a tipping frame. The hydraulically operated system is very compact and completes off-loading in 1½ minutes from when the truck is in position to when it is ready to be driven away. The bin is lifted vertically off the trailer, the contents tipped onto the feeder table and the bin returned to the truck vertically. Only two minor problems were encountered during the first season, and the operation was so successful that a second Rota-Tipper was installed in 1980 to replace the Cameco system; since 1979 the units have handled more than 1 million tonnes of cane. The Rota-Tipper has been patented in Australia, the UK and USA.

**Control of mill carriers.** R. A. H. Chilvers. *Proc. 57th Ann. Congr. S. African Sugar Tech. Assoc.*, 1983, 71-74. Requirements of a good cane carrier control system are given as: ability to maintain the level of cane in the chute of the first mill within narrow limits; reliability; assurance that equipment, torque, speed and power specifications are not exceeded during feeding of the cane through the system; stability under all conditions of cane input; and ability to bring about smooth changes in speed. Such a system is of benefit in improving throughput and extraction, steadying power consumption of the cane knives and shredder, reducing stoppages caused by chokes and overloading, and permitting mill automation. Various means of implementing a carrier control system are described, viz. discrete control, microprocessor control and integrated control. Details are given of the system installed at Mount Edgecombe to replace an earlier scheme which was inadequate. The new system is a totally integrated one which accepts inputs from the two cane knife sets, flicker and shredder as well as the carrier speed signals. The inputs are compared with the various set points and three output signals produced which are fed to the speed references of the carrier speed controllers. Descriptions are given of the salient features of the system, which was installed for the second half of the 1981 crushing season. Initial results showed a dramatic improvement in chute level control, with noticeable reductions in the load fluctuations on the cane knives and shredder. The throughput appeared to be higher than normal, and the anti-stall loop on the first mill turbine (whereby the carriers slow down when the turbine speed falls below the set value, thus reducing the chute level and the load on the turbine and preventing the turbine from stalling) was particularly beneficial when sandy cane was being fed. During the subsequent off-season, a number of small changes were made to the mill; these, together with the carrier control system, permitted a considerable increase in cane throughput.

**Mechanical vapour recompression at Pongola.** G. N. Allan, M. R. Kedian and D. E. Trattles. *Proc. 57th Ann. Congr. S. African Sugar Tech. Assoc.*, 1983, 79-84. — A Bryan Donkin RV 60 single-inlet centrifugal compressor fitted with an inlet guide vane assembly (in which the angle of the guide vane is set pneumatically and used to control machine throughput) was installed at Pongola in 1981/82 for use as a vapour recompression system in association with a new evaporator effect installed specifically for this purpose. The vapour from the vessel is compressed and then re-introduced to the calandria at steam conditions comparable to those of exhaust steam. In four test runs with the unit (rated at 31 tonnes.hr<sup>-1</sup> output) compressing the vapour from 145 to 196 kPa abs, the average output was 3.025 tonnes.hr<sup>-1</sup> (2.52-3.5 tonnes.hr<sup>-1</sup>) at a 0° vane setting and a speed of 9000 rpm. Vane control is regarded as an efficient throughput controller, being much better than a butterfly valve-type suction control since it does not lose energy. Two evaporator balances were calculated on the basis of typical clear juice flow and a refinery melt of more than 90% of the raw sugar. One, based on a compressor output of 3.2 tonnes.hr<sup>-1</sup>, showed a nett saving of 5.5 tonnes.hr<sup>-1</sup> high-pressure steam (equivalent to 0.71 tonnes.hr<sup>-1</sup> coal). Details are given of the capital and maintenance costs of the recompression system.

**Steam balance for the new Felixton II mill.** M. J. Reid and P. W. Rein. *Proc. 57th Ann. Congr. S. African Tech. Assoc.*, 1983, 85-91. — The new sugar factory at Felixton

will have a crushing capacity of 600 tch (with possible extension to 900 tch) and is sited near a paper mill which will take 30% of the total bagasse. This reduction in available fuel is a major constraint in the calculation of a suitable steam economy which is likely to be dependent on exhaust steam rather than on high-pressure steam, since no power will be needed for outside or ancillary use such as with a refinery section or irrigation, and diffusion will be used for extraction — requiring far less power than milling but more low-quality vapour for heating purposes. The effect of vapour requirements of diffusion, juice heating, the boiling house and evaporator on steam economy are discussed, as are the power requirements of the prime movers for the shredders, dewatering mills (driven by steam turbines) and induced-draught fans for the boilers. Methods of reducing steam consumption in a factory dependent on exhaust steam are examined. The requirements at Felixton II can be met by use of a quintuple-effect evaporator and bleeding 2nd effect vapour for use in the vacuum pans and diffuser. The influences of vapour bleeding and of number of evaporator effects (four, five or six) are discussed, and thermocompression compared with mechanical vapour recompression (MVR) in terms of thermal efficiency. MVR is shown to be the most promising means of improving efficiency, while further reduction in high-pressure steam consumption could be achieved by using MVR on continuous vacuum pans. The necessity to allow for flexibility should there be a change in the conditions affecting the future bagasse supply is emphasized.

**Influence on costs of the difference between reality and the plan for cane processed.** C. Freixas C., J. Muñoz A. and C. Rivero C. *Centro Azúcar*, 1982, 9, (3), 15-19 (Spanish). — An analysis is made of the variation in production cost per tonne of raw sugar as a result of differences between planned and actual factors such as cane yield, price, recovery, etc. These govern whether the profit of the sugar enterprise increases or decreases, and it is concluded that agricultural influences exist, so that it is advisable to study the payment for cane on a basis of its pol content.

**Study on the bases for automation of pans.** M. Juanes, I. Díaz and R. Consuegra. *Centro Azúcar*, 1982, 9, (3), 59-73 (Spanish). — A study has been made of the control of final massecuite boiling based on conductivity, and relationships found between conductivity and purity, soluble solids and crystal content. The limitations of conductivity as a control parameter are indicated and a solution is proposed whereby viscosity is measured by a rotational instrument or ultrasonically.

**Methodology of calculation of the strength of crown wheels in sugar cane mills.** J. Moya R., L. César F. and R. Goytisolo E. *Centro Azúcar*, 1982, 9, (3), 75-82 (Spanish). — A method is described for calculating the strength of gearing for a cane mill; the method has been tested and shown to give good accuracy.

**Implementation of recent technological innovations in the sugar industry to help the present fibre and power crisis in India.** N. A. Ramaiah. *Maharashtra Sugar*, 1983, 8, (10), 23-24, 26-27. — The author examines ways in which sugar factories in India can reduce the amount of bagasse burnt and thus provide more for the manufacture of paper while at the same time generating surplus power to be fed into the public grid.

**Working results of vertical crystallizers in the last three seasons in sugar factories.** R. B. Tagare and M. Shanmugasundaram. *Maharashtra Sugar*, 1983, 8, (10), 29-31. Details are given of a vertical crystallizer designed by J. P. Mukherji & Associates which consists of two linked vertical vessels in which the massecuite is cooled by cold water flowing in coils and circulation is provided by stirrer arms between the coils. The massecuite flows down one vessel and up the other, and is reheated in the top third of the second vessel by hot water. Results obtained during three seasons of operation at one factory have indicated a 4-5 unit drop in Nutsch molasses purity by cooling the low-grade massecuite to 40°C, giving a final molasses purity after curing in a continuous centrifugal which is 3 units lower than with conventional horizontal crystallizers. A number of the vertical crystallizers have been installed in various factories.

**Standard specifications for sugar plants — SISSTA's recommendations.** M. Anand. *Maharashtra Sugar*, 1983, 8, (10), 37, 39, 41. — While a technical committee has been formally set up to revise standard specifications for sugar factory plant in India, the author (who is president of the South Indian Sugarcane & Sugar Technologists' Association) suggests some changes to the terms of reference, and lists SISSTA recommendations.

**Polysaccharides and sugar processing. II.** D. F. Day. *Sugar Bull.*, 1983, 61, (23), 8. — The adverse effects of dextrans and gums on sugar factory processes are briefly indicated, covering cane pol determination, clarification, evaporation and boiling. Factory capacity is reduced as a result of the rise in viscosity and fall in crystallization rates caused by dextran, which also affects crystal quality by causing needle grain formation. Low-grade massecuite exhaustion is reduced, and gums in molasses are thought to be responsible for stickiness and occasional gelling in transit.

**Micro-based fuel control system for cell-type furnaces.** K. McGrew. *Sugar J.*, 1983, 46, (3), 36. — An account is given of an automatic control system for bagasse furnaces based on a microprocessor; applied to a 1500-hp Bigelow cell-type boiler, the system allowed operation throughout a crop up to 95% of the rated output. The same scheme is to be used on another boiler, and steps are to be taken to use the microprocessor to control the forced-draught air.

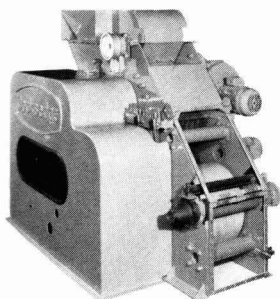
**Continuous vacuum pans for Felixton II.** P. W. Rein. *S. African Sugar J.*, 1983, 67, 366-367. — The pan station at the new factory under construction at Felixton will include two continuous pans, each of 120 m<sup>3</sup>, for A-massecuite, four 76 m<sup>3</sup> continuous pans for B- and C-massecuite (two each) and three 85 m<sup>3</sup> batch pans for supply of seed to the continuous pans. The continuous pans are provided with vertical tube calandrias, which have proved beneficial in terms of heat transfer and circulation; the calandria is positioned in the centre of the horizontal pan, with two downtake areas on each side. The cross-sectional area of the downtakes is large, so giving a good circulation ratio. The pan is divided into twelve compartments, six on each side of a baffle running down the centre. The heating surface:volume ratio in all pans is high, particularly for high-grade massecuite, but it allows the operational cycle to be prolonged even in the event of incrustation or scaling, so that the duration between pan cleaning is increased. The pans are designed to operate at low steam pressures and will use vapour from the 2nd evaporator effect. Conduct-


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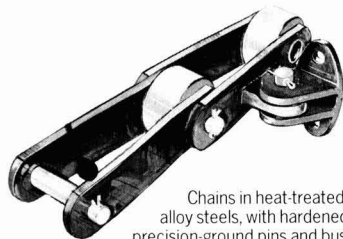
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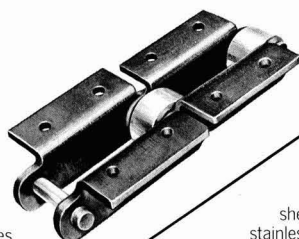
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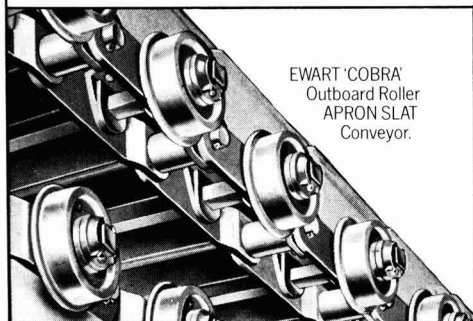
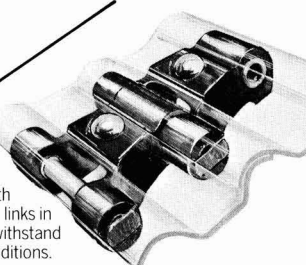
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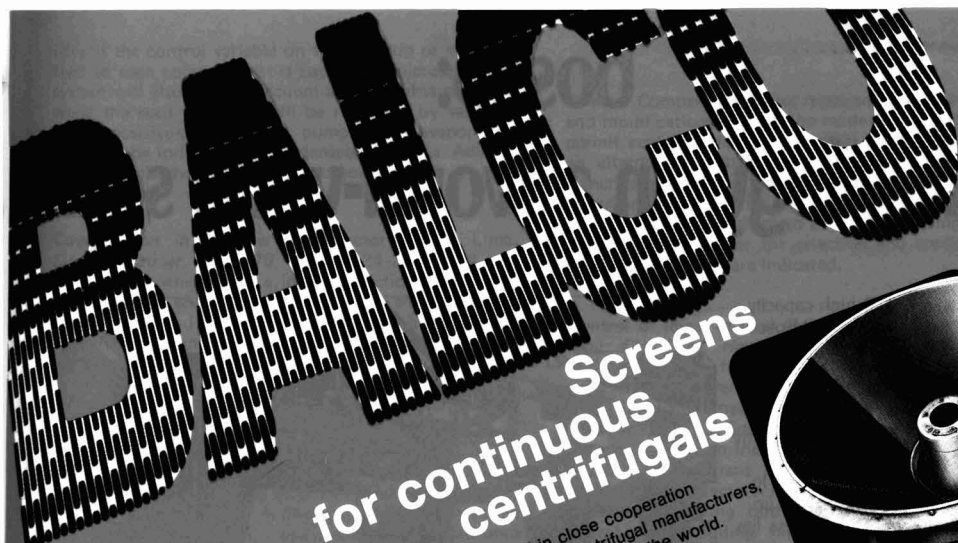
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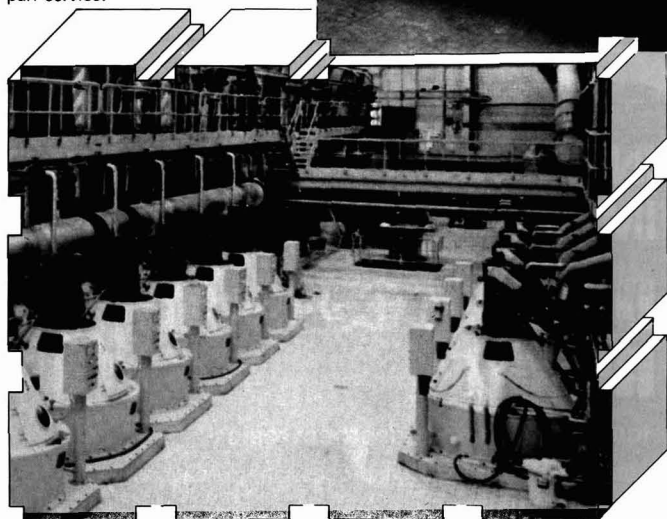
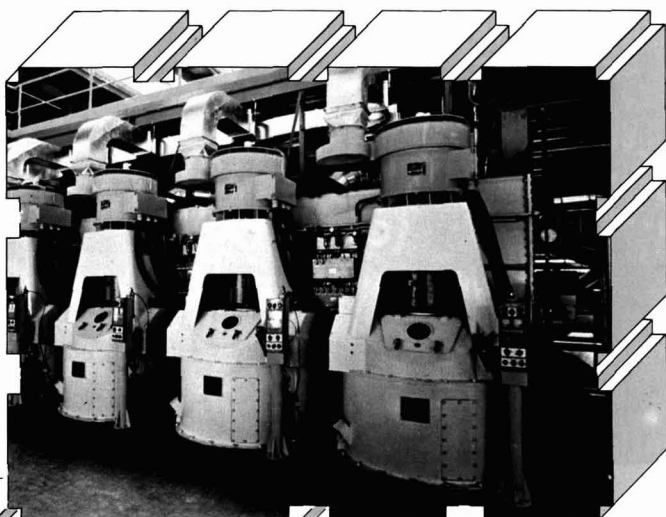
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ivity is the control variable on which syrup or molasses feed to each compartment is based; the microprocessor system will also control vacuum and calandria pressure, while the seed feed rate will be regulated by variable-speed positive-displacement pumps. Pan evaporation rates will be indicated by condensate flow rate. Advantages and disadvantages of continuous boiling are discussed.

**Cogeneration in the raw sugar factory.** J. E. Lima. *Sugar y Azúcar*, 1983, 78, (9), 32, 34-35, 38-39, 42, 44, 46. — Cogeneration, i.e. joint production of electrical or mechanical energy and useful thermal energy, is not a new concept and has gained popularity in industrialized countries since the energy crisis of 1973. Flow diagrams are given of four steam systems that could be used to improve the energy efficiency and allow generation of excess power in a medium-sized Central American raw sugar factory having a crushing rate of 4000 tcd.

**Power reduction in the condensing station of a sugar factory.** R. B. Tagare and T. S. Rao. *Maharashtra Sugar*, 1983, 8, (11), 13-16. — A description is given, with the aid of diagrams, of a multi-jet spray condenser converted to a rain-type condenser (as widely used in Queensland) for use in Indian sugar factories as a contribution to reduction in steam consumption. A concrete example is given of a factory where modifications to the condenser plant brought about a 36% reduction in power consumption.

**Technical and performance results of a NK-1100 continuous centrifugal machine manufactured by National Heavy Engg. Coop. Ltd., Pune.** N. V. Thete. *Maharashtra Sugar*, 1983, 8, (11), 19-24. — Details are given of the NK-1100 continuous centrifugal manufactured by NHEC in collaboration with BMA of West Germany. The performance of the machine is demonstrated by results of trials in which it was used as a C-sugar foreworker at a number of factories, and by normal operating results at two factories. Comparison with data obtained before installation of the centrifugals showed a reduction in molasses losses and in power consumption and increase in plant capacity through decrease in non-sugars recirculation.

**Mill work and imbibition efficiency at individual mills.** P. K. More. *Indian Sugar*, 1983, 33, 169-181. — Mathematical definition of cane milling parameters includes expressions for calculation of reduced extraction and reduced imbibition. Development and application of the various formulae are explained.

**Hydraulic cylinders eliminating downtime in Philippine sugar mill.** Anon. *Sugar y Azúcar*, 1983, 78, (10), 36-37. An outline is given of the efficacy of hydraulic seals in preventing oil leakages from the hydraulic cylinders on the two milling tandems at Canlubang sugar factory. The seals replaced hydraulic leather U-cups and have allowed substantial savings, particularly through elimination of downtime when leakages occurred.

**Coal firing of bagasse boilers.** R. R. Brooks. *Proc. 41st Conf. Hawaiian Sugar Technologists*, 1982, 103-107. While large bagasse-fired boilers have generally been designed for alternative firing with oil or gas, changing patterns of fuel economics and supply may force users to consider coal as alternative fuel, with higher pressures and temperatures possibly required for greater cycle efficiency. The author, vice president of the Foster

Wheeler Company, discusses requirements of new boilers and modifications that can be made to existing plant to permit coal in lump or pulverized form to be burnt as an alternative to bagasse or in combination with it. Concrete examples of Foster Wheeler boilers at selected Hawaiian sugar factories are examined to see what might be done to convert them to bagasse/coal firing. Commonly used criteria for the selection and application of bagasse/coal boilers are indicated.

**Control of losses in final molasses.** J. R. Albert-Thenet. *Proc. 41st Conf. Hawaiian Sugar Technologists*, 1982, 108-113. — The final molasses analyses for Hawaiian sugar factories over the period 1977-81 are tabulated and comparison made between the results for Hawaii, Louisiana, Florida, South Africa, Mauritius and Australia, as well as between the results for 1981 at 14 Hawaiian factories. The extent of molasses losses in Hawaii is discussed, and factors influencing molasses losses are examined, covering both agricultural and processing operations, including high- and low-grade boiling, crystallization, massecuite reheating and centrifugal operation. A number of recommendations are made whereby low-grade work can be monitored and problem areas located.

**Control of filter-cake losses.** T. T. Bennett. *Proc. 41st Conf. Hawaiian Sugar Technologists*, 1982, 114-119. Assessment of filter station performance and filter-cake losses is discussed, and factors influencing losses surveyed, including filter feed handling, cake thickness, drum speed, addition of bagacillo as filter-aid, wash water application, vacuum conditions and filter maintenance. It is pointed out that, since filter-cake is not among the waste products of high sugar losses, the filter station is given less attention than e.g. low-grade operations or the milling train, whereas filter-cake losses can still represent a considerable quantity of sugar over a year. The need to weigh filter-cake in order to obtain an accurate assessment of losses is stressed.

**The implications of grinding trash with cane as it is currently done at Ookala factory.** J. P. Harel. *Proc. 41st Conf. Hawaiian Sugar Technologists*, 1982, 134-139. Problems experienced with grinding of trash with cane at Ookala, including increased fuel consumption, prompted investigations of the procedures and methods used for trash and fibre determinations. Results indicated that there should have been an excess bagasse of at least 8.18 short tons per 100 tons of trash, assuming a calculated fibre % trash averaging 11.9. However, there was no excess bagasse. Comparison of direct and indirect methods of fibre determination involving two samples of prepared cane showed that the levels of trash were twice the estimated figure and that the net cane estimates at the mill had an error of  $\pm 20\%$ . Examination was also made of the adverse effect of soil or mud on the fuel economy; this indicated the possibility that the calorific value of bagasse in wet weather was reduced by the excessive amount of soil in it. It is considered necessary to wash trash properly if it is to be ground with cane; it should first be separated from the cane, then flumed and separated from soil and grit. Bagasse ash should be monitored regularly, but estimations of net cane should be carried out with great caution and should never be used as a basis for factory balances.

# BEET SUGAR MANUFACTURE

**Thoughts on continuous low-grade evaporation-crystallization at Lage sugar factory.** K. E. Austmeyer, H. J. Mosel, A. F. Schulze and B. Ekelhof. *Zuckerind.*, 1983, 108, 927-934 (German). — Existing plant at Lage was incorporated in a continuous system designed by the Braunschweig Institute in collaboration with the factory and BMA. The resultant continuous low-grade scheme comprises a cascade of five pans connected by rotary pumps. Raw sugar run-off is concentrated in the first pan, crystal is formed in the next, and the massecuite boiled in the next three pans. Investigations were carried out on residence time, grain size distribution, the use of seed slurry and heat transfer. On the basis of the results, which are discussed, the use of seed slurry in the second pan was to be replaced (for the 1983/84 campaign) with a footing of 0.15 mm average crystal size produced from a slurry in a separate batch crystallizer; mathematical simulation of residence time distribution and crystal growth with both schemes showed that the use of the footing would improve on unsatisfactory results given by the seed slurry method, the problem being attributed to the lack of uniformity in residence time. The other major modification is the installation of massecuite stirrers above the calandrias in all but the first pan.

**Energy savings in evaporation-crystallization.** P. W. van der Poel, H. P. M. Heere, C. C. Bleyenbergh, M. A. M. de Schutter, N. H. M. de Visser and P. M. T. van Heeschvelde. *Zuckerind.*, 1983, 108, 934-940 (German). — See *I.S.J.*, 1983, 85, 377.

**Monitoring of electricity supply at the Plattling factory of Süddeutsche Zucker-AG.** U. Zimmer and A. Dambach. *Zuckerind.*, 1983, 108, 940-942 (German). — Throughout the year, Plattling factory is subject to six critical phases in which the electricity supply has to be controlled by temporary load shedding — at start-up and switching-off of the factory's own power generators. Monitoring and control are based on a Ferranti Cedrec II load control unit, details of which are given as well as a description of its operation and the economics of the system.

**Liquid-coupled double-tube air preheater unit.** R. Wieser. *Zuckerind.*, 1983, 108, 942-944 (German). — Details are given of the title unit, developed in the USA and the subject of a number of patent applications, which consists of one or more primary heat exchangers to which heat (mostly in the form of flue gases) is admitted, and one or more secondary heat exchangers cooled by combustion air; circulating liquid or gaseous heat carrier is pumped through the system, and the primary heat exchangers take up heat from this and transfer it to the secondary heat exchangers. The unit is designed to act as an economizer with conventional boiler plant.

**Use of plate heat exchangers in the sugar industry.** M. Panzer. *Zuckerind.*, 1983, 108, 945-946 (German).

Advantages of plate heat exchangers, materials used in their construction (including seals), their relative costs and fields of application in the beet sugar factory are discussed. Limitations are imposed by pressure (which should not exceed 25 bar) and temperature (which should not exceed 260°C).

**Heat recovery in the low-temperature range in the sugar industry.** W. Voges. *Zuckerind.*, 1983, 108, 946-947 (German). — The author examines sources of recoverable heat in a sugar factory, starting with the hottest waste (boiler flue gases at about 160-200°C) and covering pulp dryer vapours of about 120°C, carbonatation vapours of about 95°C, pan vapours of about 50°C, water used as machinery coolant (approx. 40°C) and even exhaust air from a low-temperature dryer at about 24-26°C. Each source is discussed in turn, with an account of how to make use of the heat, while particular attention is called to the potential of heat recovery from a low-temperature dryer, which would permit 30% of the total energy requirement of such a dryer to be covered.

**Heat pumps, heat transformers and power-heat couplings and their possible incorporation in sugar factories.** — Suhr. *Zuckerind.*, 1983, 108, 947 (German). — A representative of GEA Luftkühlergesellschaft Happel GmbH & Co., suppliers of energy systems involving multiple energy utilization, describes the potentials of the heat pump (particularly in the GEA Ecoflow heat recovery system) and explains a single-stage heat transformer system which converts low-value waste heat to high-value heat almost without use of mechanical energy.

**Heat transfer recovers process heat from waste heat.** Anon. *Zuckerind.*, 1983, 108, 948 (German). — An experimental Krupp heat transformer system for waste heat recovery is described which has undergone one year's trials at the Dormagen plant of Bayer AG.

**Abrasion of sucrose crystals. I. Crystal-impeller collisions.** L. J. Kuijvenhoven, E. J. de Jong and P. J. Daudey. *Zuckerind.*, 1983, 108, 949-952. **II. Impeller-draft tube interactions.** L. J. Kuijvenhoven, E. J. de Jong and L. C. van Egmond. *ibid.*, 952-955.

**I.** The forces of impact caused by collisions between sucrose crystals and the impeller in a stirred vessel were determined as a function of impeller speed, crystal size and dynamic viscosity. A suspension of sucrose in glycerine or glycerine/ethanol was used in a 1.1-litre laboratory vessel equipped with a 6-bladed propeller carrying a piezoelectric element. The impact forces were found to be very small because of the high viscosity of the suspension and the small difference in density between the crystal and liquid; the maximum force corresponded to a collision energy of  $10^6$  joules. Secondary nucleation as a result of collision between crystals and the propeller blades was found to be highly improbable, so that there would be no need for coating of the blades. **II.** Abrasion of crystals in the space between the tip of an impeller blade and the downtake in a laboratory pan considered as a sugar "mill" was studied; the average grain size distribution of the suspended sucrose crystals after the experiment was used as criterion in preference to formation of new grain. Viscosity, "milling" time, speed of the blade tip, crystal size, concentration of the crystal component and distance between impeller and wall were factors investigated. Increase in viscosity reduced abrasion, almost all of which occurred in the initial stages of the experiment. Higher crystal concentration

rations also reduced abrasion through the consequent increase in viscosity. Abrasion occurred only when the distance between impeller and wall was less than 3 mm. Since, in conventional vacuum pans, the distance is usually greater than 10 mm, secondary nucleation resulting from abrasion is of minor importance.

**The role of storage ponds in sugar factory waste water treatment.** O. V. Demidov, S. G. Bashkurov, I. A. Sidorova, V. S. Dogadov and I. A. Stefanov. *Sakhar. Prom.*, 1983, (10), 32-34 (*Russian*). — The important role played by lagooning as initial stage in effluent treatment is demonstrated by results from one sugar factory where the BOD of waste water was reduced from an average 3510 mg.litre<sup>-1</sup> at the start of September (when it was first discharged to the ponds) to an ultimate 637 mg.litre<sup>-1</sup> at the end of May.

**Modernization of 1st carbonatation juice settlers at Leningrad and Kurganin sugar factories.** V. A. Kholodov, V. V. Kosyakov and A. G. Kavun. *Sakhar. Prom.*, 1983, (10), 42-44 (*Russian*). — Details are given of the modification of gravity settlers at the title factories to upflow types of greater settling rate and daily throughput.

**A graphical method for calculating the amount of water evaporated during concentration of beet sugar factory products.** V. A. Tkachenko and L. F. Stepanova. *Sakhar. Prom.*, 1983, (10), 45-46 (*Russian*). — The graph included in the article is designed to permit easy determination of the amount of water that will be evaporated, given the initial and final Brix of the juice or syrup.

**Industrial data processing in energy optimization of the sugar factory process.** G. Windal. *Ind. Alim. Agric.*, 1983, 100, 441-447 (*French*). — See *I.S.J.*, 1983, 85, 377.

**Application of mathematical simulation to low-grade crystallization.** P. Bonnenfant. *Ind. Alim. Agric.*, 1983, 100, 455-458 (*French*). — Application of mathematical modelling is demonstrated in the case of low-grade crystallization. The system is divided into a universal scheme of micro-kinetic elements (sugar solubility in pure and impure solution, mother liquor viscosity, massecuite consistency and crystallization rate) and a scheme of macro-kinetic elements that is specific to each crystallizer (cooling and reheating time, whether pre-spinning is used or not, potential heat transfer, residence time distribution, maximum permissible viscosity of massecuite in the crystallizer and pumps, and maximum permissible viscosity of mother liquor during centrifuging). A program was developed on the basis of the various parameters, and examples are given of its application to calculation of crystallizer performance.

**Rationalization of the molasses exhaustion process.** K. Wagnerowski. *Ind. Alim. Agric.*, 1983, 100, 463-466 (*French*). — See Wagnerowski. *I.S.J.*, 1983, 85, 211, 214, 215, 249; 1984, 86, 19.

**The kinetics of impure syrups applied to industrial sucrose crystallization.** V. Maurandi and A. Rossi. *Ind. Alim. Agric.*, 1983, 100, 469-474 (*French*). — See *I.S.J.*, 1983, 85, 377.

**Developments in heat consumption in the sugar industry.** J. Bozec. *Ind. Alim. Agric.*, 1983, 100, 477-480 (*French*). Mention is made of the contribution made by Soc. Fives-Cail Babcock to reduction of heat consumption in

sugar factories through studies at its research centre. The result is a new energy scheme, and this is compared with a conventional scheme for a factory using traditional processes, including a 3-massecuite boiling system with remelting of B- and C-sugar. The comparison does not include ancillary stations and processes such as a distillery, syrup storage or sugar pre-conditioning; pulp drying has not been studied in detail, but merely allowed for in calculations of electricity consumption. Details are given of process conditions and equipment, and it is shown that a considerable energy saving is possible, with rapid amortization of the capital equipment, installation of which may be done progressively in accordance with a gradual reduction in energy consumption.

**New systems of deliming in the sugar industry.** X. Lancron and P. Printemps. *Ind. Alim. Agric.*, 1983, 100, 501-505 (*French*). — See *I.S.J.*, 1984, 86, 41-44.

**Automation of 1st massecuite boiling at Eppeville sugar factory.** D. Vigneurt and C. Beurrier. *Ind. Alim. Agric.*, 1983, 100, 509-513 (*French*). — At Eppeville, 1st massecuite boiling in a station of seven pans (four of them equipped with massecuite stirrers) is controlled by a Sereg-Schlumberger computerized system, the Modumat 800. Control is based on massecuite consistency, which is measured by a ViscoVib vibrating-needle sensor, while other parameters measured include steam pressure, vacuum, massecuite level, the level in the massecuite feed troughs and stirrer motor speed. The benefits of the system and its mode of operation are described.

**Economy in beet sugar filtration.** H. Hagen. *Sugar y Azúcar*, 1983, 78, (9), 64, 67. — Details are given of the design, operation and performance (during the 1977 campaign) of the Hoesch filter-press installed at Appeldorn sugar factory in West Germany. (See also Weidner: *I.S.J.*, 1979, 81, 217.)

**Operational measurement of 1st carbonatation parameters at Modrany experimental sugar factory.** E. Sarka and P. Kolar. *Listy Cukr.*, 1983, 99, 231-235 (*Czech*). — Laboratory data were confirmed by measurement of normal factory parameters in 1st carbonatation, including the effect of unfiltered juice recycling. The effects of recycled juice quantity, reducing matter content and preliming temperature on the initial settling rate and filtration coefficient of the juice were determined and their correlations established.

**Measuring and control techniques at Deir es Zor sugar factory of 4000 tonnes/day slicing capacity.** J. Zaruba. *Listy Cukr.*, 1983, 99, 235-240a (*Czech*). — Measuring and control techniques used in the various process stations at Deir es Zor sugar factory in Syria are described with the aid of diagrams.

**Novel regeneration systems for ion-exchange resins in the sugar industry.** W. Pannekeet. *Chem. & Ind.*, 1983, 821-824. — A brief survey is presented of ion-exchange processes used in beet sugar manufacture, covering thin juice deliming and demineralization and molasses sugar recovery by the Quentin process, a modification of which is proposed in which a strongly acidic cation-exchange resin in Ca<sup>++</sup> form is used instead of a strongly acidic cation-exchange resin in Mg<sup>++</sup> form as used in the conventional process. The resin is regenerated with a Ca saccharate solution prepared from molasses and CaO



Advantages over the use of the Mg-form resin regenerated with  $MgCl_2$  are given as (1) no waste water problems, since the regeneration effluent can be returned to the molasses after carbonatation and filtration (to remove excess  $CaO$ ) and evaporation, and (2) savings in costs of chemicals. On the other hand, tests in which IMAC C16P resin in  $Mg^{++}$  form was regenerated with  $MgCl_2$  showed that this was more efficient (giving 90-95%  $Mg^{++}$  ion exchange against  $K^+$  ions) than regeneration with  $Ca$  saccharate (giving only 45%  $Ca^{++}$  ion exchange); this was attributed to a decrease in the  $Ca^{++}$  ion activity in the  $Ca$  saccharate solutions and competition from  $K^+$  ions in the molasses during regeneration. The potential use of  $Ca$  saccharate as regenerant for weakly basic anion-exchange resins used for juice demineralization is also discussed.

**Automation of the crystallization process at Güstrow sugar factory.** G. Möller. *Lebensmittelind.*, 1983, 30, 368-370 (German). — Details are given of the Polish automatic boiling control scheme installed at Güstrow in East Germany for a 4-massecuete system in the beet campaign and a 6-massecuete system in cane raw sugar processing. Massecuete level and viscosity are used as control parameters for A-massecuete boiling, while B- and C-massecuete boiling control is based on conductivity. Results for the 1982/83 beet campaign and raw sugar processing in 1983 are compared with those for the previous season, and demonstrate the benefits of the control scheme in terms of reduced molasses losses and improved sugar quality (particularly reduced colour content) as well as lower energy consumption and re-boiling losses during the beet campaign.

**Separation of reheat steam in a MTIPP film evaporator.** V. D. Lazarev. *Izv. Vuzov, Pishch. Tekh.*, 1983, (4), 76-80 (Russian). — Investigations on the use of centrifugal entrainment separators in association with film evaporators are reported. From the results, an equation was derived for calculation of optimum separator dimensions. Basic operational parameters obtained under optimum conditions are tabulated for a separator handling vapour from each of four effects.

**Change in the basic indices of fractional composition of crystals during mass crystallization.** B. V. Kuz'menko, V. O. Shtangeev, I. S. Gulyi and N. I. Shtangeeva. *Izv. Vuzov, Pishch. Tekh.*, 1983, (4), 83-85 (Russian). Mathematical treatment of the boiling process in both continuous and batch vacuum pans has shown that the use of forced recrystallization leads to increase in the length of a crystal of average mass, particularly at the end of the process. Such recrystallization, brought about by temperature fluctuation when the pressure of the heating steam is varied, has a considerable positive effect on the fractional composition of the crystals.

**The performance of down-flow boiling tubes in evaporators.** I. I. Sagan', I. S. Cherkunov and V. A. Yarmolenko. *Izv. Vuzov, Pishch. Tekh.*, 1983, (4), 92-94 (Russian). The performance of the boiling tubes in a falling-film, ring-type evaporator was investigated with juice of 30° and 65°Bx. Results showed that the heat transfer rate was governed by the vapour formation rate and degree of wetting, which in turn depended mainly on heat flow rate and the thermo-physical properties of the juice. An equation was derived for use in calculating the heat transfer coefficient for wall to juice film in the tubes.

**Optimum limit of cation exchanger regeneration.** P. P. Zagorodnii, K. P. Zakharov, S. S. Fil'chashkin, V. Z. Semenenko and T. P. Trifonova. *Izv. Vuzov, Pishch. Tekh.*, 1983, (4), 88-91 (Russian). — Mathematical investigation of regeneration of cation-exchange resin used in  $Ca^{++}$  form to replace the  $K^+$  and  $Na^+$  ions with  $Ca^{++}$  was aimed at establishing optimum quantities of resin and regenerant required to give maximum efficiency. Under the conditions examined, the optimum quantity of regenerant was  $0.67 m^3$  of 2N  $CaCl_2$  per  $m^3$  of resin.

**The diffusion process when press water is recycled.** M. I. Daishev and O. V. Gorodetskii. *Izv. Vuzov, Pishch. Tekh.*, 1983, (4), 98-101 (Russian). — A mathematical investigation of diffusion with press water recycling is reported. The resultant theory has been applied to evaluation of the effect of the degree of pulp pressing on the quantity of residual sugar in the exhausted cosettes and in the pressed pulp.

**Energy recovery from the evaporator stations.** L. L. Neville. *Sugar J.*, 1983, 46, (4), 5-8. — Details are given of modifications to the evaporator stations and associated uses of bleed vapour at Hamilton City, Worland and Brawley factories of Holly Sugar Corporation, and similar changes being undertaken at Hereford and Torrington factories are mentioned. The modifications have permitted a 19% reduction in fuel consumption and allowed the money spent to be recovered in less than a year.

**Heavy oil combustion in industrial boilers. New combustion catalysts: Energam series 700.** Anon. *Sucr. Franç.*, 1983, 124, 421-426 (French). — Details are given of the Energam series of catalysts produced by Gamlen Europe SA in collaboration with the Total oil refining group; suitable for boilers of any capacity, Energam increases steam output while reducing emission of unburnt solids and decreasing the excess of combustion air.

**Modern methods of sulphur combustion in sugar processing.** B. Winting. *Sugar y Azúcar*, 1983, 78, (10), 64, 66-67. — Details are given of a sulphur burner, the SBM-250, developed by AB Cellico, a member of Alfa-Laval AB. The unit burns liquid sulphur at a nominal throughput of 5 tonnes per day, although it can handle as little as 250 kg per day, producing a gas containing 19-20%  $SO_2$  v/v. The burner normally operates at a pressure of 2-3 psig but can also function effectively at up to 42 psig if required.

**Results of tests on beet knives hardened by arc welding.** V. M. Revutskii, V. A. Bakhtiarov, I. S. Tkach, M. Ya. Drai and L. T. Denisenko. *Sakhar. Prom.*, 1983, (11), 17-19 (Russian). — Electrode alloying of beet knives was found, in comparative tests at a sugar factory, to increase their wear resistance considerably in contrast to untreated knives.

**The diffusion coefficient of sugar in beet when feed water is treated by electro-coagulation.** A. Ya. Romanyuk, A. A. Lipets and A. I. Fel'dman. *Sakhar. Prom.*, 1983, (11), 19-21 (Russian). — Condensate for use in diffusion was pretreated with  $SO_2$  (with or without electro-coagulation using aluminium plates as electrodes<sup>1</sup>) or with aluminium sulphate and fed into a laboratory unit to determine the effect of pretreatment on the diffusion coefficient of sugar in cosettes. Highest values were obtained after electro-coagulation and lowest when pretreatment involved only  $SO_2$ .

<sup>1</sup> Romanyuk et al.: *I.S.J.*, 1976, 78, 281.



# LABORATORY STUDIES

**Transfer of aqueous sucrose solutions through semi-permeable membranes.** E. A. Tsapyuk and D. D. Kucheruk. *Ukrain. Khim. Zhurn.*, 1983, 49, 824-828 (Russian). — Sucrose solutions were used in investigations of reverse osmosis involving two membranes: an unnamed gelatin type and a UAM-50 acetyl cellulose membrane designed for ultra-filtration. Results showed that changes in the values of hold-up and bulk flow were a function of sucrose concentration (0.2-20%), working pressure (1-10 MPa) and temperature (298-345°K) and were evidence of the substantial effect of bound moisture, i.e. that localized at the hydrophilic centres of the dispersed phase and having a reduced dissolving capacity, on the mechanism of semi-permeability. It had earlier been suggested that the presence of bound water in the very small pores of a membrane would have a decisive effect on its properties.

**Conversion of open chain structures of monosaccharides into the corresponding Haworth forms.** D. M. S. Wheeler, M. M. Wheeler and T. Wheeler. *Sugar J.*, 1983, 45, (14), 17-18. — Representation of the structure and configuration of monosaccharides by the Haworth formula and as chair forms as opposed to the cyclic form of the Fischer formula is explained, and conversion of the open-chain structure to the Haworth form described.

**Methods of analysis of dextran in cane juice and other sugar house products.** S. Bose and L. Singh. *Sharkara*, 1978, 17, (1-4), 22-26. — A survey is presented of methods used to determine dextran, including turbidimetric procedures, polarimetric measurement after clarification, colorimetric methods, determination based on increase in viscosity and the immunological assay method. Brief mention is also made of the application of HPLC.

**The haze test for dextran.** D. F. Day. *Sugar Bull.*, 1983, 61, (21), 17. — While dextran determination by the haze method is currently the best means available for factory control and has the advantage of rapidity and high sensitivity, it is less suitable for specific determination of dextran caused by *Leuconostoc mesenteroides* since it also detects other high-M.W. alcohol-precipitable polysaccharides. For accurate analysis, starch needs to be removed e.g. by an enzymatic procedure. However, this must be done carefully since incomplete removal may leave macro-dextrins which are efficient haze producers; they produce no blue colour with iodine and so are not detected as starch.

**Micro-analysis of sugar using a chromophoric labelling reagent.** Y. Chang and C. Pan. *Shengwu Huaxue Yu Shengwu Wuli Xuebao*, 1982, 14, (2), 165-169; through *Anal. Abs.*, 1983, 45, Abs. 3C24. — The use of NN-dimethylazobenzene-4,4'-diamine as a labelling reagent for the formyl groups of sugars in the presence of  $\text{NaBH}_3\text{CN}$  is described. The derivatives thus formed can be separated by two-dimensional chromatography on

polyamide sheets and located directly as yellow spots, which turn green and then violet-blue on exposure to HCl vapour. Detection of monosaccharides is possible in the range 0.1 to 1 nmol, and the reducing end of an oligosaccharide can be determined. There is positive correlation between the migration rate and M.W. of a labelled oligosaccharide, so that the degree of polymerization can be estimated.

**Surface electrical properties of calcium carbonate.** L. M. Khomichak, S. P. Olyanskaya, N. A. Arkhipovich and O. L. Alekseev. *Izv. Vuzov, Pishch. Tekh.*, 1983, (3), 25-27 (Russian). — Knowledge of the electrokinetic properties at the surface of  $\text{CaCO}_3$  particles is important for an understanding of the adsorption process. The effect of the double electric layer formed at the solids-liquid interface on the reproducibility of results obtained in determination of the zeta-potential of  $\text{CaCO}_3$  particles was investigated. Results showed that, in a  $\text{Ca(OH)}_2$  solution to which  $\text{CaCO}_3$  had been added to give a solids:liquid ratio of 1:5, the charge of the electric layer and the quantity of water bound by it rose, as did the zeta-potential, with increase in solution pH to 11.0. The studies also demonstrated the applicability of the electro-osmotic transfer method irrespective of how the  $\text{CaCO}_3$  was prepared.

**Determination of ultramarine in beet sugar.** E. Andrzejewska. *Rocz. Panstw. Zakl. Hig.*, 1982, 33, (4), 327-329; through *Anal. Abs.*, 1983, 45, Abs. 3F12. — Ultramarine blue (I) (C. I. Pigment Blue 29) was extracted from the sample (30 g) with water and purified by column chromatography on polyamide, then detected and semi-quantitatively determined by thin-layer chromatography on MN 300 cellulose. Use of three solvent systems for TLC development allowed the identification of I and two dyes not permitted for blanching sugar, viz. indigotin and Brilliant black BN (C. I. Food Black 1). Down to 0.12 mg.kg<sup>-1</sup> of I could be detected in sugar, and the recovery of I was approx. 80%. Details of the procedures are given.

**Determination of raffinose in molasses.** M. Polacsek, I. Szep and L. Vamos. *Elelmiszerciszgalati Kozl.*, 1982, 28, (1-2), 55-61; through *Anal. Abs.*, 1983, 45, Abs. 3F13. — The sample was clarified with  $\text{ZnSO}_4$  and  $\text{K}_4\text{Fe(BN)}_6$  and the raffinose was determined by a two-step enzyme system; galactose was split off from raffinose with  $\beta$ -galactosidase at pH 4.6 (acetate buffer) and the galactose was caused to react with  $\text{NAD}^+$  in the presence of galactose dehydrogenase at pH 8.6 (Tris buffer) to give NADH, which was determined spectrophotometrically at 340 and 365 nm.

**Study on the solubility of calcium and magnesium aconitates in different media and at different temperatures.** M. Nápoles F. *Centro Azúcar*, 1982, 9, (2), 41-49 (Spanish). — Formation of aconitates of Ca and Mg occurs during sugar manufacture and their elimination during clarification is important. As a contribution to achieving this, a study has been made of their solubility at different temperatures in the presence of other salts and of sugar. The data are tabulated for Ca aconitate (Mg aconitate proved soluble) and show that its solubility was scarcely affected by sugar content between 0 and 25% but fell as the sugar content rose to 70%. Solubility was increased by the presence of NaCl or KCl and by increase in temperature. It was decreased by the presence of aconitic acid, of  $\text{K}_2\text{HPO}_4$  and by increasing the  $\text{CaCl}_2$  content.

**Adaptation of the molecular sieve method for the determination of colloiddally dispersed substances in the intermediate products of the refinery.** J. Castellanos E., R. Fajardo G., H. Estacio L. and C. Egido V. *Centro Azúcar*, 1982, 9, (2), 95-103 (Spanish). — The technique of Darias *et al.*<sup>1</sup> as used for separation of colloidal matter in raw sugar has been adapted to refinery liquors. It employs gel filtration using Sephadex G-50 but using higher concentrations — 50°Bx vs. 12°Bx. Details of the method are described.

**Moisture determination of bagasse and shredded cane using a microwave oven.** T. L. Excell and M. F. S. Koenig. *Proc. 57th Ann. Congr. S. African Sugar Tech. Assoc.*, 1983, 30-32. — Comparative tests were conducted on drying of bagasse and shredded cane samples in a microwave oven as a possible alternative to the use of hot air ovens (as currently employed by the Sugar Industry Central Board cane testing laboratories). By the conventional method, a 300-g sample of bagasse or a 500-g sample of shredded cane has to be dried for 1 hour at 105°C; the ovens cannot be maintained at this temperature easily over long periods of time and need frequent maintenance. Results showed that only 35 minutes at full power (650 W) was needed to reduce the moisture content by a maximum amount in the case of four 50-g samples dried simultaneously in the microwave oven, and the mean difference between the values obtained in 36 tests using the microwave and drying ovens was only —0.08%. Simultaneous drying of four 100-g shredded cane samples in the microwave oven took 1 hour (17 min at full power, 5 min at 70% full power and 38 min at 30% full power); full power could not be used for the entire drying time because of charring of the sample before attainment of maximum moisture loss. The mean difference between the values obtained in the microwave and hot air ovens in 37 tests was +0.19%.

**A quality assurance program for the weekly analysis of sugars in cane final molasses by gas chromatography.** K. J. Schäffler and C. M. J. Day-Lewis. *Proc. 57th Ann. Congr. S. African Sugar Tech. Assoc.*, 1983, 33-37. — In 1982-83 a chemical control program for South African raw sugar factories was officially implemented at the Sugar Milling Research Institute, whereby the SMRI undertakes analysis of weekly molasses samples for sucrose, fructose and glucose by gas-liquid chromatography. The system necessitates a quality assurance scheme for monitoring errors in GLC analysis so as to permit them to be kept within reliable limits and ensure a high probability of compliance with acceptable quality standards. Errors may arise as a result of silica build-up in the detector, septum leaks, falls in carrier gas pressure, vent line blockages, incorrect integrator settings and low or very high sugar levels in the samples. Multilevel calibration incorporated in the quality control program uses pure sugar solutions of known concentration and molasses control samples to evaluate precision on the basis of repeatability and accuracy on the basis of target values obtained for the control molasses samples; it provides both a statistically reliable response factor for the sugars and is very useful in locating drift or systematic bias. The results obtained are discussed, and it is pointed out that the quality assurance scheme described is merely a prototype and will be improved in future milling seasons.

**The effect of reduced frequency of sampling and analysis at sugar mill laboratories.** P. Mellet and A. Dunsmore. *Proc. 57th Ann. Congr. S. African Sugar Tech. Assoc.*, 1983, 52-55. — A survey was carried out at 15 sugar factory laboratories to see if it would be possible to rationalize their work in regard to frequency of sampling and analysis without adversely affecting factory control. It was found that the number of analyses could be reduced by 25% by merely cutting down on what was not strictly necessary, while a further 12-15% reduction could be achieved if the day shift performed most of the analyses while the other two shifts concentrated on essential shift analyses and compositing of samples. This would reduce the labour force requirement to about 13 per day for raw sugar factory control plus two where a refinery section was appended, instead of a laboratory staff that currently varies from 16 to 35. Details are given of a suggested analysis program which would save some R 20,000 per year, allowing for an initial outlay on automatic pH, temperature and Brix recorders and sampling equipment.

**Variation during a sugar season of the wax contents of final molasses and raw sugar.** N. Morffi, E. R. Fleites, M. Darias and I. Valentin. *Centro Azúcar*, 1982, 9, (3), 83-88 (Spanish). — Thirteen samples of molasses and raw sugar, taken at 10-day intervals during a season, were analysed for wax content by Soxhlet extraction with carbon tetrachloride. From the results it is seen that the content in molasses was always higher, and that there was a linear relationship with a relatively high correlation coefficient, as well as significant differences between both contents. The waxes extracted were examined and found to correspond in respect of melting point and I.R. spectra.

**High-performance liquid chromatography of sugars.** J. Copikova, H. Hanzlova and S. Vozka. *Prum. Potravin*, 1983, 34, (5), 243-246; through *Anal. Abs.*, 1983, 45, Abs. 4F24. — The sample (1-5 g) of e.g. molasses was dissolved in or extracted with water and an internal standard (xylose, trehalose or ribose) added. The extract was treated with Carrez solution I and II and with Herles reagent, then filtered before analysis by HPLC on a stainless-steel column (25 cm x 6 mm) containing Separon-NH<sub>2</sub> (10 µm), with degassed aqueous 75% acetonitrile or acetonitrile:2mM Na acetate (4:1) as mobile phase (1.5-2.2 ml.min<sup>-1</sup>) and detection with a refractometer at 25°. Results obtained by HPLC and by conventional methods are tabulated. It is recommended that an integrator be used with the refractometer.

**Use of ion-selective electrodes to determine ash elements in beet.** V. A. Knyazev, S. N. Kalina, L. I. Chernyavskaya, R. S. Sova and V. N. Koshevaya. *Sakhar. Prom.*, 1983, (10), 50-53 (Russian). — A method is described which is based on the use of a system of selective electrodes to determine the K<sup>+</sup> and Na<sup>+</sup> concentrations in aqueous beet extract in terms of the change in e.m.f. after addition of a known quantity of standard solution of the corresponding ion. Comparison with values obtained by flame photometry showed that those given by the selective electrodes were lower by 2.5% in the case of K<sup>+</sup> and by 1.4% for Na<sup>+</sup>. Details are given of activity coefficients in undiluted and diluted extract and pure electrolyte solutions (2-, 4- and 8-fold dilution), and Na<sup>+</sup> and K<sup>+</sup> contents in specific beet varieties are also tabulated.

<sup>1</sup> *Centro Azúcar*, 1973, 1, (1), 63; 1974, 2, (1), 25; 1976, 4, (3), 8; 1978, 5, (2), 41, 53; *Revista ATAC*, 1976, 35, (3), 12; (5), 10; 1977, 36, (1), 55.

# BY-PRODUCTS

**Low-temperature drying in conjunction with traditional pulp drying.** W. Kunz. *Zuckerind.*, 1983, 108, 868-870 (German). — Low-temperature pre-drying of beet pulp is discussed as a means of reducing sugar factory energy consumption. Factors examined include the requirements in regard to the pulp and the air used for drying, aims and requirements of a suitable low-temperature dryer, and major design parameters. A description is then given of a Swiss Combi W. Kunz AG band dryer which aims to meet all the requirements that are set out; a diagram shows one comprising four levels of belts, one above the other, designed to evaporate 20 tonnes.hr<sup>-1</sup> water and under construction for Offstein and Zeil sugar factories. The effects of a pre-dryer on the performance of an existing drum dryer are discussed.

**Enzymatic preparation of crystalline glucose from waste cellulosic materials.** I. Kusakabe, T. Yasui and T. Kobayashi. *J. Ferment. Technol.*, 1983, 61, (2), 163-170; through *S.I.A.*, 1983, 45, Abs. 83-1224. — Cellulosic wastes tested included beet pulp. Crystalline glucose was prepared from the saccharification residue after enzymatic removal of hemicellulose. In a typical procedure, 200 g (dry solids) was pretreated with 2% NaOH solution at 30°C for 6 hr, after which the xylan was removed by means of xylanase. The residue, containing 80.8 g dry solids, was hydrolysed with 16 g Cellulase AP from *Aspergillus niger* at 50°C and pH 4-4.2 for 4 days. The resulting solution, containing 53.0 g total reducing sugars, was decolorized, desalted and concentrated. Crystalline glucose (41.4 g) was obtained. The molasses was shown to contain oligosaccharides such as gentiobiose and xylobiose.

**Production of SCP and cellulase by *Aspergillus terreus* from bagasse substrate.** S. K. Garg and S. Neelakantan. *Biotechnol. Bioeng.*, 1982, 24, (1), 2407-2417; through *S.I.A.*, 1983, 45, Abs. 83-1298. — In fermentation of 1.0% untreated bagasse with *A. terreus* GNI under optimum conditions, protein recovery and cellulase production increased with time up to 7 days, but the rates were maximum during the first 3 days. There was an initial lag of up to 2 days in the utilization of cellulose, owing to utilization of water-soluble carbohydrate present in untreated bagasse. The total biomass (fungal mycelium + undegraded bagasse) recovered was 1020 mg/g bagasse substrate, and it contained 14.3% crude protein; when alkali-treated bagasse was used, corresponding figures were 820 mg/g and 20.6%. Thus, pre-treatment of the bagasse led to more protein being produced, but in order for treatment of the resulting effluent to be economical, not only the protein but also the cellulase produced must be considered as end-products.

**Solid-state fermentation of cellulase production by *Pestalotiopsis versicolor*.** M. N. A. Rao, B. M. Mithal, R. N. Thakur and K. S. M. Sastry. *Biotechnol. Bioeng.*, 1983, 25, (3), 869-872; through *S.I.A.*, 1983, 45, Abs.

83-1299. — The above fungus was grown on various cellulosic substrates. Cellulase activity was maximum when the substrate was bagasse; activity/unit volume was maximum when the moisture content, by volume, was 8-10 times the weight of substrate, but the total activity was lower than in liquid culture.

**Upgrading beet pulp by *Trichoderma harzianum* cultivation in liquid and solid medium.** S. Roussos, J. L. Garcia and M. Raimbault. *Ind. Alim. Agric.*, 1983, 100, 449-452 (French). — *T. harzianum* on a suitable substrate uses cellulose as carbon and energy source, for which it produces cellulases; the cellulose is converted to glucose, while the protein content of the medium is increased by virtue of the bacterial growth. Studies were conducted on cultivation of the organism on beet pulp in solid and liquid fermentation under controlled conditions. Solid fermentation yielded a maximum of 29.0% w/w protein on substrate (dry) after 47 hours, while liquid fermentation yielded 24.5% after 30 hours. The initial cellulose content was approximately halved. The end-product may be used as animal fodder or as cellulase source.

**Study of correlations between vinasse physical properties.** A. Duarte-Coelho, E. Dumoulin and B. Guerin. *Ind. Alim. Agric.*, 1983, 100, 483-487 (French). — Parameters of importance for multiple-effect evaporation of vinasse were investigated. Linearity was established between dry solids in the range 10-55% and density, refractive index and thermal capacity, while boiling point elevation rose exponentially with dry solids.

**Chemical reduction of the acetaldehyde present in ethanol during distillation.** J. Guerin, P. O. Cogot and P. A. Mimault. *Ind. Alim. Agric.*, 1983, 100, 515-518 (French). — During ethanol manufacture from beet and molasses in French distilleries, a small amount of acetaldehyde has occurred in the alcohol, which has necessitated adoption of one of a number of possible measures. In the USA, the problem of excessive acetaldehyde in rectified alcohol has been overcome by using sodium borohydride, which reduces aldehydes and other carbonyl constituents to their corresponding alcohols. Laboratory and factory trials were carried out in which NaBH<sub>4</sub> proved successful in decreasing the acetaldehyde content to below 1 g.hl<sup>-1</sup> pure alcohol.

**Tongaat Milling plan to beat fodder shortage.** Anon. *S. African Sugar J.*, 1983, 67, 357. — Mention is made of the decision by Maidstone factory to buy cane leaf trash and tops and bale these in the field for sale to farmers as animal fodder in an attempt to ease the severe shortage created by the South African drought. The material, in 20-25 kg bales, has a nutritive value similar to that of coarse dry grass hay and is reasonably palatable, although it provides only a limited amount of protein.

**Agricultural spraying of sugar factory waste water.** J. Abraham, J. Keller, H. Krolap and B. Kurth. *Lebensmittelind.*, 1983, 30, 371-374 (German). — Details are given of experimental spraying of sugar factory effluent on land used to grow rye and oats. After investigation of a number of important factors, it was decided that the scheme could be adopted as a normal routine and extended to sugar beet fields; it resulted in a 5% increase in yield by comparison with the average levels in other beet areas. Requirements of waste water treatment before spraying and average mineral composition of the effluent are indicated.

**Biostil — fermentation of molasses and cane juice using continuous fermentation.** L. Garlick. *Sugar J.*, 1983, 46, (4), 13-16. — Details are given of the Alfa-Laval continuous fermentation process in which ethanol is continuously removed as it is produced so as to minimize its inhibitory effect on the process and permit acceptance of more concentrated feedstock.

**Residual effect of press mud cake (PMC). II. On chemical properties of soil and uptake of nutrients in sugar cane (*Saccharum officinarum* L.).** S. P. Patil and S. P. Kale. *Indian Sugar*, 1983, 33, 91-95. — Studies are reported in which application of 25 tonnes/ha<sup>1</sup> filter-cake (supplying 300, 970 and 364 kg/ha<sup>1</sup> of N, P<sub>2</sub>O<sub>5</sub> and K<sub>2</sub>O, respectively) increased the levels of organic C, total N, available P<sub>2</sub>O<sub>5</sub> and available K<sub>2</sub>O in the soil at harvest time; the C:N ratio decreased slightly by comparison with that obtained by applying 400 kg/ha<sup>1</sup> N alone or with 170 kg/ha<sup>1</sup> each of P<sub>2</sub>O<sub>5</sub> and K<sub>2</sub>O (the recommended dose). The levels of all three nutrients increased in the cane and cane yield was substantially raised by PMC application.

**Method for processing whole cane and sweet sorghum into fuel alcohol and electric power.** F. le Grand. *Sugar y Azúcar*, 1983, 78, (10), 57-58, 60-61, 94-95. — A scheme for alcohol fermentation and distillation is described in which comminuted cane or sorghum is used as raw material, while the bagasse is recovered, dried and gasified. The process, each stage of which is based on known technology, is calculated to produce about 72 litres of fuel ethanol and 442 kWh per tonne of cane or sorghum. The economics of the scheme, which is the subject of patent applications, are discussed.

**Increased utilization of sugar factory waste heat in low-temperature drying.** P. Valentin. *Zuckind.*, 1983, 108, 1025-1033 (German). — The low-temperature beet pulp drying scheme introduced at Offstein and Zeil sugar factories is examined and aspects of energy consumption are discussed in relation to waste heat utilization. The scheme described is designed to utilize waste heat from various sources, including pan and carbonatation vapours, condensate and vapour from high-temperature pulp drying which follows the low-temperature process. The low-temperature dryer is a Swiss Combi perforated band system described earlier<sup>1</sup>. The aim is to increase the dry solids of the pressed pulp from 30% to 50-60%, with a final rise to 90% in high-temperature drying. The effects on energy consumption of (1) use of circulating air in low-temperature drying, (2) recovery of heat from the low-temperature dryer exhaust air, and (3) use of evaporator condensate for low-temperature drying together with additional power generation are discussed. Factors of importance for the design parameters of a low-temperature system are considered, and it is shown that a considerable part of the power consumed is used to feed the large quantities of air required, with pressure fall in the air heaters having an additive effect. The air flow rate is kept to a minimum in order to avoid excessive dust in the exhaust air, thus avoiding the need for special dust separators and hence reducing energy consumption. On the other hand, the large amounts of air necessitate introduction of special aerodynamic means of minimizing the pressure losses — from the design viewpoint this is of greater significance than drying rate, pulp properties, bed thickness and residual pulp moisture. Increase in the air flow rate from 1 to 2-3 m.sec<sup>-1</sup> will increase the drying rate

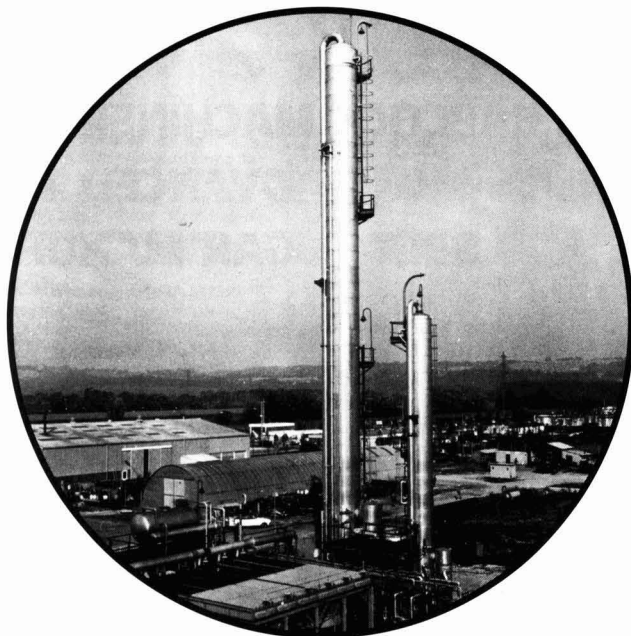
considerably, will allow reduction in the size and layout of the dryer and hence in the investment costs, but will cause a substantial rise in operating costs because of the disproportionate rise in pressure losses.

**Low-temperature pulp drying: fundamentals and economic considerations.** K. E. Austmeyer and W. Poersch. *Zuckerind.*, 1983, 108, 861-868, 1033-1041 (German). Among factors considered in relation to low-temperature predrying of beet pulp combined with conventional drum drying as the second stage are: the energy balance of the predryer (including the change in state of the air used), the quantity of air (heated by process waste heat) required to evaporate a given amount of moisture, the design of the air preheaters, and the effect of the state of the ambient air on dryer performance. Because of the small temperature gradient involved in use of low-temperature heat, the convective air streams required for a given heat transfer and mass transfer will be considerable, so that the dryer will also have to be very large; hence, it is important to find an economically optimum solution which would provide maximum evaporation at minimum costs. The design parameters of a band dryer are discussed, including the effect of non-uniformity in the thickness of the pulp bed on the velocity profile on the in- and out-flowing air, the required drying time and practical residence time. A pilot plant developed by Babcock-BSH AG permits examination of pulp drying under three different systems of air flow: over the pulp bed and through it from above and from below. Experiments carried out with the system in which air was passed through the pulp from above are reported. Results showed that the drying process could be divided into two parts: one of constant drying rate followed by one of decreasing drying rate. The drying rate fell in the direction of air flow, since the air became colder and moister as it passed through the top layer of pulp, so that the lower layer lost its moisture more slowly. The average drying rate rose linearly with temperature difference and with air flow velocity provided the pulp thickness remained above a certain value.

**Energy saving by heat recovery using a glass-tube heat exchanger and dryer vapour to dry sugar beet pulp.** H. J. Praus and W. Niemann. *Zuckerind.*, 1983, 108, 1041-1042 (German). — Details are given of a system for recovery of heat from pulp drying vapour installed at Dinklar sugar factory. Some of the vapour from the drum dryer is passed, at 106°C, through a glass-tube heat exchanger where it raises the temperature of incoming air from 15° to 87°C at an hourly air volume of 15,000 m<sup>3</sup>. The heated air is then recycled to the dryer to be mixed with boiler flue gas. Use of the glass-tube heat exchanger has reduced dust emission at the factory, while the vertical tubes in the heat exchanger are easily cleaned by occasional spraying (there is also a self-cleaning effect whereby condensate droplets inside the tubes bind and remove dirt particles from the vapour and carry them down to the collector below the tube section of the heat exchanger).

**Sugar beet vinasse as recycle for the beet field.** J. Cantstetter. *Zuckerind.*, 1983, 108, 1058 (German). Reference is made to 5-year experiments in West Germany in which concentrated vinasse of 60-70% dry solids has proved successful as a fertilizer when sprayed on fields for growing of various crops including sugar beet.

<sup>1</sup> Kunz: *I.S.J.*, 1984, 86, 191.



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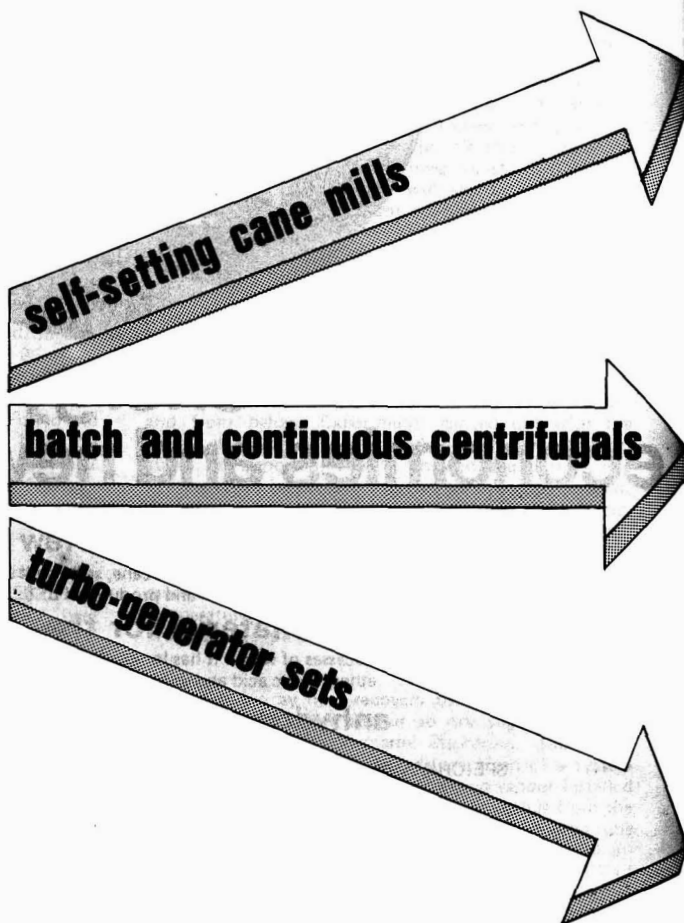
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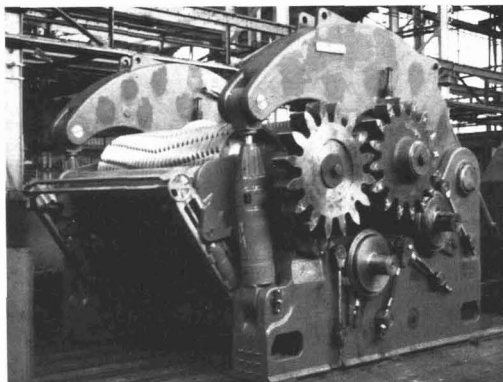
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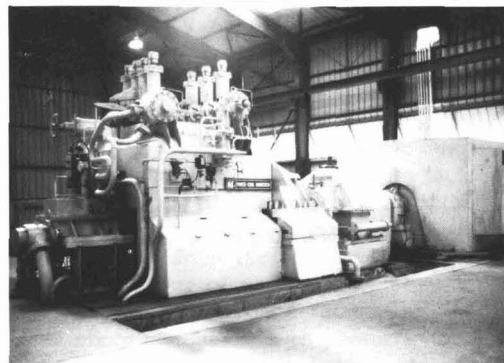
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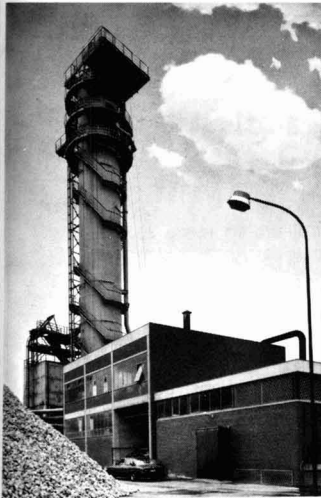
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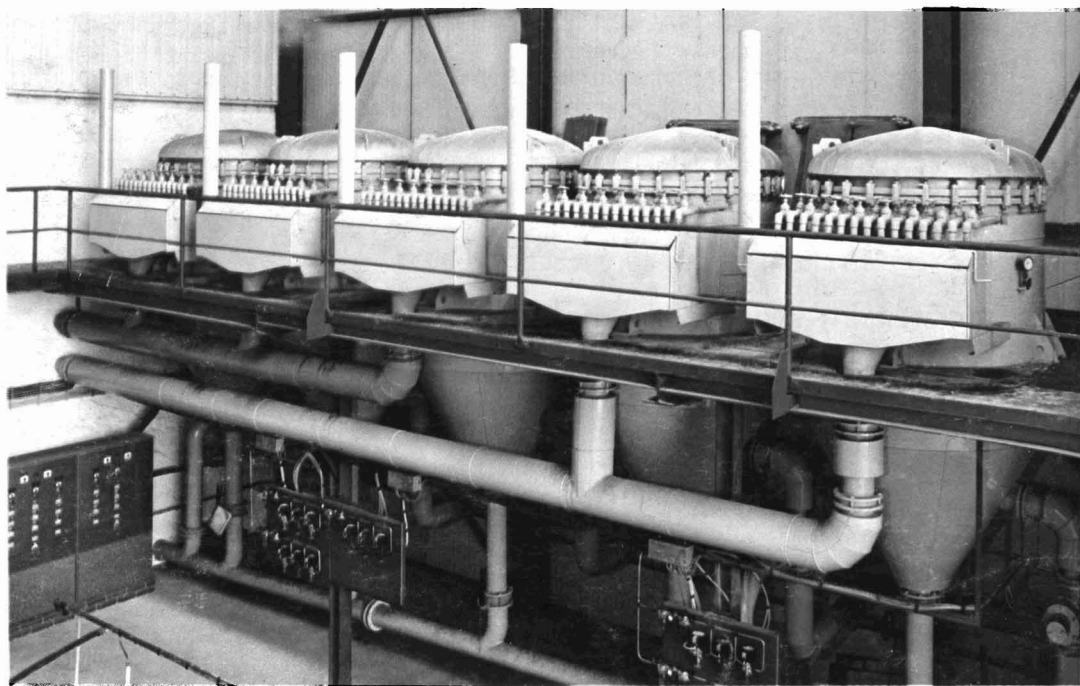
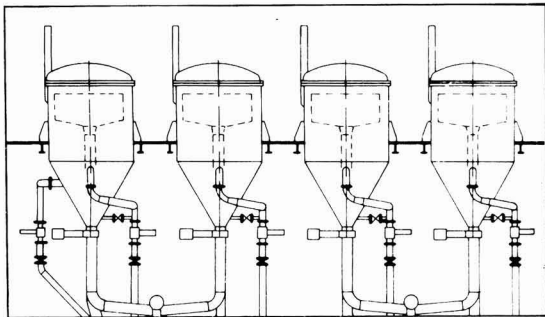


**—Putsch**

**Leaf Filters** – the Darlings in Filtration!

**—Putsch** Leaf Filters are used for Juice Polishing purposes.

**—Putsch** Leaf Filters are simple and efficient, have no problems in cake removal, need little maintenance work, are reliable and fully automated.



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After all – it's performance that counts!

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