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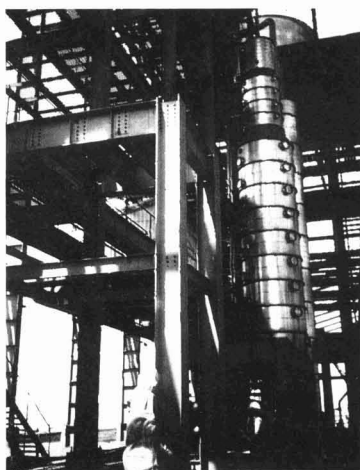
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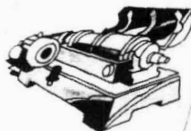
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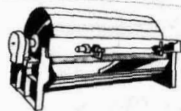
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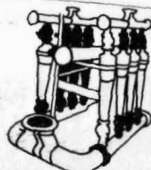
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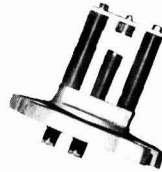
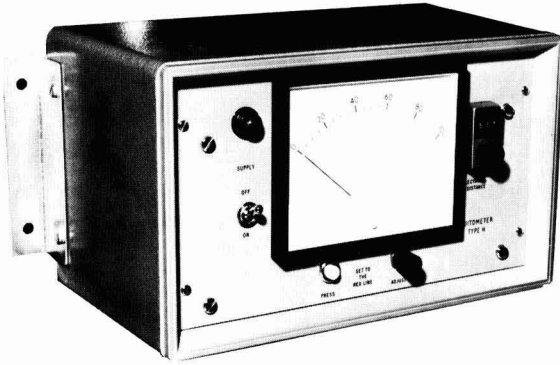


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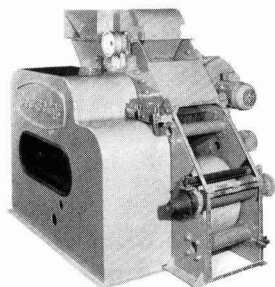
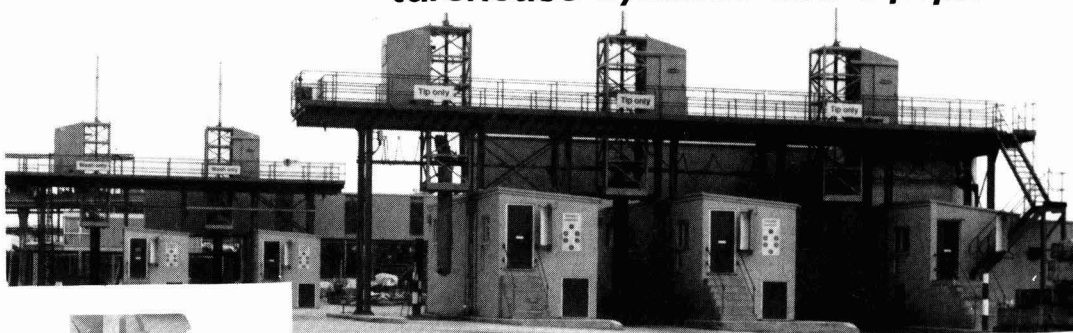
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# NOTES AND COMMENTS

## International Sugar Agreement

A routine meeting of the Executive Committee of the International Sugar Organization took place in early October. It and the Price Review Committee agreed to recommend no change to the price range of the current ISA from the present 13-23 cents/lb. Applications by Fiji, Guyana and South Africa for postponement until December 1984 of their special stocks accumulation obligation were approved.

Member countries are required to notify by May 15 and September 30 each year if they will be unable to fulfil their quotas and a further 300,000 tonnes were notified in addition to the approximately 750,000 tonnes notified in May. Cuba informed the Council that it would be unable to meet its full quota but could not specify by what amount; C. Carnikow Ltd. estimate the amount at of the order of one million tonnes<sup>1</sup>. This would halve the calculated excess of quotas in effect over requirements but no adjustment of these quotas is called for while the I.S.O. prevailing price remains below 12 cents/lb.

## Japanese sugar refining industry re-structuring

The Japanese government decided in mid-September to designate the sugar refining industry as recession-hit; under the temporary structural adjustment law enacted last May for five years, industries so designated can apply for tax relief and other government help whilst they restructure<sup>2</sup>. The refiners, hit by falling demand and large financial losses, will apply to the government for assistance.

The Japanese Ministry of Agriculture has called on the refiners to reduce capacity, currently estimated at 3.8 million tonnes, to 2.8 million tonnes under a five-year restructuring program set to start on October 1 last<sup>3</sup>. The program is based on a sweeteners demand of 3.2 million tonnes in the period October 1987/September 1988, including 600,000 tonnes of high fructose corn syrup and 550,000 tonnes of domestically produced beet sugar. The Ministry set the refiners' operation ratio at 75%, which led to a production capacity of 2.8 million tonnes.

Taito Co. Ltd., Japan's major refiner, has said it will close its refinery in Kawasaki, Kanagawa prefecture, in March 1984. The closure of the 500 tonnes/day plant will cost 61 jobs, but output of the other Taito refinery at Kobe will be raised from the current 50% to 75% of its 1000 tonnes/day capacity.

Ensuiko Seito and Toyo Seito are to merge to form a joint refining company, Taiheiyō Seito Co. Ltd., which will be owned equally by the two firms<sup>4</sup>. Toyo Seito will close its Tokyo refinery while Ensuiiko will lease its refinery in Yokohama to the new firm.

Mitsui Sugar Co. Ltd., Japan's largest sugar refiner, has announced that it will close one of its three refineries in order to streamline operations<sup>5</sup>. Mitsui plans to concentrate sugar refining at its Kawasaki refinery; the Shibaura plant will be closed and money will be invested in energy-saving measures at the Okayama plant.

## World sugar prices

On the first trading day of the month, October 3, the London Daily Price of raw sugar was £161.50 per tonne and rumours of purchases of EEC sugar by the Soviet Union, and purchases of 100-130,000 tonnes of white sugar by Saudi Arabia, carried it to £170 by October 10. Another factor had been reports that the usual transport difficulties in the USSR were hindering delivery of harvested beets to the factories with consequent loss of sugar production potential. Strong selling pressure from India emerged and by October 13 the price had fallen to £157. It then became public knowledge that Cuba was seeking to buy sugar to meet its export obligations in face of its own reduced crop and the price bounced up the next day to £162.50 after which optimism appeared to evaporate during the remainder of the month, particularly as there appeared very little of the significant Soviet buying interest which had been expected. Reports emerged of better results than previously expected from the European beet crop, and the LDP slid to £140 per tonne on October 31.

White sugar prices started the month at £181 per tonne and rose with raw sugar values at first, to reach £189 on October 7. After this it declined steadily through the month as a consequence of the large amount of sugar available and the premium over raws diminished correspondingly from around £20 per tonne at the beginning of the month to only £12.50 in the middle although it recovered somewhat by October 31 to £16, at which time the LDP(W) stood at £156 per tonne.

## USSR 1983 beet crop

It has been reported in the press that the Soviet beet crop was sown early and that it has benefited from good weather during the growing period. Harvesting operations were also able to start earlier than usual. By the beginning of October, however, progress had slowed and the benefit of the early start had been lost. It was reported that up to 14 million tonnes of beets were in the fields awaiting transport to the sugar factories and that these were suffering losses (temperatures have been remarkably warm). Beet production is likely to be up from the 71 million tonnes of 1982 but under the 96 million tonnes target. It is likely that sugar imports from other origins than Cuba will be smaller than in 1982 when they reached 3.14 million tonnes, raw value. On the other hand, Cuba seems likely to have less sugar available for the USSR so that, as is so often the case, Soviet requirements of sugar remain a matter of guesswork.

## Australian sugar industry inquiry<sup>6</sup>

The proposals of the Australian government's Industries Assistance Commission<sup>7</sup> for radical restructuring of the domestic sugar industry have been rejected by the Federal Minister for Primary Industry. The IAC's report contained five main proposals: (1) an end to the system of establishing an agreed maximum price for refined sugar on the domestic market, in favour of a market-oriented approach; (2) an end to the ban on sugar imports; (3) sugar production should not be controlled but allowed to operate freely; (4) export controls should be confined to those necessary to honour Australia's undertakings under the ISA; and (5) an end to restrictions

<sup>1</sup> *Sugar Review*, 1983, (1671), 194.

<sup>2</sup> *Public Ledger's Commodity Week*, September 24, 1983.

<sup>3</sup> F. O. Licht, *International Sugar Rpt.*, 1983, 115, 508.

<sup>4</sup> *ibid.*, 490.

<sup>5</sup> *ibid.*, 575.

<sup>6</sup> *World Sugar J.*, 1983, 6, (4), 30.

<sup>7</sup> *I.S.J.*, 1983, 85, 98.

on the entry of new farmers into cane growing and the use of new land. The IAC's proposals were criticized by the domestic industry as well as by the Minister as being unpractical and a threat to the future of the Queensland sugar industry if implemented.

#### Uruguay sugar situation<sup>1</sup>

The fact that only two of the country's four beet sugar factories were in operation in the 1982/83 campaign highlights the extent of the problem facing the local industry from illegal imports. The domestic retail price is not controlled by the government (though there is a stiff import tax on raw and refined sugar) and is thus significantly higher than that in neighbouring Brazil and also Argentina. The differential is made substantially greater, and hence very attractive to "external" suppliers, by the different foreign exchange policies (in simple terms Brazil and Argentina have devalued their currencies to such an extent that the Uruguayan peso is overvalued and hence can be exchanged for more US dollars *pro-rata*).

Such is the extent of illegal imports, facilitated by Uruguay's extensive and loosely controlled land borders with Argentina and Brazil, that consumption of domestically produced sugar has been in decline. Yet, as recently as the mid-1970's, there was active discussion on the need to contract the industry because, being a high cost producer (cane production costs were estimated to be 4.5 times those of Argentina in 1981), Uruguay was not well placed to export its burgeoning surpluses unless prices were exceptionally high. In fact, this physical contraction is under way only because of excessive imports, not exports.

At the present time, the level of domestic production, just over 80,000 tonnes, represents only some two-thirds of domestic capacity. It is true that La Sierra factory, in Pueblo Gregorio Aznarez, is being converted into a HFCS plant<sup>2</sup>, but it was the smallest (1000 tonnes per day throughput) of the country's beet sugar factories, representing less than 10% of national sugar processing capacity. Being more noted for maize than sugar, Uruguay has been a pioneer in Latin America in corn syrup production, and two plants already manufacture glucose.

There is thus 35,000 tonnes per year or more of idle capacity in the country at a time when an estimated 35,000 tonnes of sugar is being imported. Further, if recent trends are any guide, the import total will grow (only about 15,000 tonnes were imported in 1981/82) faster than national consumption, which is exhibiting little or no growth (in part because of the high prices that are attracting the imports). If this proves to be the case, a second operation could end up bankrupt; the Mercedes factory of Azucarera del Río Negro S.A. in Soriano Department closed its doors in 1981. This factory had a capacity of 20-25,000 tonnes per year, slightly larger than the next most likely candidate for closure, RAUSA's factory at Montes in Camelones Department.

Most of the current domestic output of sugar comes from the Paysandú beet sugar factory and the Bella Unión cane sugar factory. Each has a 3000 tonnes per day capacity and together produce some 75,000 tonnes of sugar per year. The cane sugar factory in Salto Department (Ingenio El Espinillar) is a small government-owned mill which is primarily concerned with production of alcohol which is a state monopoly; only small quantities of low-quality white sugar are produced.

#### Japan import surcharge on HFCS producers<sup>3</sup>

The domestic sugar producers in Japan are subsidized by a sliding scale surcharge on raw sugar imported for refining, based on the difference between their average import price and a target price set by the Ministry of Agriculture, Fisheries and Food. With declining consumption, imports have fallen, and the yield of the surcharge has also fallen, in spite of the increase in the rate. A surcharge has consequently been placed on HFCS production, the proceeds of which will be used to reduce the surcharge on imported sugar. If the market price becomes lower than the theoretical market price for refined sugar a second surcharge will be imposed on sugar and/or HFCS.

#### Middle East sugar demand increasing<sup>4</sup>

Despite efforts in the past to step up domestic production, demand for sugar in the Middle East and North Africa has continued to expand faster than output, so that imports for the area grew to 4.8 million tonnes in 1982/83 against an estimated 4.6 million tonnes the season before. Because most of the importing countries lack extensive refining capacity for raw sugar, the bulk of the imports are in refined form. Egypt, for example, the second largest Middle East sugar importer after Iran, buys all its 750,000 tonnes of annual requirements in refined form. Consumption in that country is growing, albeit at a relatively slow pace, owing to increased population, higher per caput income, and low fixed prices for sugar on the domestic market.

#### Nigerian sugar company difficulties<sup>5</sup>

The Savannah Sugar Company (SSC), owners of the Numan sugar project, owe N 28 million (\$38 million), according to its General Manager, but is only receiving a "totally inadequate" N 1.5 million subvention from the government each year. It is estimated that additional investment of N 150 million is needed to bring the project to its full production capacity of 100,000 tonnes a year.

SSC has suffered disasters ever since its inception in 1970; after ten years of preparation and planning it produced 23 tonnes of sugar in 1979/80, 10,000 tonnes in 1980/81 and 11,000 tonnes in 1981/82. Production should have reached 50,000 tonnes in 1979/80, according to the original plans, yet it is doubtful whether production will have been increased in 1982/83. The most crucial part of the project, in which the government invested N 300 million, is the Kiri dam on the Gongola river, which is still not complete. The project depends on this dam and its associated irrigation canals; as a result only 5000 acres of the planned 50,000 have been planted to cane.

The company's finances deteriorated even further after the government took over the management of SSC after this had been voluntarily surrendered by the Commonwealth Development Corporation in 1982 following great difficulty in obtaining the necessary federal finance.

\* \* \*

St. Vincent sugar production, 1983<sup>6</sup>. — Sugar production in St. Vincent in 1983 reached 2203 tonnes, 540 tonnes more than in 1982. Additionally, the island's single state-owned sugar factory produced 28,941 US gallons of molasses.

1 F. O. Licht, *International Sugar Rpt.*, 1983, 115, 420-421.

2 *I.S.J.*, 1983, 85, 127.

3 F. O. Licht, *International Sugar Rpt.*, 1983, 115, 490.

4 *Public Ledger's Commodity Week*, September 24, 1983.

5 *World Sugar J.*, 1983, 6, (2), 37-38.

6 F. O. Licht, *International Sugar Rpt.*, 1983, 115, 487.



# Large-capacity crushing mill

By P. MAYO and D. WRIGHT  
(A. Goninan & Co. Limited, Newcastle, N.S.W., Australia)

## Introduction

Following the success of the 2.5 m cane crushing mill in the Australian sugar industry, Farleigh Cooperative Sugar Milling Assoc. Ltd. chose to go one step further and increase the size of their new mill to an unprecedented 2.75 m width to match their need for increased throughput, combined with improved extraction efficiency. In line with current Australian mill technology, the crushing mill incorporates basically three crushing rolls, force-fed by two pressure feeder rolls via a diverging chute with an additional underfeed roll to promote feeding from a vertical feed chute. The five crusher and feeder rolls measure 2.75 m wide and 1.375 m diameter, each weighing 42 tonnes. The underfeed roll measures 2.75 m wide but is only 1.3 m in diameter and weighs 38 tonnes (Figure 1).

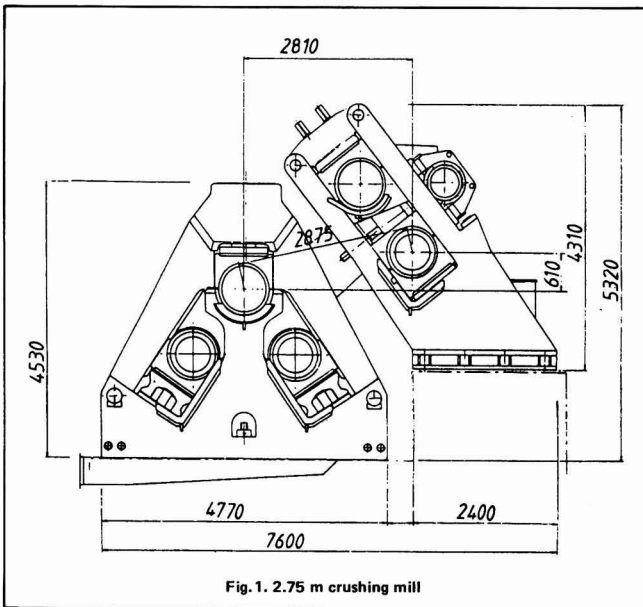
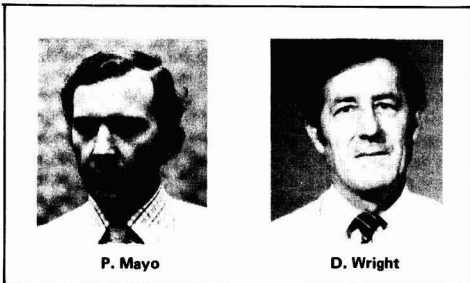


Fig. 1. 2.75 m crushing mill



The design of a mill and pressure feeder larger than ever attempted by any manufacturer necessitated departures from some of the generally accepted features of mill and mill drive construction. Conventional mill-mounted pinions failed to satisfy the normal stress analysis criteria and the most favourable solution was for these pinions to be relocated adjacent to the low speed gearing.

In another step, gearing more familiar to mining applications was incorporated into the low speed drive. The introduction of machine-cut, large-pitch, helical gearing was considered a significant improvement in the area previously dominated by spur gears.

## Turbines

For the nominated roll speed of 3.5 r.p.m. (15.5 m/min) the mill and pressure feeder were estimated to require a total of 1500 kW of power. Two options for providing this power were considered. Either one multi-stage turbine connected to a single high-speed gearbox and intermediate gearbox or, alternatively, two single-stage 820 kW turbines with dual high-speed and intermediate gearboxes driving the low-speed gearing. The latter arrangement was chosen for two main reasons:

- (a) A cost comparison revealed savings of approximately 15% were attainable in choosing the twin drive arrangement.
- (b) A comparison of the main gear wheel options indicated the single drive version would require a gear of 7.5 m in diameter and 1.0 m wide. On the other hand, the main gear wheel needed to be only 6 m in diameter and of 0.75 m face width when driven by two pinions, thus reducing the maximum crane lift required and easing installation problems.

The use of dual power plants avoids the usual difficulties associated with load sharing between two pinions when driven by a single power source. Pairs of turbines are commonly used on shredders and in other industries such as paper mills. Equal power output and speed from the turbines would be easily accomplished by the usual interlock governing system.

The particular turbines used have integral high-speed gearing incorporated into the turbine housing which dispenses with the external high-speed coupling and separate foundation. To ensure identical rotation of both turbine rotors, the high-speed gears on one turbine

\* Paper presented to the Australian Soc. Sugar Cane Tech., 1983.

### Large-capacity crushing mill

were made the mirror image of the other to impart the correct output shaft rotation. A schematic diagram showing the relationship of the two turbines and the overall gear configuration appears in Figure 2.

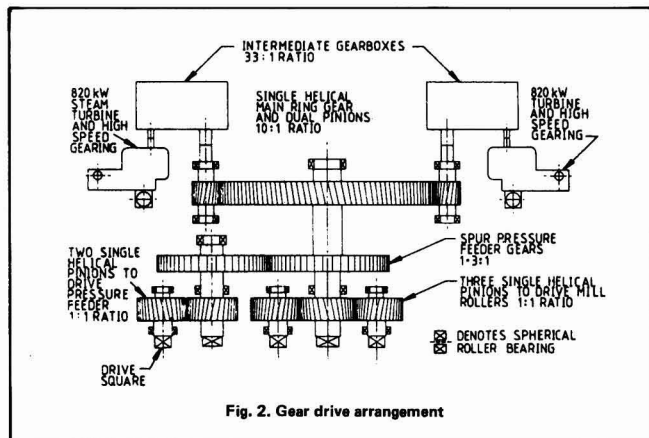


Fig. 2. Gear drive arrangement

### Intermediate gearboxes

Aside from having two complete gearboxes, the intermediate gearing would be considered similar to conventional units. High precision, hardened and ground single helical gearing with parallel shafting was chosen for its compactness and reliability.

### Low speed gearing

With conventional Australian (5-roll mill) sugar mill drives, the necessary overall gear ratio of approximately 1200:1 has been composed of individual ratios which have tended to remain close to the following values:

- 5:1 single reduction high-speed
- 12:1 double reduction intermediate
- 20:1 double reduction low-speed gearing.

In an endeavour to provide more effective use of the necessary reductions, the intermediate gear ratio was increased to a level of 33:1 to permit a single low-speed gear reduction. Many years of experience with large gear ratios in the range 10:1 up to 14:1 with metalliferous ore grinding mills and similar applications indicated that a single reduction of approximately 10:1 would not pose any undue problems for the sugar mill final reduction, provided the usual precautions necessary with such large ratios were observed.

Another feature of the grinding mill type gear offering benefits to the sugar industry concerns large pitch single helical gearing. Aside from the increased smoothness in operation, helical gears, for a given size and material, transmit approximately 60% more torque than the equivalent spur gearing.

In keeping with the grinding mill style of gear, a shallow ring type main gear wheel was chosen for the following reasons:

- (a) The weight of such a gear at 22 tonnes was much less than its conventional shaft mounted counterpart of comparable dimensions.
- (b) Being six metres in diameter, the gear is split into segments further improving handling, transportation, assembly and disassembly requirements.
- (c) Ring gears of this type are made symmetrical about the vertical cross section to permit future reversal of the gear wheel to run on the unworn tooth flank. Figure 3 shows the symmetrical shape referred to in both the pressure feeder and main gear wheels.
- (d) The design permits the use of relatively inexpensive mild steel fabricated centre support while still employing alloy steel in the gear rim where higher hardness and strength of the gear teeth is necessary to

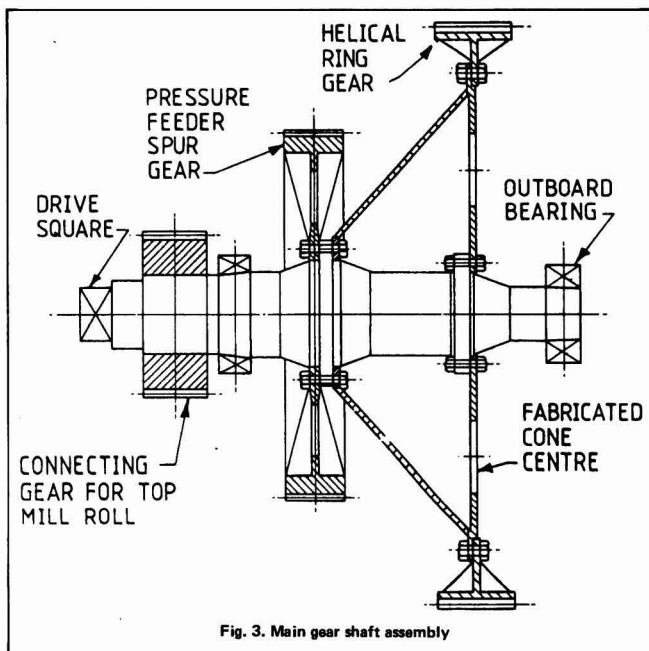


Fig. 3. Main gear shaft assembly

achieve the desired gear rating. The cone shaped gear centre was so designed to accept the flange mounted ring gear and resist axial forces created by the helical gearing.

Instead of using keys which are expensive and difficult to fit in such large applications, a method used in attaching mine winder drums to shafts was adopted for the main gear shaft. The main shaft incorporates two separate, upset flanges to which the cone centre and pressure feeder gear attach by friction grip high strength studs. This type of connexion also attaches the ring gear to the outer flanged perimeter of the cone centre. The

direction of the helix of the two gears on the main shaft provides a constant axial thrust in opposition to the tailbar thrust. The pressure feeder driving gear was again designed as a ring gear but with spur teeth only.

To guard against indefinable external loads tending to displace the inner bearing race along the shaft, thrust rings and shrink rings have been located at strategic positions. The external perimeter of both the shrink rings and thrust collars double as the rubbing surface for the lip seals used in the bearing housings.

Fabricated one piece steel housings were used for the bearings of the dual input pinions and non-drive bearings on the main shaft and pressure feeder shaft. These housings were considered more robust than the commercially available cast plummer block type housings.

### Mill pinions

The absence of mill-mounted pinions would be the most conspicuous change from past mill designs. Evaluation of the requirements for mill pinions indicated pinions on both ends of each shaft would have been necessary to limit both contact and bending stresses without having excessive face width. However, the use of pinions on both ends of a shaft would have been prone to synchronizing problems from torsional deflections and the usual inaccuracies of cast teeth. The proposed relocation of the pinions away from the mill and pressure feeder by the introduction of fully enclosed, fixed centre helical gearing was enthusiastically received by mill personnel. The most significant benefit would be that the pinions are fixed in locations and therefore retain geometry despite roll lift or adjustment — centre distance and axial alignment (parallelism) remain fixed. The advantages of such a change would be obvious to most people concerned with the servicing and maintenance of mill pinions. Other benefits include:

- (a) Tooth geometry — Improved design by elimination of the coarse pitch, deep flank, non-involute gear teeth often prone to undercutting.
- (b) Lubrication — Remains free of contaminants.
- (c) Fixed centres — No longer necessary to interchange pinions.
- (d) Helical gearing — Quieter and smoother operation.
- (e) Design — The gear teeth can be engineered in accordance with AGMA standards.
- (f) Teeth profile — Gear teeth accurately machine cut.
- (g) Mill roll — Shorter mill roll shaft reduces weight and cost.
- (h) Appearance — Being fully enclosed, no leakage of lubricant occurs.

A useful device, termed an 'eccentric sleeve' (Figure 4), sometimes installed in large gearboxes such as those found in draglines used for open cut mining, was considered necessary for achieving precise alignment of the mill pinion teeth. Alignment of gear teeth in general has always been crucial for achieving the design life of the gears. After assembly of the gearing, alignment of the mill pinion teeth utilizes these eccentric sleeves incorporated into the bearings of the three mill pinions mounted on stub shafts which drive the feed, delivery and top pressure feeder rolls. The eccentricity is only 0.4 mm, but by selective rotation is sufficient to correct minor inaccuracies in assembly of the low speed gears.

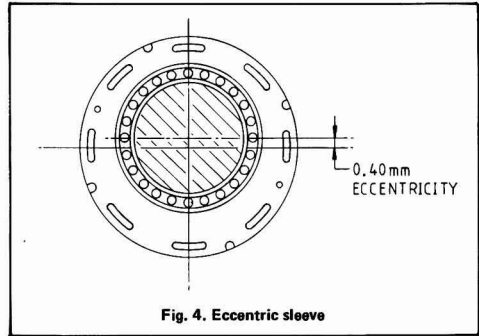


Fig. 4. Eccentric sleeve

### Foundation

The usual box-sectioned, raft-type gearbed simplifies installation, but in service becomes redundant when the concrete can be designed to accommodate all the imposed loads. The complexity of the current gearing arrangement virtually excluded the common raft-type gearbed. The configuration of dual pinions driving a large ratio gear, combined with pressure feeder and connecting gears, lent itself to developing an essentially concrete foundation similar to those being used in many diverse industrial applications including mineral processing, mining equipment and others.

Two spherical roller bearings support the main shaft and the outboard end bearing uses a plummer block; the drive end sits in the special housing designed to enclose the pinions for driving the three mill rollers. Similarly, the pressure feeder gearing drives a pair of fixed centre pinions which replaces the conventional pair of long tooth pinions on the pressure feeder rolls.

The turbines and gearboxes are placed on raised foundations to take advantage of the opposing radial gear forces on the main wheel from the input pinions. However, economic considerations limit the height of these particular foundations to a level below that which produces diametrically opposite radial forces (Figure 5).

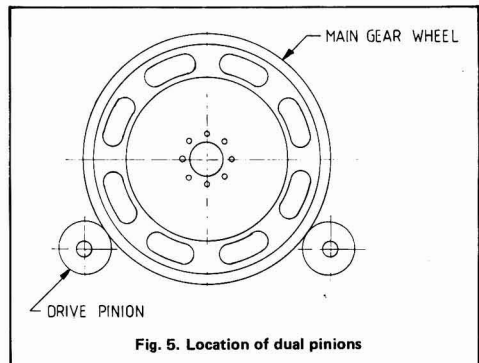


Fig. 5. Location of dual pinions

### Tailbars

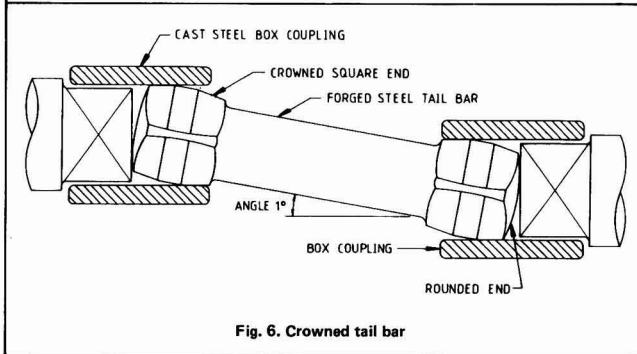
Much consideration was given to tailbar alignment with the fixed centre mill pinion arrangement. All of the alternative arrangements and special couplings which were considered proved extremely costly. The decision was made to maintain the conventional square couplings and minimize the mis-alignment forces produced in this

### Large-capacity crushing mill

type of coupling in three principal ways:

1. Lengthening the tailbars.
2. Preferential setting of tailbars.
3. Crowning the squared ends.

By lengthening the tailbars and aligning them to the mid-wear position of the rolls, the angular misalignment of the tailbars was limited to 1 degree maximum (see Figure 6). The worst case exists only with the rolls in the new and the fully worn conditions.



Forces generated by the couplings operating at the maximum misalignment have been incorporated into the design and selection of the adjacent bearings.

### Lubrication

Lubrication of the main wheel and pressure feeder gear takes advantage of the system developed to apply heavy bitumastic type lubricants to coarse-pitch open gears by spray applications. Several nozzles attached to the gear cover direct lubricant to the loaded face of the gear and apply, intermittently, a small, controlled amount. To permit spraying of the very viscous lubricant, a diluent reduces the fluid viscosity to levels suitable for pumping.

The advantages of such a system are several:

1. heavy viscosity lubricant can be employed which promote effective lubrication film on contacting surfaces,
2. small quantities of lubricants are involved,
3. only the gear teeth receive lubricant and in turn transfer a sufficient amount to the pinions,
4. sealing against leakage and contaminants becomes simple and effective,
5. the system is fully automatic, and (6) there is only a small amount of residue.

As a precaution, alarm switches on each spray nozzle piston warn of blockages of failure of the lubrication system. A manual back-up pump permits servicing of the automatic system during the crushing season.

The design of the gear covers enclosing the main gear and pressure feeder gears are typical of those used on metalliferous grinding mill gears. Such covers have proved to be effective, easily removable and economical to manufacture.

### Crushing mill

In contrast to the innovations in the gear drive, the mill was designed more conservatively considering the increased size over previous mills.

After careful consideration of various mill frame configurations, the Squier one-piece saddle concept (or

'A' frame) was employed to embrace the bearing on each end of the three mill rolls. These frames, each weighing 17 tonnes, may be lifted off separately and have the additional feature of a top cross beam linking the two frames. This allows removal of the complete assembly including scrapers and permits free standing of the two frames on a nearby floor.

Traditional bronze bearings were chosen for their reliability and lower cost compared with roller bearings. Roller bearing technology was not considered sufficiently advanced to be seriously considered in the new mill.

### Pressure feeder chute

The pressure feeder chute retains the same length of 1.950 m used on the 2.5 m mills. The knowledge that frictional drag increases with chute length prompted this limitation to that which had already proven satisfactory. In conjunction with establishing the minimum length of chute, sufficient clearance between mill and pressure feeder for removing the feed roller was also accommodated.

Considerable thought went into devising special adjustable connexions between the top and bottom chute surfaces and the side plates.

This connexion resists the tendency of the chute to bulge in the centre portion thereby decreasing the risk of choking from this undesirable change in internal shape.

A special mounting pivot pin and sliding block provides a flexible connexion between the mill and pressure feeder cheeks and full roller life adjustment for angular and vertical movements to the chute body. This large pin also provides for the thrust forces on the chute.

### Acknowledgement

On behalf of the company, the authors wish to express their appreciation to the Board of Directors and the management of Farleigh Cooperative Sugar Milling Association Ltd. in placing their faith in the ability of A. Goninan & Co. Ltd. to design and manufacture the new mill.

### Summary

The design of an entirely new mill of unprecedented size provided the opportunity to produce a mill and gear drive with features new to the sugar industry. Some of these concepts originated by necessity from the increased size and loading over smaller mills. Other ideas were introduced from technology developed in other industries such as mining and paper manufacture, etc. Considerable thought, planning and evaluation resulted in the emergence of a completely new mill and drive which is expected to improve cane throughput and efficiency as well as reducing downtime because of ease of maintenance.

### Moulin à canne de grande capacité

Le développement d'un moulin tout à fait nouveau de dimensions sans précédent a permis la production d'un moulin et d'une boîte de vitesses avec des caractéristiques nouvelles pour l'industrie sucrière. Certains des concepts trouvaient leur origine dans le besoin créé par la capacité accrue et par la surcharge des petits moulins. D'autres idées furent introduites de la technologie développée dans d'autres industries, telles que les mines et la papeterie. Beaucoup de réflexion de plans et d'éval-



uation ont résulté en la production de ce nouveau moulin dont on attend qu'il améliorera le travail de la canne et son efficacité et qu'il diminuera les temps d'arrêt, suite à la facilité d'entretien.

### Rohrmühle großer Kapazität

Der Entwurf einer völlig neuen Mühle von bisher nicht bekannter Größe ergab die Möglichkeit, eine Mühle und einen Mühlenantrieb mit für die Zuckerindustrie neuen Eigenschaften zu bauen. Einige dieser neuen Konzepte entsprangen der Notwendigkeit nach größeren Ausmaßen und Kapazitäten als die kleinen Mühlen. Andere Ideen wurden von Technologien aus anderen Industrien wie Bergbau und Papierherstellung usw. übernommen. Beträchtliche Denkarbeit, Planung und Auswertung resultierte in der Entwicklung einer völlig neuen Mühle mit Mühlenantrieb, von der erwartet wird, daß sie die Rohrdurchsatzleistung und Effektivität

sowie Stillstandszeiten infolge leichterer Instandhaltung verbessern werden.

### Molino de gran capacidad

El diseño de un molino enteramente nuevo, de un tamaño sin precedente, ofreció la oportunidad para producir un molino y engranaje con aspectos nuevos para la industria azucarera. Algunos de estos aspectos originan, por necesidad, del tamaño más grande y carga mayor que con molinos de menor tamaño. Otras ideas se han introducido de tecnología desarrollada en otras industrias como minería y fabricación de papel. Pensamiento, planificación y valoración notable han resultado en la aparición de un molino y engranaje totalmente y, por la simplicidad de entretenimiento, con un período reducido fuera de servicio.

## Conglomerate formation in sugar crystallization Part II. Fundamental approach

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### Introduction

In the first part of this study<sup>1</sup>, the effect of process conditions on the formation of conglomerates in a 1.4 m<sup>3</sup> pilot-plant crystallizer was described and the main conclusion was: conglomerate formation predominantly takes place in regions with a high supersaturation level and is strongly related to nucleation.

This latter can be visualized by means of Fig. 1, in which the conglomerate content (CC)\* is presented as a function of the nucleation rate for runs performed in that 1.4 m<sup>3</sup> crystallizer. At high nucleation rates a high CC is found, whereas at decreasing nucleation rate the CC also decreases.

In this present study an approach is made to describe the formation of conglomerates in a fundamental way.

As already mentioned in the first part of this study, at least four possible mechanisms for the formation of conglomerates can be distinguished:

- (i) building in of a molecule into two single crystals<sup>2</sup>
- (ii) sealing of single crystals owing to collisions<sup>3, 4</sup>
- (iii) spontaneous formation, and

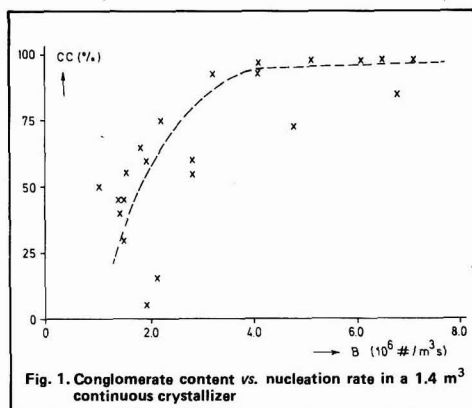


Fig. 1. Conglomerate content vs. nucleation rate in a 1.4 m<sup>3</sup> continuous crystallizer

(iv) surface nucleation.

Of these mechanisms only the last two could be studied successfully, while yielding only qualitative results.

### Experimental

The experiments were performed in a 100 ml thermostated vessel, with glass windows to make visual observation possible. A schematic diagram of this vessel is

\* CC =  $\frac{\text{number of conglomerates}}{\text{total number of crystals}} \times 100\%$ .

1 Kuijvenhoven et al.: *I.S.J.*, 1983, 85, 201-207.

2 Powers: *Sucker Handl.*, 1964, 19, (5), 51.

3 Moller: *I.S.J.*, 1947, 49, 182.

4 Idem: *Sugar*, 1954, 49, (11), 49.



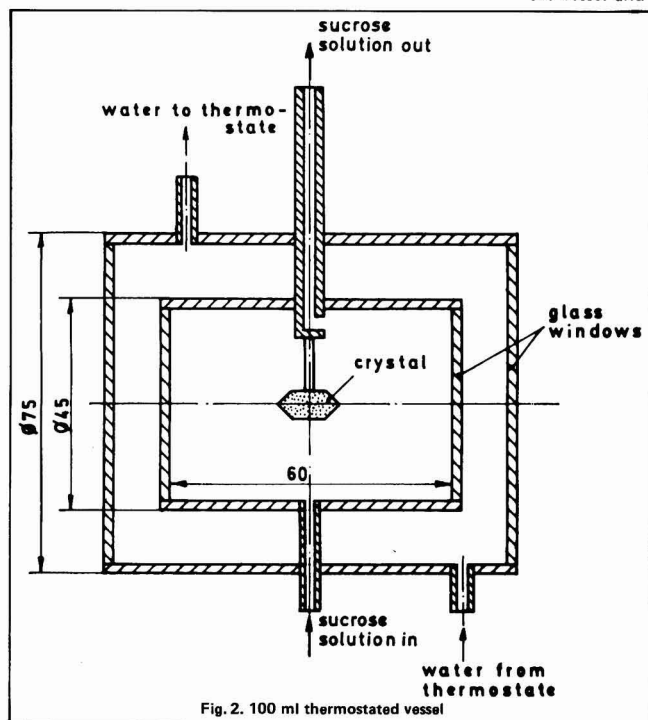


Fig. 2. 100 ml thermostated vessel

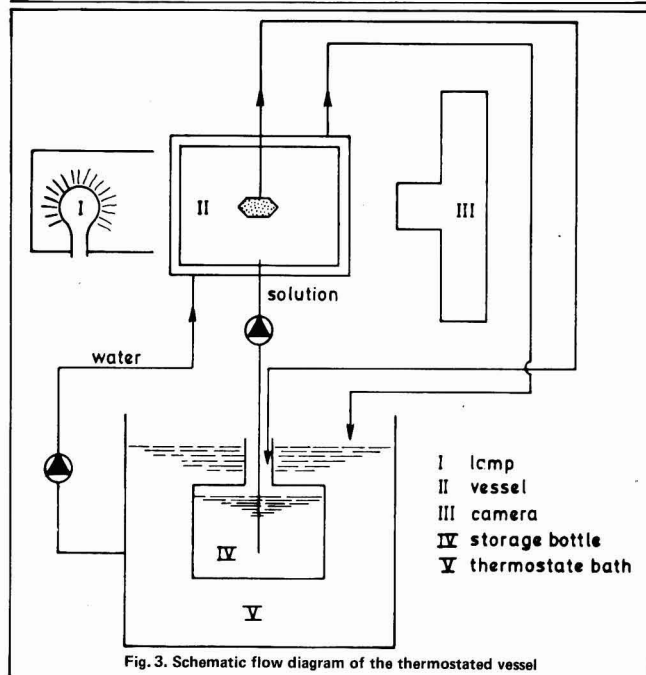


Fig. 3. Schematic flow diagram of the thermostated vessel

shown in Fig. 2. The vessel could be filled with a (super-) saturated sugar solution, which could circulate through the vessel and a storage bottle as shown in Fig. 3. In to the top of the vessel a parent crystal, glued to a rod, could be placed, in either of two positions at 90° to each other.

The experimental conditions were a working temperature between 60° and 80° C and a supersaturation ratio (S) ranging from 1.0 to 2.0. The experiments to study spontaneous conglomeration were performed by filling the 100 ml vessel with a sugar solution of the desired supersaturation and waiting until nucleation/conglomeration took place when no parent crystal was present; alternatively, the sucrose solution was circulated through the vessel and the storage bottle. The experiments to study the mechanism of conglomerate formation due to surface nucleation were performed similarly to this latter procedure. Before starting the actual experiment, however, the solution was kept for several hours at a low supersaturation level ( $S < 1.05$ ), by raising the temperature of the water bath, at which supersaturation only growth will take place, in order to exclude non-regular growth of the crystal due to the irregularity of the crystal surface. After this period the temperature of the water bath was adjusted to obtain the desired supersaturation and the circulation was stopped when the solution in the 100 ml vessel had reached this temperature/supersaturation.

Results

Fig. 4 shows the CC as a function of the supersaturation, as obtained with the spontaneous conglomeration experiments. The observation of the crystals was performed by means of photographs which were enlarged to a suitable size. The total number of conglomerates that could be examined varied from 20 to 500, while the time between the start of the experiment and the examination varied from one to two hours, which time is necessary to obtain a visible crystal size.

From this figure it is apparent that the CC increases with supersaturation and also that the presence of a parent crystal influences the conglomeration considerably. If a parent crystal is present, the formation of conglomerates starts at a much lower supersaturation,

<sup>5</sup> Maurandi: *Zuckerind.*, 1981, 106, 993.



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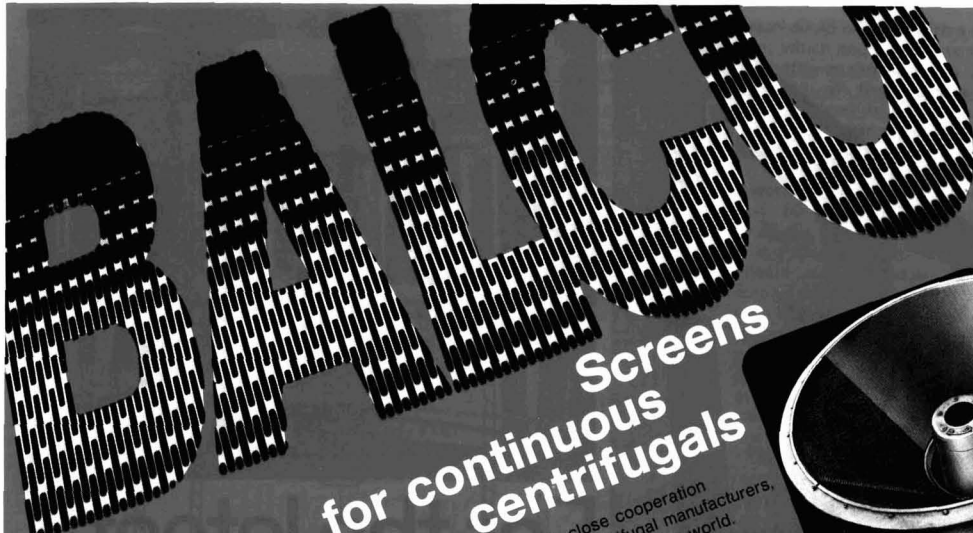
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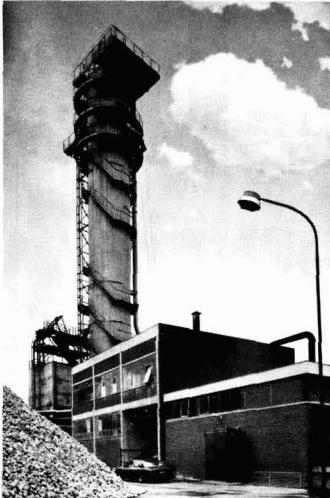
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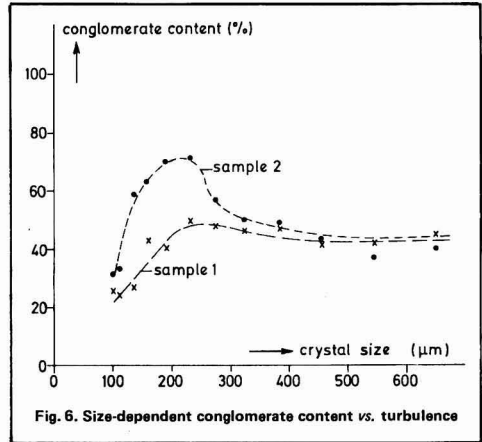
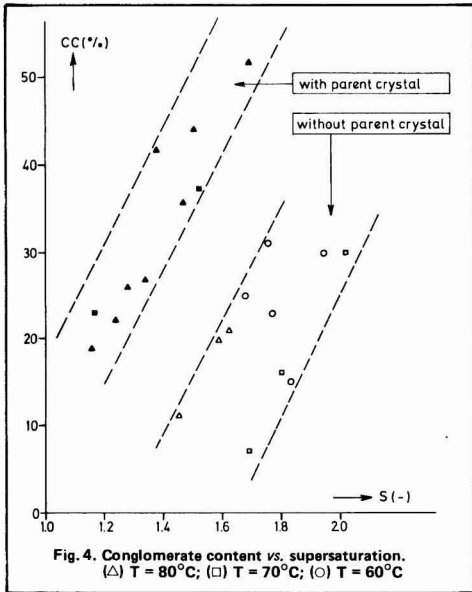
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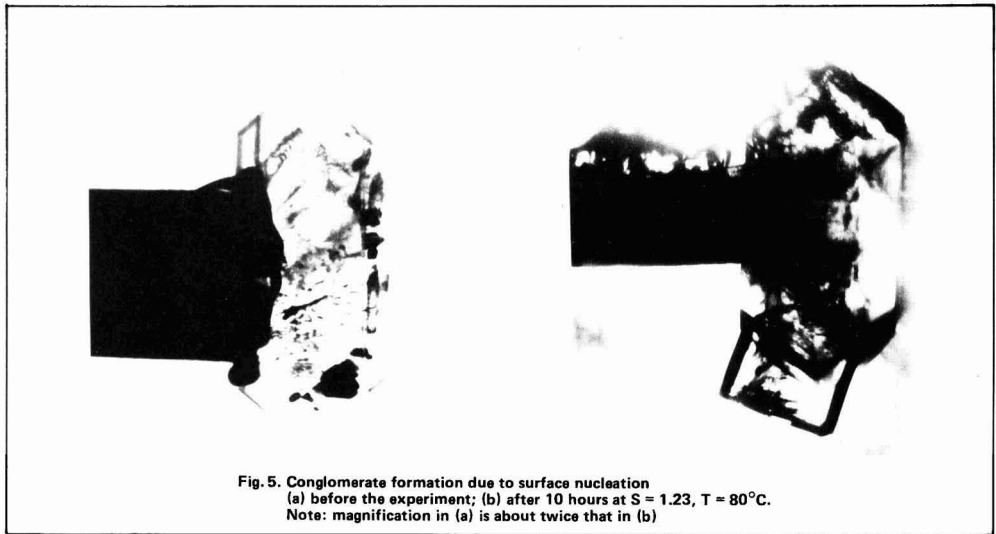
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spontaneous formation of conglomerates and surface nucleation, do exist. This statement is confirmed by the results of experiments performed in a 45-litre cooling crystallizer, which is described extensively elsewhere<sup>6</sup>, as presented in Fig. 6. This figure shows the CC as a function



which is in accordance with the nucleation phenomena as already mentioned in the first part<sup>1, 5</sup>, spontaneous nucleation occurring at  $S > 1.3$  while secondary nucleation already can be found at  $S < 1.2$ . No effect was found of the temperature at which the experiments were performed.

In Fig. 5 a parent crystal before and after a test run is shown in the case of a "surface nucleation" experiment (note that the magnification in the picture 5a is about twice the magnification in 5b). From these pictures it is clear that the parent crystal has become a conglomerate, which must have been caused by surface nucleation and not by crystal-crystal collisions, as no other crystals were present in the solution.

These experiments show that both mechanisms, viz.

of crystal size and the impeller rotational speed in a bulk-crystallization experiment. The first sample was withdrawn from the vessel after several hours of steady operation, showing an increasing value of the CC in the region 100-200  $\mu\text{m}$  and a stationary value of 40-50% for crystals larger than about 200  $\mu\text{m}$ . After this sample was taken, the impeller speed was decreased from 8.8 to 3.5 rps, and after one hour a second sample was taken. The CC as a function of the crystal size now shows a steep increase in the region 150-250  $\mu\text{m}$  to a value of about 70% and decreasing at larger size to the original value of 40-50%. From this experiment one can conclude that decreasing the impeller speed increases the conglomerate content.

<sup>6</sup> Kuijvenhoven & de Pree: *Zuckerind.*, 1983, 108, 35.

### Conglomerate formation in sugar crystallization

This effect was also found in batch sugar crystallization<sup>7-9</sup> and in the crystallization of potassium alum<sup>10</sup> and potassium dichromate<sup>11</sup>. It is caused by the fact that, owing to a higher turbulence around the crystals, the boundary layer, in which surface-nucleation takes place, is disturbed and the nuclei are removed from the crystal surface. If these nuclei are larger than the critical size, they will grow to macroscopic sizes; otherwise they will dissolve again. Another aspect that can be deduced from Fig. 6 is the fact that conglomerates are predominantly formed in the smaller size range, as the CC for sizes above 250  $\mu\text{m}$  is still unchanged.

### Discussion

From the experiments described in the foregoing part it was concluded that both surface nucleation and spontaneous formation of conglomerates do exist, but no evidence was found that the mechanisms as proposed by Moller and Powers should be excluded.

either recorded on a digital memory oscilloscope or on a pulse height analyser.

With this impact meter it was possible to measure the collision force between a crystal and the blade with the element. In Fig. 8 some results are shown that were obtained with different sieve fractions in liquids of a viscosity of the order of that of a saturated sugar solution. It is apparent from this figure that the force due to a collision between a crystal and the impeller is very small, especially at the lower size range. As the relative velocity between crystal and impeller is much more than that between two crystals it is likely that a collision force between two crystals is also very small and consequently that the kinetic energy of the crystals might not be high enough for them to approach close enough to form a conglomerate. As the CC is not a function of crystal size in the case of a stationary working crystallizer (see Fig. 6, sample 1) and the fact that crystal-crystal collisions are unlikely to occur, it may be concluded that conglomerates will only be formed at small crystal sizes.

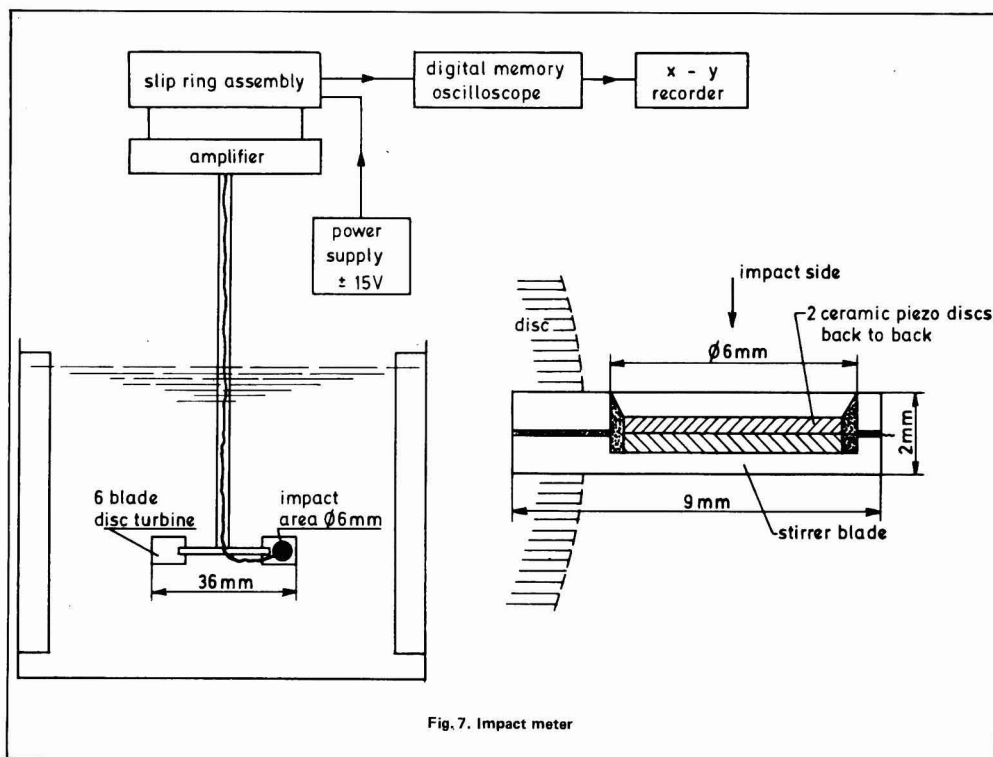


Fig. 7. Impact meter

These mechanisms are based on the possibility of a collision between two crystals, but results obtained in a separate equipment<sup>12</sup> suggest that the probability of such a collision is only small, owing to the small density difference between crystals and mother liquor and to the high viscosity of the system. This equipment was developed by Bemer *et al.*<sup>13</sup> and consisted of a 1.1 litre flat-bottom vessel, agitated by a 6-blade disc turbine impeller. A measuring device (a piezo electric element) was built into one of the blades, as is shown in Fig. 7. After amplification the electric pulses obtained, caused by a collision between the element and a crystal, could be

This is in accordance with the observation that most of the conglomerates found were composed of equally sized parts. This means that the formation of conglomerates by surface nucleation only will also take place at small crystal sizes. As it is not likely that no nuclei are

<sup>7</sup> Madsen: *Zuckerind.*, 1980, 105, 234.

<sup>8</sup> van der Poel: *ibid.*, 237.

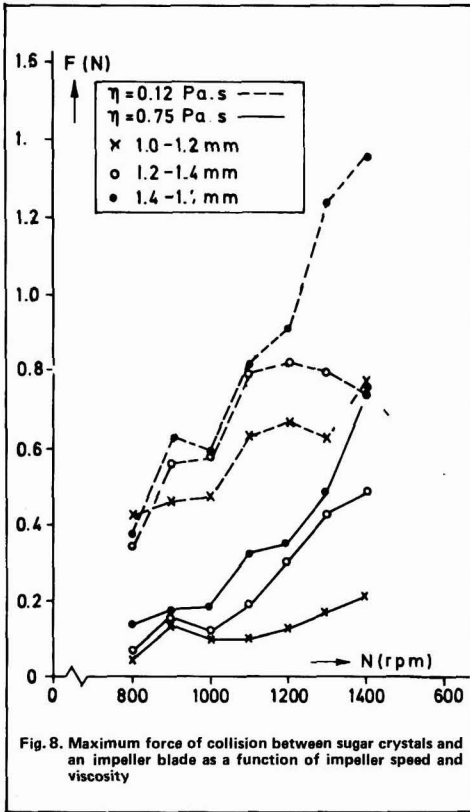
<sup>9</sup> Austmeyer: *ibid.*, 227.

<sup>10</sup> de Leer: *Ph.D. Thesis* (Delft University of Technology), 1981.

<sup>11</sup> Janse: *Ph.D. Thesis* (Delft University of Technology), 1977.

<sup>12</sup> Kuijvenhoven: *Ph.D. Thesis* (Delft University of Technology), 1983.

<sup>13</sup> *Chem. Eng. Sci.*, 1978, 34, 747.



formed on large crystals, those nuclei will disappear somehow. They may be overgrown by the parent crystal, or removed from the crystal surface by fluid shear.

As the crystals are, most of the time, in a laminar flow and only briefly in the direct sphere of influence of the impeller, the fluid shear caused by the relative velocity between a large crystal and mother liquor (the free-fall velocity) should be enough to remove the nuclei from the crystal surface.

The main conclusions of this study are, consequently, that: conglomerates are formed both spontaneously and by a surface nucleation collisions between crystals resulting in a conglomerate are very unlikely.

*Acknowledgement*

The authors wish to thank "Suiker Unie" for the finance and help that made this study possible.

*Summary*

In this study results are presented concerning the fundamentals of conglomerate formation as observed in a 100 ml vessel. It appeared that conglomerates are predominantly formed at small crystal sizes, either spontaneously or by surface nucleation. No evidence was found to exclude the view that conglomerated crystals may also be formed by collisions between existing crystals but, as was concluded from separate experiments, as collision between crystals is very un-

likely owing to the high viscosity of the sugar/water system and the low density difference between crystals and mother liquor.

**Formation de conglomerats au cours de la cristallisation du sucre. Partie II. Approche fondamentale**

On présente ici les résultats se rapportant à la formation de conglomerats tels qu'observés dans un flacon de 100 ml. On a noté que les conglomerats se forment surtout à des petites tailles des cristaux, soit spontanément, soit par nucléation à la surface. On n'a pas trouvé d'évidence qui permettrait d'exclure les vues selon lesquelles les cristaux conglomerés pourraient aussi être formés par collision entre les cristaux existant. Cependant, comme on l'avait déjà conclu à la base d'essais antérieurs, une collision entre cristaux est peu probable suite à la haute viscosité du système sucre/eau et à la faible différence de densité entre les cristaux et l'égout-mère.

**Konglomeratbildung bei der Zuckerkristallisation. 2. Teil. Grundlegende Arbeiten**

In dieser Arbeit werden Untersuchungsergebnisse über die Gründe für die Konglomeratbildung, beobachtet in einem 100-ml-Kolben, dargestellt. Es zeigte sich, daß sich Konglomerate hauptsächlich bei kleiner Kristallgröße bilden, entweder spontan oder durch Oberflächenkeimbildung. Es konnte kein Nachweis für den Ausschluß der Theorie erbracht werden, daß sich Konglomeratkristalle auch bei Kollisionen zwischen vorhandenen Kristallen bilden. Jedoch zeigten andere Versuche, daß eine Kollision wegen der hohen Viskosität des Zucker/Wassersystems und des geringen Dichteunterschiedes zwischen Kristallen und Muttersirup unwahrscheinlich ist.

**Formación de conglomerados en cristalización de azúcar. Parte II. Examen fundamental**

En este examen los resultados se presentan respecto de los fundamentos de la formación de conglomerados como observada en un vaso de 100 ml de capacidad. Parece que los conglomerados se forman en mayor parte con tamaños pequeños de cristal, tampoco en manera espontánea o por nucleación superficial. Los autores no encuentran evidencia para exclusión de la opinión que cristales conglomerados pueden formarse por colisión entre cristales existentes pero, como han concluido de otros experimentos, consideran que una colisión entre cristales es muy improbable debido a la viscosidad alta de la sistema azúcar/agua y a la pequeña diferencia entre las densidades de los cristales y del madre-licor.

**Turkey sugar imports and exports, 1982<sup>1</sup>**

|                | 1982              | 1981   | 1980    |
|----------------|-------------------|--------|---------|
|                | tonnes, raw value |        |         |
| <i>Imports</i> |                   |        |         |
| Brazil         | 0                 | 12,989 | 24,237  |
| EEC            | 0                 | 17,718 | 177,493 |
| Rumania        | 0                 | 0      | 10,870  |
| USA            | 0                 | 43,126 | 11,984  |
|                | 0                 | 73,833 | 224,584 |
| <i>Exports</i> |                   |        |         |
| Cyprus         | 4,022             | 3,912  | 3,532   |
| Iran           | 179,590           | 0      | 0       |
|                | 183,612           | 3,912  | 3,532   |

<sup>1</sup> I.S.O. Stat. Bull., 1983, 42, (6), 40-41.

# Densities of sucrose solutions containing potassium chloride

By THELMA M. HERRINGTON  
and ROBERT J. JACKSON  
(Department of Chemistry, University of Reading,  
Reading RG6 2AD, England)

## Introduction

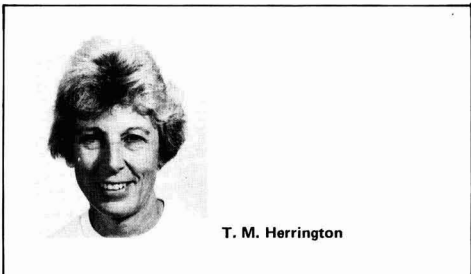
Sucrose solutions handled industrially contain both other sugars and inorganic salts as impurities, often at quite high levels. Exact physical data on the density changes in sucrose solutions with added electrolyte provide the necessary information on the volume changes to be expected in factory purification. We have extended our earlier work<sup>1</sup> on pure sucrose solutions and that of Mantovani & Indelli<sup>2</sup> on impure sucrose solutions at 25°C to higher temperatures. The ternary system chosen for study was aqueous sucrose plus potassium chloride at 30, 50 and 70°C.

## Experimental

The deviation in the apparent molar volume,  $\delta\phi V$ , caused by an error of  $\pm 1 \times 10^{-5}$  g.cm<sup>-3</sup> in the density and an error of  $\pm 5 \times 10^{-5}$  mol.kg<sup>-1</sup> in the molality is only  $\pm 0.01$  cm<sup>3</sup>.mol<sup>-1</sup> in a 1 molal solution, but ten times this value in a 0.1 molal solution. An error of  $\pm 1 \times 10^{-5}$  g.cm<sup>-3</sup> in the density of the sucrose solutions may be caused either by a temperature fluctuation of 10<sup>-2</sup>°K or a pressure fluctuation of 150 torr; the thermostat was constructed to maintain a given temperature to within  $\pm 5 \times 10^{-3}$ °K.

In order to obtain an accuracy of a few parts per million, the densities were measured differentially using three pyrex Ostwald-Sprengel type pycnometers of about 20 cm<sup>3</sup> capacity. Two of the pycnometers were filled with solution and one with water. The heights of the menisci above fixed marks were measured with a cathetometer. The pycnometers containing solution were each weighed against the water-filled tare on a double-pan balance. The differential arrangement minimizes errors due to changes in the temperature of the thermostat, and also decreases the errors due to the absorption of moisture on the surface of the pycnometers and in the buoyancy corrections. The absolute temperature of the thermostat was measured with an N.P.L.-certified platinum resistance thermometer to  $\pm 3 \times 10^{-3}$ °K.

The uniformity of the bore of the capillary tubes was checked before sealing into place, by measuring the length of a known weight of mercury in different positions. The lengths of the thread were measured on



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a Carl Zeiss Abbé comparator<sup>3</sup> to a precision of  $\pm 2 \times 10^{-4}$  cm. The volume of each pycnometer was determined using conductivity water, using an empty pycnometer as a weight tare. The results of several calibrations differed by less than  $\pm 1 \times 10^{-4}$  cm<sup>3</sup>. For the density of water the compilation of Kell<sup>4</sup> was used. The density of the stainless steel weights was taken as 8.0 g.cm<sup>-3</sup> and of pyrex glass as 2.25 g.cm<sup>-3</sup> for the buoyancy corrections; an error of 10% in these values would not affect the results by one part in 10<sup>6</sup>. The atmospheric pressure was read on a mercury barometer, corrected for temperature and the local value of the gravitational acceleration. A hair hygrometer was used for the relative humidity. Using these values, the air density was calculated from N.P.L. tables.

## Materials and solutions

The sucrose (Tate and Lyle research grade, 99.993% purity) containing 0.002% invert sugar, 0.002% ash and 0.003% organic matter was not further crystallized but dried *in vacuo* at 60°C and stored under vacuum. The water used was doubly distilled and had a conductance of less than 10<sup>-6</sup> S.cm<sup>-1</sup>. The water was always deaerated immediately before use. The potassium chloride (B.D.H. AR reagent) was recrystallized three times from conductivity water, dried *in vacuo* at 60°C and stored under vacuum. The solutions were prepared by weight. The molar mass of potassium chloride,  $M_3$ , was taken to be 74.555 g.mol<sup>-1</sup>, that of sucrose,  $M_2$ , 342.3019 g.mol<sup>-1</sup> and that of water,  $M_1$ , 18.0153 g.mol<sup>-1</sup>.

## RESULTS

### Binary system: Sucrose + water

The apparent molar volume was calculated from the density of the solution,  $\rho_2$ , using the equation

$$\phi V_2 = (\rho_0 - \rho_2) / \rho_0 \rho_2 m_2 + M_2 / \rho_2 \quad (1)$$

where  $m_2$  is the molality of sucrose,  $M_2$  its molar mass and  $\rho_0$  the density of water.

The average of duplicate runs at 30, 50 and 70°C is given in Table I. The apparent molar volume of sucrose was fitted to a power series in the molality of the form

$$\phi V_2 = V_2^\circ + Am_2 + Bm_2^2 + Cm_2^3 + \dots \quad (2)$$

The polynomial which gave the best statistical fit to the data was found to be of degree 3 at each temperature. The coefficients are given in Table IV. The plot of the apparent molar volume as a function of molality is shown in Fig. 1. The smooth curves were calculated from equation (2).

<sup>1</sup> Garrod & Herrington: *J. Phys. Chem.*, 1970, 74, 363.

<sup>2</sup> *J.S.J.*, 1966, 68, 104.

<sup>3</sup> Carl Zeiss Scientific Instruments Ltd., 31 Foley Street, London W.1.

<sup>4</sup> *J. Chem. Eng. Data*, 1967, 12, 66.



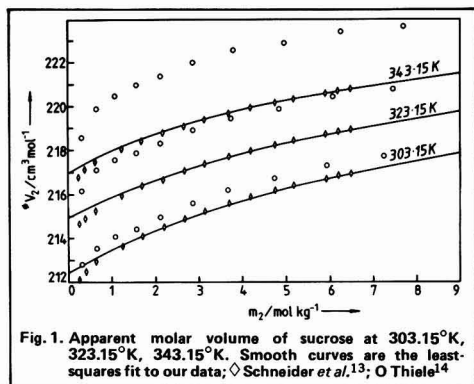


Fig. 1. Apparent molar volume of sucrose at 303.15°K, 323.15°K, 343.15°K. Smooth curves are the least-squares fit to our data;  $\diamond$  Schneider *et al.*<sup>13</sup>;  $\circ$  Thiele<sup>14</sup>

The partial molar volumes of water,  $V_1$ , and of sucrose,  $V_2$ , are given by

$$v_1 = v_1^\circ - M_1 m_2^2 (d^\beta v_2 / dm_2) \quad (3)$$

and

$$v_2 = \beta v_2 + m_2 (d^\beta v_2 / dm_2) \quad (4)$$

Values of  $V_2$  calculated using equations (4) and (2) are tabulated in Table I.

#### Binary system: Potassium chloride + water

The experimental results at 30, 50 and 70°C are collected in Table II. The apparent molar volume,  $\phi V_3$ , was fitted to a power series in  $m_3^{1/2}$ , where  $m_3$  is the molality of potassium chloride, given by the equation

$$\phi V_3 = v_3^\circ + k m_3^{1/2} + \beta m_3 + \dots \quad (5)$$

where the limiting slope,  $k$ , is given by Debye-Hückel theory. Values for  $k$  were taken from the recent calculations of Bradley & Pitzer<sup>5</sup>. The best fit to the data was given by just two coefficients,  $k$  and  $\beta$ . Values for these at each temperature are given in Table IV.

For an electrolyte equations (3) and (4) are modified as follows:

$$v_1 = v_1^\circ - M_1 m_3^{3/2} (d^\beta v_3 / dm_3^{1/2}) / 2 \quad (6)$$

$$v_3 = \beta v_3 + m_3^{1/2} (d^\beta v_3 / dm_3^{1/2}) / 2 \quad (7)$$

Values for  $V_3$  calculated using equations (5) and (7) are tabulated in Table II. The plot of the apparent molar volume as a function of molality with the appropriate value of  $k m_3^{1/2}$  subtracted is shown in Fig. 2.

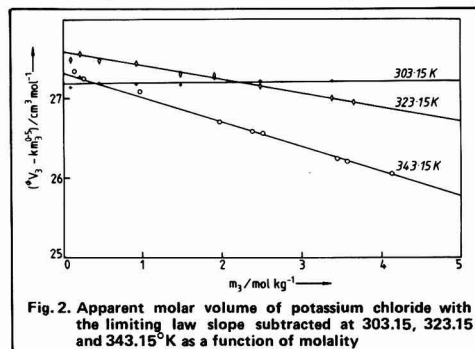


Fig. 2. Apparent molar volume of potassium chloride with the limiting law slope subtracted at 303.15, 323.15 and 343.15°K as a function of molality

Table I. Densities, apparent molar volumes and partial molar volumes of sucrose in aqueous solution at 30, 50 and 70°C

| $m_2$<br>(mol.kg <sup>-1</sup> ) | $\rho_2$<br>(g.cm <sup>-3</sup> ) | $\phi V_{2,obs}$<br>(cm <sup>3</sup> .mol <sup>-1</sup> ) | $V_2$<br>(cm <sup>3</sup> .mol <sup>-1</sup> ) |
|----------------------------------|-----------------------------------|---|--|
| T = 303.15°K                     |                                   |   |  |
| 0.00000                          | 0.995644                          | —   | 212.53   |
| 0.52963                          | 1.05735                           | 213.07  | 213.56   |
| 0.78513                          | 1.08268                           | 213.33  | 214.03   |
| 0.95635                          | 1.09838                           | 213.41  | 214.33   |
| 1.53554                          | 1.14465                           | 213.90  | 215.27   |
| 1.81760                          | 1.16392                           | 214.20  | 215.70   |
| 2.53072                          | 1.20566                           | 214.78  | 216.66   |
| 2.93874                          | 1.22582                           | 215.07  | 217.15   |
| 3.39908                          | 1.24596                           | 215.37  | 217.64   |
| 4.19364                          | 1.27543                           | 215.84  | 218.35   |
| 4.47354                          | 1.28456                           | 215.98  | 218.56   |
| 5.55135                          | 1.31443                           | 216.54  | 219.19   |
| 6.65055                          | 1.33874                           | 216.99  | 219.55   |
| T = 323.15°K                     |                                   |   |  |
| 0.00000                          | 0.988042                          | —   | 215.02   |
| 0.52963                          | 1.04887                           | 215.52  | 215.92   |
| 0.95635                          | 1.08938                           | 215.77  | 216.58   |
| 1.53554                          | 1.13500                           | 216.25  | 217.40   |
| 1.81760                          | 1.15407                           | 216.50  | 217.76   |
| 2.53072                          | 1.19538                           | 216.99  | 218.59   |
| 2.93874                          | 1.21534                           | 217.24  | 219.00   |
| 3.39908                          | 1.23533                           | 217.49  | 219.41   |
| 4.19364                          | 1.26450                           | 217.94  | 219.99   |
| 4.47354                          | 1.27361                           | 218.04  | 220.16   |
| 5.55135                          | 1.30326                           | 218.56  | 220.64   |
| 6.65055                          | 1.32746                           | 218.95  | 220.86   |
| T = 343.15°K                     |                                   |   |  |
| 0.00000                          | 0.97778                           | —   | 216.92   |
| 0.12293                          | 0.99303                           | 216.97  | 217.18   |
| 0.27099                          | 1.01033                           | 217.24  | 217.49   |
| 0.40396                          | 1.02499                           | 217.35  | 217.75   |
| 0.41093                          | 1.02577                           | 217.36  | 217.77   |
| 0.62917                          | 1.04807                           | 217.60  | 218.17   |
| 1.00125                          | 1.08203                           | 217.94  | 218.80   |
| 1.63444                          | 1.13029                           | 218.41  | 219.70   |
| 1.88811                          | 1.14687                           | 218.61  | 220.01   |
| 1.96707                          | 1.15168                           | 218.71  | 220.10   |
| 2.42227                          | 1.17772                           | 218.97  | 220.56   |
| 2.77774                          | 1.19557                           | 219.24  | 220.87   |
| 3.85746                          | 1.24055                           | 219.77  | 221.58   |
| 5.26516                          | 1.28372                           | 220.36  | 222.19   |
| 5.56930                          | 1.29155                           | 220.42  | 222.31   |
| 6.19959                          | 1.30597                           | 220.65  | 222.57   |
| 6.66491                          | 1.31553                           | 220.80  | 222.78   |
| 7.44219                          | 1.32971                           | 221.06  | 223.22   |
| 7.80899                          | 1.33583                           | 221.14  | 223.48   |
| 8.60067                          | 1.34771                           | 221.35  | 224.19   |

#### Ternary system: Sucrose + potassium chloride + water

The apparent molar volume of the sucrose in the ternary system,  $\phi V_3^*$ , was calculated using the equation

$$\phi V_3^* = (1 + M_3 m_3) (\rho_3 - \rho^*) / \rho_3 m_2 + M_2 / \rho^* \quad (8)$$

where  $\rho^*$  is the density of the ternary system and  $\rho_3$  the density of the electrolyte binary system at molality  $m_3$ . The apparent molar volume of the potassium chloride in

<sup>5</sup> J. Phys. Chem., 1979, 83, 1599.

**Table II. Densities, apparent molar volumes and partial molar volumes of potassium chloride in aqueous solution at 30, 50 and 70°C**

| $m_3$<br>(mol.kg <sup>-1</sup> ) | $\rho_3$<br>(g.cm <sup>-3</sup> ) | $\phi V_3$ obs<br>(cm <sup>3</sup> .mol <sup>-1</sup> ) | $V_3$<br>(cm <sup>3</sup> .mol <sup>-1</sup> ) |
|----------------------------------|-----------------------------------|---|--|
| T = 303.15°K                     |                                   |   |  |
| 0.00000                          | 0.995644                          | —   | 27.19  |
| 0.11692                          | 1.00108                           | 27.83   | 28.20  |
| 0.23867                          | 1.00661                           | 28.22   | 28.63  |
| 0.48599                          | 1.01766                           | 28.55   | 29.25  |
| 0.95627                          | 1.03787                           | 29.10   | 30.09  |
| 1.50660                          | 1.06043                           | 29.58   | 30.83  |
| 1.93686                          | 1.07714                           | 29.98   | 31.32  |
| 2.50778                          | 1.09862                           | 30.32   | 31.90  |
| 3.39665                          | 1.12996                           | 30.83   | 32.68  |
| T = 323.15°K                     |                                   |   |  |
| 0.00000                          | 0.988042                          | —   | 27.55  |
| 0.11692                          | 0.99341                           | 28.27   | 28.72  |
| 0.23867                          | 0.99886                           | 28.71   | 29.20  |
| 0.48599                          | 1.00973                           | 29.11   | 29.86  |
| 0.95627                          | 1.02956                           | 29.73   | 30.70  |
| 1.50660                          | 1.05177                           | 30.18   | 31.40  |
| 1.93686                          | 1.06826                           | 30.55   | 31.83  |
| 2.50778                          | 1.08945                           | 30.87   | 32.32  |
| 3.39665                          | 1.12044                           | 31.33   | 32.94  |
| 3.66933                          | 1.12955                           | 31.45   | 33.11  |
| T = 343.15°K                     |                                   |   |  |
| 0.00000                          | 0.977781                          | —   | 27.32  |
| 0.16733                          | 0.98539                           | 28.46   | 28.98  |
| 0.28323                          | 0.99054                           | 28.76   | 29.43  |
| 0.99438                          | 1.02062                           | 29.88   | 30.94  |
| 1.99004                          | 1.05968                           | 30.64   | 32.01  |
| 2.40154                          | 1.07482                           | 30.92   | 32.31  |
| 2.53631                          | 1.07962                           | 31.02   | 32.40  |
| 3.45071                          | 1.11141                           | 31.44   | 32.88  |
| 3.57478                          | 1.11556                           | 31.50   | 32.94  |
| 4.14047                          | 1.13387                           | 31.75   | 33.15  |

the ternary system,  $\phi V_3^*$ , was calculated from

$$\phi V_3^* = (1 + M_2 m_2) (\rho_2^* - \rho) / \rho_2^* m_3 + M_3 / \rho^* \quad (9)$$

where  $\rho_2$  is the density of the binary sucrose plus water solution at molality  $m_2$ .

The apparent molar volumes of the sucrose and of the potassium chloride in the ternary system were fitted to power series in the molalities (see discussion). The coefficients of the power series are given in Table V. The partial molar volumes of the solutes were then calculated in an way analogous to that used for the binary systems

$$V_2^* = \phi V_2^* + m_2 (\partial \phi V_2^* / \partial m_2)_{m_3} \quad (10)$$

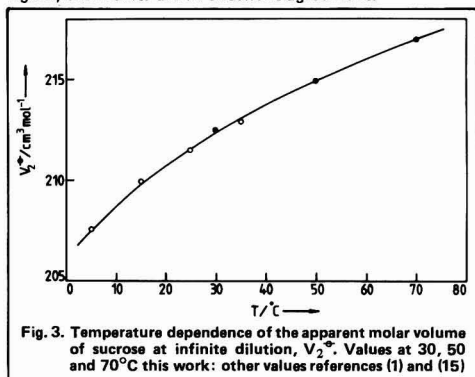
and 
$$V_3^* = \phi V_3^* + m_3 (\partial \phi V_3^* / \partial m_3)_{m_2} / 2 \quad (11)$$

**DISCUSSION**

*Binary system: Sucrose + water*

Our results for the apparent molar volume of sucrose at 30, 50 and 70°C are compared graphically in Fig. 1 with those of Schneider, Schliephake & Klímmek<sup>6</sup> and of Thiele<sup>7</sup>. The agreement with the results of Schneider *et al.* is good, but there appears to be a systematic discrepancy with the work of Thiele. Thiele used a dilatometric method and it is tentatively suggested that the

use of this method at high temperatures may have caused some of the sucrose to invert. In Fig. 3 the results of the present work are compared with previous work in this laboratory<sup>1,8</sup>. As can be seen from the figure, the results are in excellent agreement.

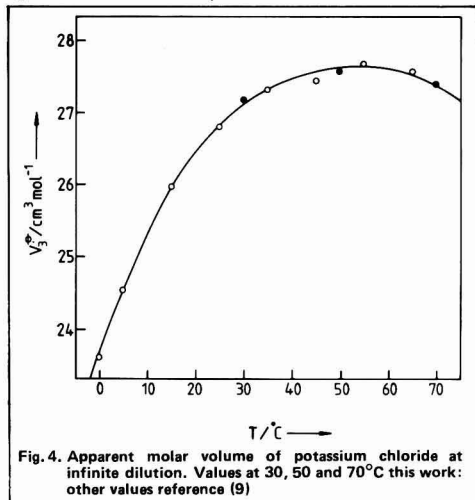


**Fig. 3. Temperature dependence of the apparent molar volume of sucrose at infinite dilution,  $V_2^*$ . Values at 30, 50 and 70°C this work; other values references (1) and (15)**

In order to compare data for the ternary system at 30, 50 and 70°C with those of Mantovani & Indelli<sup>2</sup> at 25°C, values were needed of the coefficients A, B and C for aqueous sucrose solutions at 25°C. Our own previous data at 25°C<sup>1</sup> were used, together with the four values of Mantovani & Indelli<sup>2</sup>; the best statistical fit values of the coefficients are given in Table IV.

*Binary system: Potassium chloride + water*

Our results for the apparent molar volume of potassium chloride at infinite dilution,  $\phi V_3^*$ , are compared with those of Dunn<sup>9</sup> in Fig. 4. It can be seen that the agreement is within experimental error. Dunn used a



**Fig. 4. Apparent molar volume of potassium chloride at infinite dilution. Values at 30, 50 and 70°C this work; other values reference (9)**

dilatometric method and was able to determine the apparent molar volume to  $\pm 0.01$  cm<sup>3</sup>.mol<sup>-1</sup> at molalities as low as  $3 \times 10^{-3}$  mol.kg<sup>-1</sup>. In order to interpret

6 Zucker, 1963, 16, 17.  
 7 Zeitsch. Zuckerind., 1962, 87, 424.  
 8 Mole: Thesis (Reading University), 1973.  
 9 Trans. Faraday Soc., 1968, 64, 2951.  
 10 Z. Physik. Chem., 1936, 34B, 1.  
 11 *ibid.*, 1934, 26B, 81.  
 12 Trans. Faraday Soc., 1968, 64, 1899.

Table III. Densities, apparent molar volumes of sucrose and of potassium chloride in their ternary aqueous mixtures at 30, 50 and 70°C

| $m_2$<br>(mol.kg <sup>-1</sup> ) | $m_3$<br>(mol.kg <sup>-1</sup> ) | $\rho^*$<br>(g.cm <sup>-3</sup> ) | $\phi V_{2\text{obs}}^*$<br>(cm <sup>3</sup> .mol <sup>-1</sup> ) | $\phi V_{3\text{obs}}^*$<br>(cm <sup>3</sup> .mol <sup>-1</sup> ) | $V_2^*$<br>(cm <sup>3</sup> .mol <sup>-1</sup> ) | $V_3^*$<br>(cm <sup>3</sup> .mol <sup>-1</sup> ) |
|----------------------------------|----------------------------------|-----------------------------------|---|---|--|--|
| T = 303.15°K                     |                                  |                                   |   |   |  |  |
| 0.00000                          | 0.00000                          | 0.995644                          | —   | —   | 212.53   | 27.19  |
| 1.05513                          | 0.33177                          | 1.11810                           | 213.95  | 29.63   | 214.60   | 29.64  |
| 0.96483                          | 0.48202                          | 1.11548                           | 214.16  | 29.95   | 214.62   | 29.92  |
| 1.15347                          | 0.63699                          | 1.13561                           | 214.48  | 30.30   | 214.88   | 30.38  |
| 1.29713                          | 0.72181                          | 1.14890                           | 214.70  | 30.56   | 215.00   | 30.63  |
| 1.43956                          | 0.95092                          | 1.16545                           | 215.08  | 30.93   | 215.13   | 31.12  |
| 1.61523                          | 1.17551                          | 1.18295                           | 215.43  | 31.25   | 215.54   | 31.57  |
| 1.84472                          | 1.46143                          | 1.20375                           | 215.88  | 31.66   | 215.01   | 32.09  |
| T = 323.15°K                     |                                  |                                   |   |   |  |  |
| 0.00000                          | 0.00000                          | 0.988042                          | —   | —   | 215.02   | 27.55  |
| 1.05513                          | 0.33177                          | 1.10881                           | 216.31  | 30.13   | 216.87   | 29.72  |
| 0.96483                          | 0.48202                          | 1.10623                           | 216.49  | 30.41   | 216.90   | 29.88  |
| 1.15347                          | 0.63699                          | 1.12610                           | 216.76  | 30.75   | 217.13   | 30.19  |
| 1.29713                          | 0.72181                          | 1.13926                           | 216.93  | 30.94   | 217.23   | 30.36  |
| 1.61523                          | 1.17551                          | 1.17294                           | 217.55  | 31.59   | 217.36   | 30.91  |
| 1.84472                          | 1.46143                          | 1.19357                           | 217.90  | 31.94   | 217.22   | 31.16  |
| T = 343.15°K                     |                                  |                                   |   |   |  |  |
| 0.00000                          | 0.00000                          | 0.977781                          | —   | —   | 216.92   | 27.32  |
| 0.32984                          | 0.14323                          | 1.02280                           | 217.53  | 28.97   | 217.88   | 28.34  |
| 0.51035                          | 0.56118                          | 1.05721                           | 218.56  | 30.30   | 218.80   | 28.59  |
| 1.39596                          | 1.48207                          | 1.15591                           | 219.53  | 31.52   | 219.22   | 28.53  |
| 1.84895                          | 1.11503                          | 1.17372                           | 219.53  | 31.52   | 221.27   | 29.19  |
| 2.34269                          | 1.87885                          | 1.21659                           | 220.27  | 32.26   | 222.05   | 28.20  |
| 3.01319                          | 2.69804                          | 1.25964                           | 220.95  | 32.88   | 222.51   | 26.45  |
| 3.81333                          | 3.32645                          | 1.29508                           | 221.43  | 33.31   | 222.70   | 25.13  |

the ternary data of Mantovani & Indelli<sup>2</sup> at 25°C, values of  $k$  and  $\beta$  were required at this temperature. The data of Kruijs<sup>10</sup>, Geffcken & Price<sup>11</sup> and of Dunn<sup>12</sup> were used; the best fitting values are given in Table IV.

appropriate binary systems of molalities  $m_2$  and  $m_3$ , respectively,  $\gamma_{23}$  represents the contribution of the electrolyte to the activity coefficient of the non-electrolyte in the ternary system and  $\gamma_{23}$  that of the non-

Table IV. Coefficients for calculating the apparent molar volume of sucrose,  $\phi V_2$ , and of potassium chloride,  $\phi V_3$ , in the aqueous binary systems

| T(°K)  | $V_2^{\text{obs}}$<br>(cm <sup>3</sup> .mol <sup>-1</sup> ) | A<br>(cm <sup>3</sup> .mol <sup>-2</sup> .kg) | B x 10 <sup>2</sup><br>(cm <sup>3</sup> .mol <sup>-3</sup> .kg <sup>2</sup> ) | C x 10 <sup>3</sup><br>(cm <sup>3</sup> .mol <sup>-4</sup> .kg <sup>3</sup> ) | $V_3^{\text{obs}}$<br>(cm <sup>3</sup> .mol <sup>-1</sup> ) | k<br>(cm <sup>3</sup> .mol <sup>-3/2</sup> .kg <sup>1/2</sup> ) | $\beta \times 10^2$<br>(cm <sup>3</sup> .mol <sup>-2</sup> .kg) |
|--------|---|---|---|---|---|---|---|
| 298.15 | 211.27 ± 0.11   | 1.680 ± 0.191                                 | - 24.5 ± 7.4  | 16.9 ± 7.7  | 26.82 ± 0.02  | 1.87  | 11.4 ± 3.8  |
| 303.15 | 212.53 ± 0.07   | 1.019 ± 0.090                                 | - 5.57 ± 0.30   | 0.73 ± 0.03   | 27.19 ± 0.03  | 1.96  | 0.993 ± 0.001   |
| 323.15 | 215.02 ± 0.08   | 0.887 ± 0.095                                 | - 5.03 ± 0.31   | 0.61 ± 0.03   | 27.55 ± 0.02  | 2.37  | - 17.0 ± 0.9  |
| 343.15 | 216.92 ± 0.02   | 1.101 ± 0.020                                 | - 11.29 ± 0.70  | 5.3 ± 0.55  | 27.32 ± 0.02  | 2.91  | - 36.8 ± 0.7  |

#### Ternary system: Sucrose + potassium chloride + water

In order to calculate  $\phi V_2^*$  and  $\phi V_3^*$  at any molalities  $m_2$  and  $m_3$  within the molality range investigated, it was necessary to express them as power series in the molalities. It is preferable to base the series on theoretical equations and the theory of Debye & McAulay<sup>13</sup> was chosen which we had used very successfully previously<sup>14</sup> for the osmotic coefficients of this ternary system. Following our previous notation<sup>14</sup>, the activity coefficients  $\gamma_2^*$  and  $\gamma_3^*$  of the non-electrolyte and electrolyte, respectively, in the ternary system may be written as

$$\ln \gamma_2^* = \ln \gamma_2 + \ln \gamma_{23} \quad (12)$$

$$\ln \gamma_3^* = \ln \gamma_3 + \ln \gamma_{32} \quad (13)$$

where  $\gamma_2$  and  $\gamma_3$  are the activity coefficients in the INT. SUGAR JNL., 1983, VOL. 85, No. 1020

electrolyte to the electrolyte activity coefficient. The activity coefficients  $\ln \gamma_{23}$  and  $\ln \gamma_{32}$  can be written as power series in the molalities

$$kT \ln \gamma_{23} = \eta_1 m_3 + \eta_2 m_3^{3/2} + \dots \quad (14)$$

$$v_k T \ln \gamma_{32} = \sigma_1 m_2 + \sigma_2 m_2 m_3^{1/2} + \dots \quad (15)$$

where the coefficients  $\eta_i$ ,  $\sigma_i$  are functions of the ionic strength and the dielectric constant of the solution. A quantity  $\Delta_{ij}$  is defined in terms of experimental quantities by

$$\Delta_{ij} = \phi_{ij} (m_2 + v m_3) - m_2 \phi_2 - m_3 \phi_3 \quad (16)$$

where  $\phi_2$  and  $\phi_3$  are the osmotic coefficients of aqueous

<sup>13</sup> Z. Phys., 1925, 26, 22.

<sup>14</sup> Herrington & Meunier: J. Chem. Soc., Far. Trans. 1, 1982, 78, 225.

solutions of component 2 only and of 3 only, respectively, and  $ij$  is the mole ratio of  $m_2$  to  $m_3$ . If  $\Delta_{ij}$  is represented as a polynomial in the form

$$\Delta_{ij}/m_2 m_3 = \sum_{p=0,1,2 \dots} \sum_{q=0, \frac{1}{2}, 1 \dots} X_{pq} m_2^p m_3^q \quad (17)$$

then it can be shown<sup>15</sup> by application of the Gibbs-Duhem equation that

$$1n\gamma_{23} = \sum \frac{p+1}{p+q+1} X_{pq} m_2^p m_3^{q+1} \quad (18)$$

$$1n\gamma_{32} = \frac{1}{v} \sum \frac{q+1}{p+q+1} X_{pq} m_2^{p+1} m_3^q \quad (19)$$

Thus  $v_2^* - v_2 = \sum \frac{p+1}{p+q+1} Y_{pq} m_2^p m_3^{q+1}$  (20)

and  $v_3^* - v_3 = \sum \frac{q+1}{p+q+1} Y_{pq} m_2^{p+1} m_3^q$  (20)

where  $Y_{pq} = RT(\partial X_{pq} / \partial P)_T$

Hence  $m_2(\phi_{v_2}^* - \phi_{v_2}) = m_3(\phi_{v_3}^* - \phi_{v_3})$  (21)  
 $= \sum \frac{1}{p+q+1} Y_{pq} m_2^{p+1} m_3^{q+1}$

To make the best use of our data we chose to fit

$$[(\phi_{v_2}^* - \phi_{v_2})/m_3 + (\phi_{v_3}^* - \phi_{v_3})/m_2]/2 = y_{00} + \frac{2}{3} y_{0\frac{1}{2}} m_3^{\frac{1}{2}} + \frac{1}{2} y_{01} m_3 + \frac{1}{2} y_{10} m_2 + \frac{2}{5} y_{01\frac{1}{2}} m_3^{3/2} + \frac{2}{5} y_{1\frac{1}{2}0} m_2^{\frac{1}{2}} + \dots \quad (22)$$

The best statistical fit to the data was obtained by adding terms that significantly improved the fit. For our data at 30 and 50°C the best fit was obtained with three terms — a constant term plus terms in  $m_3^{1/2}$  and  $m_2$ . At 70°C the same three terms gave an adequate fit, but not significantly better than two terms — a constant term plus a term in  $m_3^{1/2}$ . For the data of Mantovani & Indelli<sup>2</sup> at 25°C, the best fit was obtained with four terms — a constant term plus terms in  $m_3^{1/2}$ ,  $m_2$  and  $m_2 m_3^{1/2}$ . The coefficients for the best statistical fits to the data are given in Table V.

negative terms in  $Y_{0\frac{1}{2}}$  and  $Y_{10}$  counteract  $Y_{00}$  depends on the molalities of both components. It is perhaps significant that with increasing temperature only the extra term in  $Y_{0\frac{1}{2}}$  is required, implying that the long range electrostatic forces are dominating the intermolecular forces. The effect of changing molality on  $\phi_{v_2}^*$  and  $\phi_{v_3}^*$  is best illustrated graphically. In Figs. 5-8, smoothed values of  $\phi_{v_2}^*$  and  $\phi_{v_3}^*$  have been calculated using the coefficients of Tables IV and V.

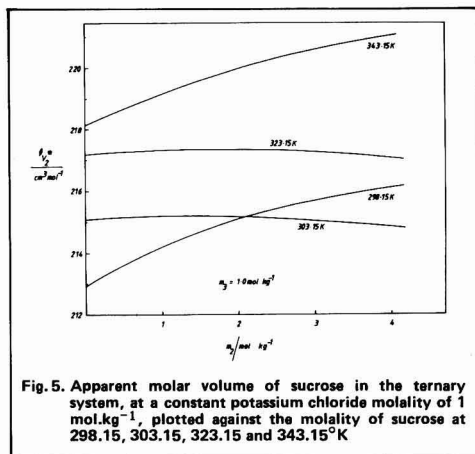


Fig. 5. Apparent molar volume of sucrose in the ternary system, at a constant potassium chloride molality of 1 mol.kg<sup>-1</sup>, plotted against the molality of sucrose at 298.15, 303.15, 323.15 and 343.15°C

The apparent molar volume of sucrose in the ternary system,  $\phi_{v_2}^*$ , as a function of its molality,  $m_2$ , is plotted in Fig. 5 for a constant potassium chloride molality of 1 mol.kg<sup>-1</sup> at 30, 50 and 70°C. The values for 25°C are calculated from the data of Mantovani & Indelli<sup>2</sup> using the coefficients given in Table V. It can be seen that  $\phi_{v_2}^*$  is essentially independent of the sucrose concentration in the region of 30-50°C, but increases with increasing molality outside this temperature range. In Fig. 6 the apparent molar volume of potassium chloride in the ternary system,  $\phi_{v_3}^*$ , is plotted against  $m_3^{1/2}$  for a constant sucrose molality of

Table V. Coefficients for calculating the apparent molar volumes of sucrose,  $\phi_{v_2}^*$ , and of potassium chloride,  $\phi_{v_3}^*$ , in the ternary system

| T(°K)  | $Y_{00}$<br>(cm <sup>3</sup> .mol <sup>-2</sup> .kg) | $2/3 Y_{0\frac{1}{2}}$<br>(cm <sup>3</sup> .mol <sup>-5/2</sup> .kg <sup>3/2</sup> ) | $1/2 Y_{10}$<br>(cm <sup>3</sup> .mol <sup>-3</sup> .kg <sup>2</sup> ) | $2/5 Y_{1\frac{1}{2}}$<br>(cm <sup>3</sup> .mol <sup>-7/2</sup> .kg <sup>5/2</sup> ) |
|--------|--|--|--|--|
| 298.15 | 2.201 ± 0.011  | - 0.603 ± 0.042  | - 0.236 ± 0.017  | 0.0737 ± 0.0117  |
| 303.15 | 1.653 ± 0.021  | 0.894 ± 0.167  | - 0.876 ± 0.117  |  |
| 323.15 | 1.688 ± 0.015  | 0.494 ± 0.156  | - 0.726 ± 0.108  |  |
| 343.15 | 2.317 ± 0.025  | - 1.084 ± 0.231  |  |  |

Thus  $\phi_{v_2}^*$  and  $\phi_{v_3}^*$  can be calculated from the formulae

$$\phi_{v_2}^* = \phi_{v_2} + y_{00} m_3 + \frac{2}{3} y_{0\frac{1}{2}} m_3^{3/2} + \frac{1}{2} y_{10} m_2 m_3 + \frac{2}{5} y_{1\frac{1}{2}} m_2^2 m_3^{3/2} \quad (23)$$

$$\phi_{v_3}^* = \phi_{v_3} + y_{00} m_2 + \frac{2}{3} y_{0\frac{1}{2}} m_2 m_3^{\frac{1}{2}} + \frac{1}{2} y_{10} m_2^2 + \frac{2}{5} y_{1\frac{1}{2}} m_2^2 m_3^{\frac{1}{2}} \quad (24)$$

At all temperatures  $Y_{00}$  is positive, so that considering this term alone, the effect of increasing the molality of potassium chloride increases  $\phi_{v_2}^*$  and *vice versa*. The effect of the other terms is more subtle and whether the

1 mol.kg<sup>-1</sup>. Again the values for 25°C are calculated from the Mantovani & Indelli data using the coefficients of Table V. Here, at all temperatures,  $\phi_{v_3}^*$  increases with increasing molality of potassium chloride.

To show the effect of increasing the concentration of the other solute at constant temperature, values for  $\phi_{v_2}^*$  and  $\phi_{v_3}^*$  are plotted in Fig. 7 and Fig. 8, respectively, at molalities of 1, 2 and 3 mol.kg<sup>-1</sup> of the other component. From Fig. 7 it can be seen that the effect on  $\phi_{v_2}^*$  of increasing the molality of sucrose depends markedly on the constant molality of potassium chloride. On the other hand, at a sucrose molality of 4 mol.kg<sup>-1</sup>,  $\phi_{v_2}^*$  is not very dependent on the molality of potassium

<sup>15</sup> Meunier: Thesis (Reading University), 1980.

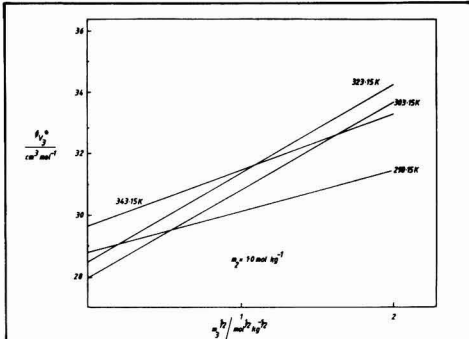


Fig. 6. Apparent molar volume of potassium chloride in the ternary system, at a constant sucrose molality of 1 mol.kg<sup>-1</sup>, plotted against the square root of the molality of potassium chloride at 298.15, 303.15, 323.15 and 343.15°K

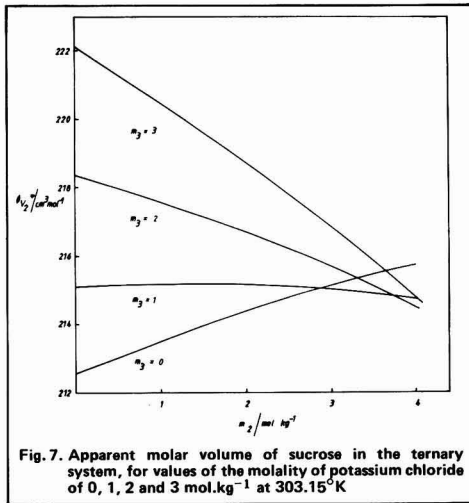


Fig. 7. Apparent molar volume of sucrose in the ternary system, for values of the molality of potassium chloride of 0, 1, 2 and 3 mol.kg<sup>-1</sup> at 303.15°K

chloride. In Fig. 8 it can be seen that increasing the molality of potassium chloride has the greatest effect on  $\bar{V}_3^*$ , the higher the molality of sucrose.

*Acknowledgement*

We wish to thank Tate and Lyle Ltd. for their interest in and support for this work and Dr. Peter Avery of the Department of Applied Statistics for help with the statistical analysis of the data.

*Summary*

Apparent molar volumes of sucrose and of potassium chloride in aqueous solutions and in their ternary mixtures have been determined over a wide range of concentration using a pycnometric technique at 30, 50 and 70°C. The data have been fitted to power series in the molalities and values of the partial molar volumes at infinite dilution determined. Values of the apparent molar volume can be calculated at any combination of molalities in the concentration range studied using the polynomial coefficients.

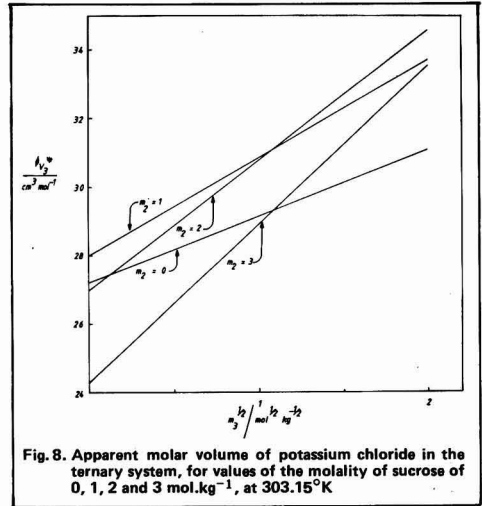


Fig. 8. Apparent molar volume of potassium chloride in the ternary system, for values of the molality of sucrose of 0, 1, 2 and 3 mol.kg<sup>-1</sup>, at 303.15°K

**Densités de solutions sucrées contenant du chlorure de potassium**

Le volume molaire apparent de sucrose et de chlorure de potassium en solution aqueuse et dans leurs mélanges ternaires ont été déterminés sur une vaste gamme de concentrations utilisant une technique picnométrique à 30, 50 et 70°C. Les renseignements ont été appliqués à des séries de puissances molales et les valeurs des volumes molaires partiels à dilution infinie ont été déterminées. Les valeurs des volumes molaires apparents peuvent être calculées à n'importe quelle combinaison molaire dans la gamme de concentration étudiée en utilisant les coefficients polynomes.

**Dichte von Zuckerlösungen, die Kaliumchlorid enthalten**

Das scheinbare molare Volumen von Saccharose und Kaliumchlorid in wässrigen Lösungen und ternären Gemischen der drei Substanzen wurde über einen weiten Konzentrationsbereich unter Verwendung einer pyknometrischen Methode bei 30, 50 und 70°C bestimmt. Aus den Daten wurde eine Potenzreihe für Molalitäten entwickelt und die Werte des partiellen Molarrvolumens bei unendlicher Verdünnung bestimmt. Werte des scheinbaren molaren Volumens können für jede Kombination der Molalitäten im untersuchten Konzentrationsbereich unter Verwendung der Polynomkoeffizienten berechnet werden.

**Densidades de soluciones de sacarosa que contienen cloruro de potasio**

Los volúmenes molares aparentes de sacarosa y de cloruro de potasio en soluciones acuosas y en sus mezclas ternarias se han determinados sobre un amplio alcance de concentraciones empleando una técnica picnométrica a 30, 50 y 70°C. Los datos se han aplicados a series de poderes molales y los valores de volúmenes molares parciales a dilución infinita se han determinado. Valores del volumen molar aparente puede calcularse para cualquier combinación de molalidades en el alcance de concentraciones estudiadas por uso de los coeficientes polinomios.



# CANE SUGAR MANUFACTURE

**Advantages of the introduction of cooling towers.** J. A. Penichet, E. Corona and C. Ramírez C. *ATAC*, 1982, 41, (3), 22-28 (Spanish). — The use of water-cooling towers for reducing the temperature of condenser return water has been studied at Central Orlando Nodarse in comparison with a fan-ventilated cooling module. Details are given of the design and performance of the equipment, and it is concluded that induced-draft towers are to be preferred, providing a greater and more stable vacuum with smaller power consumption.

**Evaluation of different evaporation schemes. II. Economic aspects.** M. Salerno and C. Vázquez. *ATAC*, 1982, 41, (3), 29-32 (Spanish). — It was earlier<sup>1</sup> concluded that, from the technical viewpoint, pressure evaporation was preferable. Economically, the savings in fuel have to be set against potential extra losses of sucrose by degradation at higher temperature. These are examined, bearing in mind variations in sugar and oil prices, and it is concluded that a pressure evaporation system is profitable for oil prices in the range \$14-25/tonne when the price of sugar varies between \$100 and \$600/tonne.

**General study of the sulphitation process in the sugar industry.** O. Jimenez C. *ATAC*, 1982, 41, (3), 58-65 (Spanish). — A literature survey from 1965 of the sulphitation process applied in cane sugar processing reveals the limited data on mass transfer and equilibrium. The increasing application of the process to syrup is noted as is its use in combination with other processes such as carbonatation. The quality of the raw material in manufacture of direct white sugar is a topic of much work, as is the quality of the sugar produced.

**Is it possible to improve energy efficiency in evaporation?** C. Vázquez, D. Clerch and G. González. *ATAC*, 1982, 41, (4), 14-18 (Spanish). — Calculations have been made for six variants of evaporation, based on the same fundamental features, and the energy consumption, steam usage, bagasse savings, etc. relative to the first are tabulated. This comprises a pre-evaporator and quadruple-effect evaporator with an operating pressure of 1.7 kg.cm<sup>-2</sup>. The second case (4% saving) is a quintuple-effect evaporator operating at 2.72 kg.cm<sup>-2</sup>, the third (22.5% saving) is the same but with vapour bleeding to heaters and pans from effects 1, 2, 3 and 4. The fourth case (23.5% saving) is the same as the third but with an operating pressure of 2.94 kg.cm<sup>-2</sup>. The fifth (27.3% saving) is identical to the fourth, but with an operating pressure of 3.00 kg.cm<sup>-2</sup> and the final effect at 0.6 kg.cm<sup>-2</sup>. The sixth case (31.6% saving) is a quintuple-effect evaporator with bleeding from all vessels, use of a thermo-compressor and an operating pressure of 3.1 kg.cm<sup>-2</sup>.

**Quality assurance in the manufacture of sugar mill plant and equipment.** R. F. Lumb and C. B. Venton. *Proc. Australian Soc. Sugar Cane Tech.*, 1983, 29-36. — Qual-

ity assurance (QA) is defined as "the means by which the purchaser ensures that the plant or equipment is inherently satisfactory for its planned duty, and that this satisfaction is not prejudiced by inadequate quality control during manufacture". It is pointed out that manufacturers have tended to be less concerned with the checking of incoming raw materials and components, inspection of work in progress and formal investigation of defects than with detection of defects in the finished product, whereas the proper approach would ensure that the incidence of defect was limited. QA is a matter of cooperation between the manufacturer and customer or independent consultancy, and can benefit both parties. The subject is discussed with reference to the metallurgical aspects of the failure of an induced-draft fan (and of its replacement) on a boiler at Cattle Creek sugar factory. Discussion on the best practical welding repairs between the manufacturers and factory staff led to a satisfactory conclusion, and the repaired fan operated satisfactorily during the 1982 season.

**The use of crystallizers and continuous centrifugals in the treatment of high-grade massecuite.** L. K. Kirby and A. G. Noble. *Proc. Australian Soc. Sugar Cane Tech.*, 1983, 123-130. — The performances of the *B*-massecuite crystallizers and continuous centrifugals at Racecourse factory were assessed during 1982. The eight air-cooled Blanchard crystallizers are arranged in two sets of four and have a total working capacity of 109 m<sup>3</sup>, providing a residence time of about 5.7 hours; the two continuous centrifugals are BMA K1100 machines. Results showed that the crystallizers reduced molasses purity by about 5.7 units and increased massecuite crystal content by 5.6%. On the other hand, there was a 3.7 unit rise in molasses purity in the centrifugals; excessive sugar losses have been found to occur in both batch and continuous machines handling *B*-massecuite and there is need for care in the spinning of this massecuite. Throughput during the trial period averaged 18.1 tonnes.hr<sup>-1</sup> per centrifugal; although the extent of crystal breakage in the machines has not yet been quantified, the problem was not serious enough to cause any problems in boiling (60% of the *B*-sugar is used as footing for shipment sugar, while the rest is remelted and returned to the syrup tank).

**Combined low-grade separation trials — Victoria mill.** N. J. Sichter, L. K. Kirby and G. A. Brotherton. *Proc. Australian Soc. Sugar Cane Tech.*, 1983, 131-136. Trials were conducted on low-grade massecuite purging in Western States CC6 continuous centrifugals, the performances of which were compared with that of a CC4 machine. Results showed that the CC6 had an average hourly throughput of 10.5 tonnes of massecuite, compared with 6.5 tonnes.hr<sup>-1</sup> in the CC4, although sugar losses in the CC6 ranged from 0.9 to 2.6 units compared with 0.5-1.7 in the CC4. Comparison of perforated with non-perforated loading pots showed no difference in performance.

**Processing low-purity C-massecuites at Racecourse mill.** R. Broadfoot, K. F. Miller and G. A. Wallace. *Proc. Australian Soc. Sugar Cane Tech.*, 1983, 137-148. Trials in 1981 and 1982 of the modified boiling scheme at Racecourse<sup>2</sup> are reported. The earlier trials showed that reduction in low-grade massecuite purity failed to increase sugar recovery in terms of final molasses purity, and it was considered possible to achieve any significant improvement only if the size of crystal and the masse-

<sup>1</sup> *I.S.J.*, 1983, 85, 344.

<sup>2</sup> Black & Wallace: *ibid.*, 177.

cuite crystal content in the continuous pan were increased and centrifugal performance improved. However, despite modifications to the processes and considerable improvement in centrifuging of both normal- and low-purity massecuites in 1982, there was still no increase in sugar recovery. The difficulties are attributed to problems in continuous boiling, including uncontrolled variations in massecuite Brix, particularly in the first vessel; occasional crystal dissolution was evident, despite stable conductivity, implying a satisfactory Brix. The decision not to boil-out the pan throughout the entire season may have contributed to the problems.

**A nephelometer for estimation of mud solids in juice.** O. L. Crees and E. Whayman. *Proc. Australian Soc. Sugar Cane Tech.*, 1983, 165-170. — While many factories have installed flocculant:juice ratio controllers which are certainly an advance on manual setting of positive-displacement pumps used for flocculant addition, there is need of a suitable suspended solids monitor, since settling rates are dependent on the ratio of added flocculant to suspended solids and on the actual solids concentration; hence, both liquid flow and solids content, i.e. solids flux, must be measured. The answer seems to lie in an optical device, because fibre absorbs ultrasonic signals and differences in density approach those caused by variation in juice Brix. However, until recently, no simple nephelometer system had been capable of operating in the virtually opaque environment of limed juice, but CSIRO have developed a new optical-fibre, back-scatter nephelometer. The instrument is briefly described, and tests with it are reported. To simulate factory conditions, bagacillo was washed on a 200-mesh screen and added as wet fibre to expressed juice adjusted to 15° Bx. A range of dried soil types were then ground by hand and added to aliquots of the juice. Measurements of the turbidity are shown graphically for six soils. Doubling the fibre level in some tests had very little effect on the nephelometer readings, indicating that variations in bagacillo content presented no problem. The wide range of readings for fixed amounts of soil showed that turbidity is clearly not a direct measure of soil concentration; however, in view of the fact that some soils were coarse and others fine, and assuming that the reading is a function of quantity and particle size, i.e. surface area, this relationship may be of greater value for controlling flocculant addition than concentration alone. Juice samples were subjected to the standard SRI batch settling test after addition of different amounts of flocculant, and the quantity required to give an initial preset settling rate of 40 cm.min<sup>-1</sup> thus established. A broad correlation was found between flocculant quantity and nephelometric measurement of turbidity before settling. Results showed that, although rapid settling could be achieved with juice of high soil content, the clarity obtained was markedly worse than at low nephelometer readings; as these results were characteristic of a flocculant hydrolysis below the optimum, it is suggested that the type of flocculant is affected by soil content, and a higher hydrolysis material would have to be used under conditions of variable soil input such as in the trials.

**The application of juice pH to the automatic control of dextranase addition.** P. A. Inkerman, J. W. Riddell and L. J. Riddell. *Proc. Australian Soc. Sugar Cane Tech.*, 1963, 205-208. — While the use of dextranase is considered a suitable means of treating deteriorated cane, it has been found that such cane exhibits considerable variability in its dextran content as a result of variations in climatic conditions, burn-to-cut and cut-to-crush

times, cane variety and size and degree of damage of the cane billet, so that there is need for a continuous and instantaneous method of monitoring the cane dextran content to permit adjustments to the enzyme dosage rate. Various procedures have proved unsatisfactory, and an investigation was carried out at Marian factory during the latter part of the 1981 crushing season, in which pH was used as an index of dextran concentration. The pH of first expressed juice was measured in the laboratory, and the signal from the meter inverted through a signal-conditioning circuit and fed into the automatic control terminal of a peristaltic pump responsible for delivering the enzyme solution to a sample bypass trough for combination with bypass juice intended for cane payment analysis; the bypass juice was then transferred to the mixed juice pit. The delay between crushing at the 1st mill and pH measurement in the laboratory was 2.6 minutes, while a further 3-minute delay occurred between addition of the enzyme and arrival of the juice at the mixed juice pit. The pH meter was calibrated over the range 4-5 in such a way that the pump operated at maximum speed at the lower value and virtually stopped at the higher value, so that the pump speed-dependent dosage rate was approximately proportional to the pH of first expressed juice. Results showed that the automatic control system approximately halved the amount of enzyme that would have been used to treat the cane; the 6-minute delay between crushing and dextranase addition did not appear to have any significant effect on performance of the system, but the correlation between pH and dextran concentration was not valid under all conditions. In the pH range 5-5.5 (the upper value being that of juice from fresh cane), pH tends to over-estimate the dextran level, but the problem is not considered serious; the lack of correlation is attributed to the presence in burnt cane of acid-producing organisms other than those responsible for formation of dextran.

**Gassing rates in molasses.** E. T. White, C. Tokody and P. G. Wright. *Proc. Australian Soc. Sugar Cane Tech.*, 1983, 209-214. — Storage of molasses (or massecuite) at high temperatures may cause gassing (foaming) as a result of chemical reactions that liberate CO<sub>2</sub>. Of the reactions, the most significant is believed to be the Maillard reaction between carbohydrates (usually reducing sugars) and amino-acids or amides. Gassing reduces the sugar content, causes changes in the reducing sugars:ash ratio and increases viscosity as a result of polymerization. The CO<sub>2</sub> bubbles formed are small and are retained in the mass for some time, so causing expansion, with subsequent overflowing or bursting of the seams of a sealed tank. If the temperature is sufficiently high, the gas generation rate may be high enough to cause an explosion. Investigations similar to those of Newell<sup>1</sup> were carried out to measure the rate of gas generation in molasses and determine the effects of a number of variables. Temperature had the greatest effect, while other factors having influence were the source of sample, Brix (the rate rising sharply at >60° Bx) and purity (final molasses showing a much higher gassing rate than A- or B-molasses at a similar Brix). Addition of KOH caused no measurable change in gassing rate. Adding mineral acids to give a pH of about 3 also caused little change, but did increase the reducing sugars content; addition of acid sufficient to reduce the pH to 1 caused a substantial increase in the gassing rate. Addition of glucose had no effect on the rate; this was also the case

<sup>1</sup> *I.S.J.*, 1981, 83, 54.

when asparagine was added, but additional glycine caused a 30% increase. Surfactants failed to reduce the gassing rate, although powdered sodium sulphite, bisulphite and dithionite mixed with the molasses gave small reductions in the rate. On the other hand, increased quantities of dithionite increased gassing and liberated  $\text{SO}_2$ . Since the investigations showed that there is little that can be done to reduce gassing once it starts, it is recommended to keep storage temperatures low and storage periods short.

**An evaluation of a horizontal vacuum filter.** O. L. Crees and A. L. Willersdorf. *Proc. Australian Soc. Sugar Cane Tech.*, 1983, 215-218. — Further trials on the use of horizontal vacuum filters to handle clarification mud are reported<sup>1</sup>. While washing efficiency was initially high where hydraulic displacement of feed liquid from the pores of the cake was the major mechanism and the amount of wash water used was approx. 200% on mud solids, increase in the amount of water (necessary to remove residual liquid trapped in pockets and small pores) led to a predominance of a diffusion mechanism, which is of low efficiency. The effect of the molecular weight of Sedipur TF2 flocculant was as found earlier; at a given dose, polymers of higher molecular weight give less porous cakes, and it is suggested that changes in the operating procedure would be needed for maintenance of a constant mud solids output at acceptable pol levels. The degree of hydrolysis of the surfactant had only very slight effect on residual pol. At a wash water rate of about 1000% on mud solids, and using flocculant, it is suggested that pol could be kept below 10% on mud solids at a loading up to  $35 \text{ kg.hr}^{-1}$ , equivalent to about 4.5 tch (at a mud solids level of 0.75% on cane). For a factory of 400 tch crushing capacity, this would necessitate a total filter area of about  $100 \text{ m}^2$ , by comparison with about  $225 \text{ m}^2$  required in the case of a rotary filter. On the other hand, trials<sup>2</sup> have indicated the possibility of improving rotary filter performance, while the capital costs per unit area of horizontal filters are of the order of three times those of rotary filters. Thus, despite a possible reduction in pol loss, wash water usage and filtrate recycle, it is considered unlikely that horizontal filters will offer any real economic advantage over rotary filters.

**Direct infra-red measurement of moisture in sugar and bagasse.** R. W. A. Luxford and C. Steggles. *Proc. Australian Soc. Sugar Cane Tech.*, 1983, 225-229. Tests were carried out on Anacon 106 and Quadbeam 475 infra-red moisture analysers with the aim of finding a better means of sugar moisture control in place of electrical conductivity as used for many years at Victoria factory but subsequently found to have insufficient correlation ( $r = 0.65$ ) with laboratory estimates of moisture content. The relative merits of the two instruments were not considered. Data for 40 samples, 20 each of two brands of sugar, showed that both instruments gave a correlation coefficient of 0.992 with one type of sugar, while the Anacon analyser gave  $r = 0.986$  and the Quadbeam instrument  $r = 0.982$  for the other sugar. Laboratory estimation of moisture content appeared to have a repeatability similar to that of the analysers and gave values within  $\pm 0.06\%$  of their readings. A limited trial was also conducted on use of the Quadbeam analyser to measure bagasse moisture (the Anacon instrument having been tested previously<sup>3</sup>). The instrument was mounted above a bagasse conveyor, which made determination of its accuracy very difficult; how-

ever, readings followed a pattern that would be expected under various milling conditions, and the instrument was sensitive to small changes in mill speed, feed level, etc. Both analysers are considered potentially useful tools for "fine tuning" of mills. However, because of their relatively high costs, decisions on their installation would depend largely on their application and the possible monetary savings they could bring about.

**Scale inhibitors for sugar mill evaporators.** O. L. Crees. *Proc. Australian Soc. Sugar Cane Tech.*, 1983, 231-235. In laboratory investigations, samples of Antiprex A, Busperse 49, Nalco 7400 and Spersol were analysed for their polyacrylate contents by conductimetric titrations and for molecular weight by viscosity in  $1 \text{ M NaNO}_3$  at  $30^\circ \text{C}$ ; residual monomer was determined by HPLC, and the ability of each scale inhibitor to retard the precipitation of calcium sulphate was also assessed. Results are tabulated, but it is pointed out that, because of the very simplified nature of the tests, they cannot be used to predict factory performance and should be used only as a rough guide until better test methods are developed. Factory trials, in which each additive was used for two weeks at 2.5 ppm active polymer, showed little difference in performance between the inhibitors, so that the main criterion for selection appears to be simply one of cost per unit active polymer until a reliable and more precise method of performance evaluation is available. Samples of scale collected from the evaporator showed a predominance of calcium silicate with low levels of magnesium and phosphate. There appeared to be no significant differences in thickness or composition between the scales formed in the presence or absence of the inhibitors, although failure to use any additive led to complete covering of all tubes in vessels 4A and 4B (found by experience at the factory to be the worst for scale accumulation) with a thin layer of scale at the weekend; when additives were used, areas of bare metal were visible in many tubes.

**Heat transfer coefficients for natural-circulation evaporators.** S. Y. Guo, E. T. White and P. G. Wright. *Proc. Australian Soc. Sugar Cane Tech.*, 1983, 237-244. Relationships between evaporation variables and heat transfer coefficients obtained by various authors have been based primarily on factory measurements where it has been difficult to make controlled changes in operating conditions. With statistical analysis to obtain possible correlations between variations in the coefficient and naturally occurring changes in plant conditions, it is sometimes difficult to separate effects, since the changes often result from changes in other conditions. Investigations were carried out with an experimental triple-tube evaporator at the University of Queensland. The effects of static liquor level (for which water was used), temperature difference  $\Delta T$ , boiling point of the liquor at the disengagement space pressure  $T_b$  and Brix on the value of the maximum heat transfer coefficient  $U_{\text{max}}$  were determined separately, and the results given in graph form. Multiple linear regression of the data gave the following equation:

$$U_{\text{max}} = 0.16 (100 - Bx)^{0.40 \pm 0.08} T_b^{0.25 \pm 0.20}$$

which correlates 95% of the data within  $\pm 25\%$ . The correlation is shown as straight lines for  $T_b = 70^\circ$  and  $100^\circ \text{C}$ ; the effect of  $\Delta T$  is not significant. Similarly,  $U_{\text{max}} = 1.1\mu^{-0.13 \pm 0.03}$ , where  $\mu$  = viscosity ( $\text{Pa}\cdot\text{sec}^{-1}$ ) and the previous dependence of  $T_b$  is now taken up in

<sup>1</sup> Hale *et al.*: *I.S.J.*, 1975, 77, 150.

<sup>2</sup> See also Crees *et al.*: *ibid.*, 1983, 85, 148.

<sup>3</sup> Luxford: *ibid.*, 1979, 81, 348.

the viscosity term. The effects of subcooled and superheated feed and tube plate submergence on evaporator performance should be quantified in further work; however, some of the effects need to be investigated with large factory evaporators, where there are no special provisions for downtake recirculation.

**An automatic scheme for juice handling and effect control.** J. A. Barbat, J. F. Knight and P. J. Mitchell. *Proc. Australian Soc. Sugar Cane Tech.*, 1983, 245-253. Details are given of the components of an automatic scheme for juice flow and evaporator control installed at Cattle Creek factory. The microprocessor-based digital system includes mimic displays of process values, valve positions, alarm conditions and drive status arranged logically for quick scan evaluation of the complete area. The cascade system used for juice flow control makes use of smaller tanks than would be required with individual tank controls; the secondary juice tank is controlled on the basis of the actual level in the evaporator supply juice tank — as the level in the latter tank rises, the set point for the former tank also rises, while the mixed juice tank uses the secondary juice tank level as its set point in the same way. Use of the combined volume of juice in all three tanks greatly reduces the tendency of any one tank to over-fill or empty. Details are given of evaporator steam and juice Brix control, as well as control of primary and secondary juice heaters.

**Reducing boiler tube wear.** N. C. Farmer, M. K. Moir, K. C. Jones and V. Mason. *Proc. Australian Soc. Sugar Cane Tech.*, 1983, 255-263. — A boiler of 181 tonnes.  $\text{hr}^{-1}$  steam capacity installed at Macknade factory and first operated in 1977 had to be shut-down on four occasions in 1980 because of tube erosion; despite extensive inspection and repairs for the 1981 season, a 25 x 25 mm hole was found in a furnace exit tube, and several other tubes in the row were found to be thin in October, while in December most of the mild steel shields at the entry to the convection tube bank were found to be eroded or oxidized to such an extent as to render them useless. Efforts were made in the following year to find practical solutions to the problem, and involved: changes to the flue gas flow path within the boiler in an attempt to reduce maximum flow velocities, the use of tube shields resistant to high-temperature oxidation, and the application of spray-on or brush-on tube coatings. Some reduction in tube wear was achieved after a triangular flow deflection "nose" was fitted to the convection tube bank where flow velocity was high (model tests having predicted a 12% decrease in peak velocity at this point), but the improvement could not be attributed with certainty solely to the change in flow conditions, since the rate of wear would also have been reduced as a result of a 6% fall in total ash throughput (because of a dry year) and operation of the boiler at a 4% lower excess air level. Two stainless steel and a nickel alloy shield withstood the high-temperature conditions without difficulty, the least wear being experienced by Sandvik 253MA stainless steel; however one difficulty lies in the means of strapping the shields to the tube, and this is discussed. Of 18 types of metallic coating sprayed onto tubes, only carbidic and ceramic types had a wear rate that was consistently better than with bare tubes, while ferrous and nickel alloy types are unsuitable. Two types of refractory pastes were also applied to a few tubes that had been brushed to remove loose deposits. Refractite lasted the entire season and would appear to be useful as a means of erosion control, although correct application is very important.

**Combustion characteristics of bagasse suspension boilers.** T. F. Dixon. *Proc. Australian Soc. Sugar Cane Tech.*, 1983, 265-271. — Investigations showed that carbon monoxide concentrations in flue gas from bagasse suspension boilers were less than 100 ppm during normal operation, indicating a high combustion efficiency. However, the large-scale turbulent mixing that occurs in the ignition zone adjacent to the bagasse inlets creates regions of low oxygen concentration which therefore contain residual CO; the passage of these discrete elements of gas through the furnace results in a recording of CO that is effectively discontinuous, as found in boilers at Pleystowe and Pioneer factories. When the oxygen concentration falls below the threshold level, the CO concentration increases rapidly to a peak value before declining at a slower rate. The unsteady nature of the combustion process results in oxygen fluctuations that are typically 1.5-1.8% between peaks as opposed to a concentration of 1% when combustion is very stable. The rear wall secondary air curtain was found to have greatest effect on CO emission; the curtain redirected the flame path towards the centre of the furnace and removed unburnt CO. No significant changes in combustion occurred as a result of variations in the front wall secondary air curtain. Uniform bagasse distribution is essential for stable ignition; intimate mixing must occur between the hot furnace gases and bagasse fibres to allow drying, fibre heating and ignition to proceed rapidly and uniformly, and it is therefore important for a uniform thickness of bed to form on the bagasse feeder belt and for the bagasse spinner to be able to effect complete disintegration of the bed. Where there is a lack of uniformity in bagasse supply and the distributor pressure is inadequate, excessive grate build-up may result, and bagasse deposits greater than 1 m are common. Under certain conditions, combustion cycling may take place over most of the grate area, resulting in considerable fluctuation in flue gas oxygen concentration, deterioration in stability and difficulties to maintain automatic control. Use of CO concentration as a combustion air trim signal has limitations because of the nature of the CO response and a restriction on the minimum oxygen level to satisfy grate cooling and char burn-out requirements. Oxygen trim control is recommended for maintenance of a long-term air:fuel ratio in the optimum range and for improvement in ignition stability during periods of poor combustion. A reliable indication of the flue gas oxygen concentration is essential for good operation.

**Alloy development: shredder hammer tips.** K. F. Dolman. *Proc. Australian Soc. Sugar Cane Tech.*, 1983, 281-287. The requirements of cane shredder hammer tips are discussed, the three basic types of material used for hammer tips in most Australian sugar factories are reviewed, viz. tungsten carbide, white iron and a hardfacing weld deposit, and their properties indicated. Tungsten carbide gives a maximum performance rating for 6-12 weeks (in contrast to 2-4 weeks and 1-2 weeks for white iron and hardfacing, respectively) but it is expensive and has low shatter resistance; hardfacing material is cheap, but the labour costs of welding are high, while white cast iron hammer tips display intermediate performance ratings in most cases. Details are given of a new material developed by Vickers Australia Ltd.; the structure comprises discrete particles of alloy carbides in a steel matrix — the carbides function as the wear-resistant medium while the steel matrix acts as



carbide binder. Castings of the material are similar in composition and structure to chromium carbide hard-facing weld deposits, but have a number of advantages over them, including a higher fracture resistance and the ability to be used in thick sections (greater than 25 mm) for hammer tip manufacture. While the wear resistance is improved by increasing the proportion of carbide, this leads to a corresponding decrease in fracture resistance; however, the problem has been overcome by bonding the casting to a tough, mild steel backing plate to produce a composite material called Dua Block, for which a patent application has been filed. Full-scale production trials at a number of sugar factories indicated that a Dua Block hammer tip of optimum size will give a service life equivalent to 500,000 tonnes of cane, although it is considered possible to double this by introducing some material and design modifications.

**The effects of varying cane fibre characteristics on milling performance.** V. Mason, B. P. Edwards and R. N. Cullen. *Proc. Australian Soc. Sugar Cane Tech.*, 1983, 289-295. — In recent years, some processing problems have occurred in Australian sugar factories because of an increased variability in cane fibre levels and fibre characteristics; some individual cane varieties are difficult to handle and process on their own (the shredded cane may have undesirable properties, may produce bagasse of high moisture content or may require a high milling torque), while cane varieties having entirely different processing characteristics may follow each other through the mill. As a result, engineers have had to make physical alterations to milling equipment and modify the milling process. Investigations of the effect of variation in cane fibre characteristics on milling performance are reported, and possible means of handling cane of excessively high or low fibre levels are indicated. Canes of low fibre content have good Brix: fibre ratios but, when shredded, have a porridge-like consistency which causes problems such as reabsorption; canes of high fibre content can be easily fed through the milling train, but their relatively low Brix: fibre ratio limits extraction. As part of a program to assess cane "millability", the Sugar Research Institute is to use a small-scale shredding and milling test rig to evaluate the characteristics of small parcels of cane.

**Programmed arcing of mills.** E. L. Hornblow and J. F. Knight. *Proc. Australian Soc. Sugar Cane Tech.*, 1983, 297-299. — Electric arcing of mill roller surfaces has become widely used, with engineers relying on preset arcing programs, visual inspection or estimation of roughness by feel to decide on the amount of arcing necessary for good mill performance. However, arcing is very costly in terms of labour and electrode consumption, so that there is need for a method of determining the minimum amount of arcing required for maintenance of good mill performance. Details are given of a semi-quantitative control chart technique developed to this end. Sample data were taken from the daily shift analysis sheets, and a simple computer program written in Basic for calculation of re-absorption differences, with fixed constants included such as top and delivery roll diameters, tooth depths, roll lengths, gearing ratios and delivery set openings. Variables to be entered included crushing rate, cane fibre content, juice purity, bagasse pol and moisture content and turbine speed. After analysis, calculated reabsorption differences for each shift were averaged to give a single daily value which

could then be compared with the preset control value, the primary aim being to keep the daily average above this. Results, demonstrated by a chart comparing the effects of arcing on a conventional basis with programmed arcing, have been satisfactory, with a 55% reduction in arcing costs over a 16-week period, while mill performance and roll condition were maintained at the same levels as in the previous season despite a mud content of 5.26% on cane compared with 4.81% in the preceding year.

**Torsional vibration of sugar mill gear systems.** J. G. Loughran. *Proc. Australian Soc. Sugar Cane Tech.*, 1983, 301-305. — When low-speed steam engines were used as cane mill drives, any inherent vibration or shock loading caused by imperfect mating of gear teeth was of minimal consequence, since it was simply adsorbed by the conservatively sized gearing and the physical size of the power plant. However, high-speed turbines coupled through various intermediate gearboxes to the low-speed mill gearing cannot absorb torsional vibrations which may occur throughout the entire gear train when individual systems in the train (often supplied by different manufacturers) are coupled together. A case study is described, in which a severe "clunking" noise could be heard emanating from the high-speed gearbox after installation of a drive train coupled to a No. 1 mill. A torsional dynamic model was used to predict natural frequencies and mode shapes for the gear system, and indicated very severe torsional oscillations. Replacement of the gear coupling with a torsionally soft coupling considerably reduced the amplitude of the oscillatory strain, although the oscillations are still significant. While Wilson<sup>1</sup> has indicated that torsional oscillations should be limited to less than 25% of the mean transmitted torque, the periodic oscillations in the case study exceeded 50% of the mean transmitted torque even in the modified system.

**Cush-cush.** S. J. Clarke. *Sugar Bull.*, 1983, 61, (17), 8. The quantity of cush-cush (bagacillo) occurring in milling depends on the cane variety, but will be typically in excess of 10% on cane. Bagacillo has a high sugar content and so has to undergo further extraction. In Louisiana the general practice is to return it to the main carrier. Analyses carried out during cane preparation tests showed considerable variation in moisture content (76-82%), fibre content (9.2-10.0%) and in Brix of the juice (9.8-16.5°); the difference resulted from differences in the direction of the bagacillo drag conveyor. Common practice is for the drag conveyor to carry the bagacillo over the screens counter to the direction of the main carrier, so that bagacillo from later mills will be washed by the juice of increasing concentration from the earlier mills, in contrast to good diffusion in which the material is washed with juice of decreasing concentration. Juice already extracted is recycled to the mill, bringing with it problems associated with juice deterioration. The difference between the two Brix extremes is equivalent to about 40% pol extraction, and it would be logical to return the bagacillo at the point in the milling train where the juice Brix matches that of the juice in the bagasse mat. Alternatively, the bagacillo may be extracted by a single-stage operation, e.g. by the last mill roll or by screw press, but intermediate washing is essential to avoid considerable pol losses. Other methods of bagacillo treatment are to be investigated later.

<sup>1</sup> "Practical solution of torsional vibration problems" (Chapman & Hall, London) 1956.



# BEET SUGAR MANUFACTURE

**The factory scale operation of the metathesis process and the processing of deteriorated beets.** D. Voit and T. C. Chadwick. *Sugar J.*, 1983, 45, (10), 16-19. — After research had shown that it was possible to remove  $\text{Ca}^{++}$  ions from solution using solid magnesium carbonate, a metathesis (double decomposition) process was developed and patented; it involves addition of freshly prepared Mg carbonate to filtered carbonatation juice, whereupon the Mg rapidly replaces the dissolved Ca, creating a mixed Ca-Mg carbonate. A buffer reaction takes place, stabilizing the pH of the thin juice. From the variation in the quantity of lime salts that can be removed by this means, it is concluded that the calcium is complexed to different degrees by compounds present in the juice. Filtration following addition of the carbonate proved difficult during the 1981 campaign at Imperial Valley, so that it was decided to allow the juice to enter the evaporators unfiltered; the results proved satisfactory, without any obvious change in the extent of scaling. Despite the new process, scaling proved a problem because of the poor quality of the beets, and the amount of Mg carbonate added was kept deliberately lower than that needed for removal of the dissolved Ca. Introduction of the process two-thirds of the way through the campaign stopped a gradual fall in pH, raising the entire pH profile of the juice after carbonatation and reducing the pH drop across the evaporator. No other pH control measure was required. The amount of Mg used was gradually increased in an effort to combat the worsening situation, and the final quantity equivalent to 0.9 lb MgO per short ton of beets sliced proved satisfactory. The use of thick juice for purity control combined well with the process, major advantages of which are listed.

**Percolating extraction of beet cosettes.** H. Zaorska. *Ind. Alim. Agric.*, 1983, 100, 171-175 (French). — See *I.S.J.*, 1981, 83, 343.

**Coagulation of mud particles in 1st carbonatation juice with the use of flocculants.** I. F. Bugaenko, T. N. Samoilova, E. P. Ishina and A. R. Saponov. *Sakhar. Prom.*, 1983, (5), 34-37 (Russian). — Experiments to determine the effect of active silicic acid, added as flocculant, on the zeta-potential of 1st carbonatation mud particles are reported. Model solutions with pure  $\text{CaCO}_3$  were used, and the flocculant added in quantities of 1-7.5 ml of 0.1% acid per 200 ml solution. On the basis of the fall in zeta-potential with increase in silicic acid addition, the mechanism of mud coagulation is explained in terms of formation of aggregates consisting of  $\text{CaCO}_3$  particles with smaller silicic acid particles between them; the carbonate and acid became linked as a result of differences in their electrical charge (the carbonate having a positive and the acid a negative charge) and under the effect of molecular forces of attraction. However, in factory juices, the  $\text{CaCO}_3$  particles acquire a negative charge through the dominant effect of macromolecular polyelectrolytes which, when

adsorbed on the carbonate, form bridges with some mud particles, but leave enough of their own particles free to prevent neutralization of the charge. In these circumstances, coagulation takes place as a result of bond formation between  $\text{Ca}^{++}$  ions and the acid groups in the flocculant and polymers.

**Heating low-grade massecuite with ultra-high frequency currents.** L. I. Trebin, I. G. Bazhal, L. A. Kupchik and I. S. Gulyi. *Sakhar. Prom.*, 1983, (5), 39-40 (Russian). UHF (microwave) reheating of low-grade massecuite was tested at a frequency of 2450 MHz and a heating rate of 3, 5 and 12°C per minute. Raising the temperature from 40°C to 65°C at the two higher heating rates caused insignificant dissolution of crystals and the mother-liquor pol reading remained unchanged; at a rate of 3°C per min, there was some crystal dissolution. The massecuite was spun without any difficulties, and molasses viscosity and purity were below the minimum standards set.

**Experience in the use of hydrostatic membrane transducers for level measurement in beet sugar manufacture.** K. F. Gerbut, E. V. Deryuga and V. I. Radalovskii. *Sakhar. Prom.*, 1983, (5), 44-45 (Russian). — A pneumatic system incorporating a diaphragm unit mounted on the wall of a vessel, an air feed controller and a differential manometer is described. The system has proved satisfactory as a means of measuring level as a function of the pressure exerted by the column of liquid in the case of massecuite and milk-of-lime, and has also been applied to measurement of syrup Brix and milk-of-lime density.

**Computer simulation of the forced convection cooling of sugar beets.** R. M. Holdredge and R. E. Wyse. *Trans. ASAE*, 1982, 25, (5), 1425-1431; through *S.I.A.*, 1983, 45, Abs. 83-300. — A simple one-dimensional mathematical model was developed to predict the temperature variation in a beet pile subject to forced ventilation. A digital computer program enabled beet temperatures to be estimated as a function of time and position, given the ambient air temperature, initial pile temperature and ventilation flow rate. Predicted temperatures agreed well with measured values in an actual storage pile during the initial cool-down period; agreement was better at a ventilation rate of 5.2 m<sup>3</sup>.ks<sup>-1</sup>.tonne<sup>-1</sup> than at 10.4 or 20.8 m<sup>3</sup>.ks<sup>-1</sup>.tonne<sup>-1</sup>. The model is not applicable to frozen beet.

**Cavitation-erosive wear of metals in corrosive media.** A. I. Nekoz, G. A. Preis and N. A. Sologub. *Trenie i Iznos*, 1981, 2, (4), 596-604; through *S.I.A.*, 1983, 45, Abs. 83-340. — Experiments on corrosion-erosion of metals by water and aqueous solutions (sugar, salt, acid, alkali) are reported. The physical action of the solutions was intensified by relative movement: (a) jet impact, (b) rapid rotation, (c) magnetostriuctive vibration (ultrasound). Diffusion juice caused more wear than water, but less than (acetic or citric) acid at pH 6.5; addition of 15% sucrose to the latter considerably decreased wear. The microhardness (adhesion of surface layers) of armco iron and stainless steel increased considerably during the action of 15 or 30% sucrose solution (more than with water, acid or alkali); this is ascribed to higher density giving more intense micro-impact and longer relaxation time.

**The sugar industry in Denmark.** Anon. *Zuckerind.*, 1983, 108, 435-438. — A survey is presented of the Danish sugar industry, as represented by the five factories owned by De Danske Sukkerfabrikker and one factory

belonging to Sukkerfabriken Nykobing Limiteret. A brief account is given of the history and development of the industry, as well as information on the various activities of the two companies and on diversification.

**Extraction in the sugar industry within the framework of a more general model.** J. Drago. *Zuckerind.*, 1983, 108, 439-444 (German). — The Silin diffusion model is examined. While it is only approximate, a precise monoporous model is obtainable by applying Fick's law to the non-steady mass balance for volume change within the particle. The main weakness of the model is its inability to explain the reduction in diffusion coefficient with decrease in particle size and with increase in the diffusion time. A more general model, of biporous form, is proposed in which a solid particle has a double porous structure (macro- and micro-porosity) and which offers explanations for anomalies in diffusion coefficient changes. It is shown that at the start of extraction and low Fourier numbers, the process is dominated by the macro-structure, whereas at high Fourier numbers (as generally experienced with commercial diffusers) the micro-structure dominates and determines diffusion losses. The proposed model includes the precise Silin model as a special case and takes account of the effect of external film resistance. A factor representing the difference between actual and theoretical diffusion is introduced; calculated from operational data, it is mathematically the number by which the solids residence time must be multiplied so as to equate actual with theoretical losses.

**A filter station for 2nd carbonatation juice.** L. Jaromersky, M. Sterzinger and M. Horinek. *Listy Cukr.*, 1983, 99, 99-103 (Czech). — The performance of a KZF 68 filter-thickener used to treat 2nd carbonatation juice was determined during 1979-81. A flow diagram is given of the 3-unit station at Opava, and results indicate the suitability of the KZF 68 for both 1st and 2nd carbonatation juice. At an hourly throughput of 0.9-1.2 m<sup>3</sup> juice per m<sup>2</sup> filtration area (compared with a stipulated 0.6 m<sup>3</sup>.m<sup>-2</sup>.hr<sup>-1</sup>) and a feed pressure of 25-30 kPa (compared with a stipulated 70 kPa), initial mud concentration was 500-600 g.litre<sup>-1</sup> (compared with a stipulated 450 g.litre<sup>-1</sup>) but fell to 200 g.litre<sup>-1</sup> after 10 minutes.

**Choice and behaviour of material for batch sugar centrifugal baskets.** H. K. Maushagen. *Zuckerind.*, 1983, 108, 531-540 (German). — Requirements of materials used for the construction of batch centrifugal baskets are discussed with regard to the stresses that commonly occur, including those in the monitor casing under the effect of centrifugal force and those resulting from metal fatigue and corrosion. Determination of fatigue strength and causes of failure are described, and causes and types of corrosion are explained with the aid of photographs. The mechanical properties, corrosion resistance and workability of certain steels are indicated and the importance of proper planning, manufacture and operation of centrifugal baskets for safety in daily use is discussed.

**Effect on pressure loss of a plate heat exchanger in the fluidized bed of a continuous fluidized bed dryer for crystal sugar.** R. Glaser and M. Styczynska. *Zuckerind.*, 1983, 108, 542-546 (German). — Until now, no equations have been developed to describe air flow conditions in a fluidized bed equipped with additional elements such

as a heat exchanger intended to increase the sugar cooling rate. Investigations were carried out on an experimental unit provided with a 1-, 2- and 3-plate heat exchanger. Results are given in graph form showing the effect of air flow velocity on the height of the stationary fluidized bed and on flow resistance. Correlation equations were derived from the results which showed that: the pattern of change in flow resistance in the presence of a heat exchanger (necessary for continuous operation) differs widely from that of a dryer of constant diameter without heat exchanger; the pressure loss in the fluidized bed is a function of the heat exchanger geometry and of the height of the sill above and to the side of the grid, and theoretically decreases with increase in air flow up to a given maximum interspace volume; and the optimum with respect to level of resistance is a 3-plate heat exchanger.

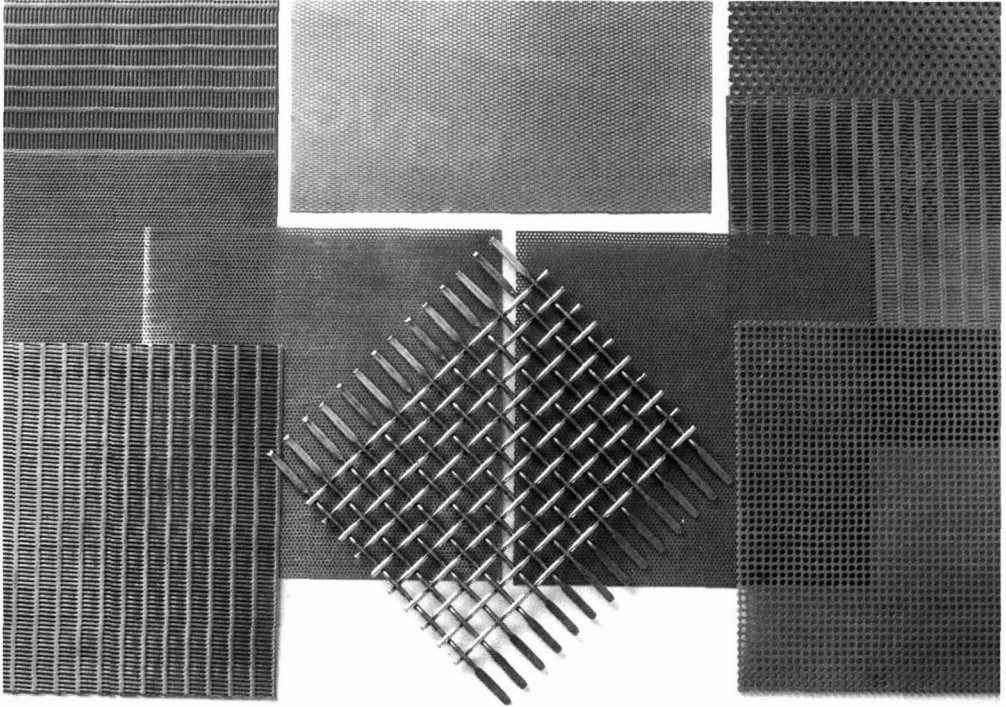
**New method for the continuous crystallization of sugar.** G. H. de Vries. *Sugar Tech. Rev.*, 1983, 10, 3-38. — The theory and aims of continuous boiling are discussed and details given of a continuous process and unit that were the subject of a patent application in 1969 and are the basis of a multi-feed unit currently under construction in Holland. This comprises a seed mixer, a ripener and a 5-compartment crystallizer. Liquor is first saturated in a concentrator under a given pressure. It then passes to the ripener, in which initial grain formation takes place under the effect of a seed slurry from the mixer. The liquor then flows to the crystallizer, where the growth zone and maximum rate of growth decline with flow from the first to the last compartment, so that the time taken for the liquor to flow through each compartment remains the same. A series of papers by the author and published between 1935 and 1960 are appended to illustrate the development of the theory of boiling on which the proposed technique is based.

**A study on the formation factors of crystal size distributions in industrial sucrose crystallizers.** A. Pol, L. J. Kuijvenhoven, L. H. de Nie and E. J. de Jong. *Paper presented at 17th Gen. Assembly CITS*, 1983, 24 pp. Studies of factors affecting crystal size distribution carried out in laboratory, pilot plant and factory equipment are reported. A masecuite stirrer was used, and both batch and continuous boiling carried out. It was concluded that growth rate in industrial pan boiling for crystals >100 μm is governed by the volume diffusion mechanism and may be described mathematically on the basis of a modification of Fick's equation. This mechanism is affected by differences in the hydrodynamic conditions of the mother liquor surrounding the crystals, causing size-dependent growth. For crystals <100 μm the growth rate increases rapidly in favour of the smaller crystals as a result of replacement of the volume diffusion mechanism by surface diffusion as the rate-determining factor. Secondary sucrose nucleation occurred predominantly on the surface of small crystals. Fines destruction may occur, probably as a result of heterogeneity in process conditions, although the optimum conditions have not been established. Conglomeration occurred only with crystals smaller than 100 μm and was probably based on the same mechanism as secondary nucleation; instead of being removed from the crystal surface, the nuclei became linked to the parent crystals, after which both grew out together as a conglomerated crystal. Attrition, abrasion and crystal breakage were of no significance in crystallization, provided the space between crystals was large relative to the average crystal size. Small seed crystals markedly favoured nucleation, whereas large crystals had hardly





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any effect. Poor mixing of highly concentrated feed with the pan contents could induce excessive nucleation and conglomeration.

**Some aspects of the chemistry of pulp pressing aids.** M. Shore, J. A. Adams, N. W. Broughton, N. Bumstead and G. C. Jones. *Paper presented at 17th Gen. Assembly CITS*, 1983, 27 pp. — As part of laboratory studies of pulp pressing aids, an investigation was conducted on the uptake of certain inorganic cations by beet pulp. Results showed that the affinity was in the decreasing order:  $Al^{+++} > Ca^{++} > Mg^{++} > Na^{+} > K^{+}$ . This behaviour of the pulp is analogous to that of carboxylic weakly acidic cation exchange resins and is probably due to ion exchange at the carboxyl groups on the pectin molecules in the pulp. At about pH 5, maximum adsorption of Ca on the pulp was prevented by previous extraction of the pulp with water; this is explained by the greater affinity of carboxyl groups for  $H^{+}$  ions than for  $Ca^{++}$  ions as demonstrated by a reduction in Ca adsorption at lower pH. The likely extent of cation adsorption on pulp, and hence its suitability as a pressing aid, can be rapidly estimated on the basis of the cation exchange resin analogy. From this, it is seen that Mg salts are unlikely to be as effective as Ca salts.

**The application of hyperfiltration for water removal in the sugar industry.** R. F. Madsen. *Paper presented at 17th Gen. Assembly CITS*, 1983, 15 pp. — Experiments during 1981-82 are reported on the application of new, improved thin-film composite membranes for hyperfiltration of thin juice as a means of reducing the water content. Hitherto a major limitation has been the inability of membranes to operate at temperatures above 35°C, whereas the HR membranes used in the experiments were able to operate at up to 80°C. At a pressure of 40 bar and a juice Brix of 14°, flux rose with temperature up to 80°C; however, because of increase in membrane compaction, flux at 80°C as a function of time would fall to a greater degree than at e.g. 60°C, so that it might be possible to obtain higher capacities at the lower temperature. Within a range of 8-10, pH had no effect on flux. Water removal was approximately 20% on beet. The membranes were cleaned daily with 0.5% nitric acid at 20-30°C in 1980 and at 50-60°C (followed by use of an alkaline detergent) in 1981, after which water flux and salt permeability were measured. There was a tendency for flux to fall over the test period — from 70 to 35 litres.m<sup>-2</sup>.hr<sup>-1</sup> after the first few days in 1981; salt permeability remained at 1-2% over the period. Enzymatic determination of sucrose in the permeate revealed up to 30 ppm; GLC analysis revealed the same content of volatile compounds as in the condensate from the 1st effect of a factory evaporator. Despite the improvement in performance, the capacities of the membranes are still insufficient to make the process economically viable as replacement of part of the evaporation process.

**Energy savings in the crystallization station.** 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. *Paper presented at 17th Gen. Assembly CITS*, 1983, 22 pp. — Details are given of a vacuum pan automation system developed by CSM at their Breda sugar factory in collaboration with Süddeutsche Zucker-AG and Selwig & Lange. The program was intended to optimize steam flow in the pan station, control the rate of boiling and develop a separate unit for preparation of 300 µm seed crystals. Reduced water evaporation in the pans permitted the use of 3rd vapour for 1st and 2nd product boiling. Temperature set points can be changed through control

of the absolute pressure. (Careful control of the temperature gradients is necessary to avoid too fast a boiling process and hence formation of false grain and higher entrainment losses.) Powerful nucleation was found at all stages of the boiling cycle, particularly during and after seeding. While a high feed syrup Brix (up to 75°) is desirable from the point-of-view of fuel economy, it favours the survival of small nuclei and thus adversely affects crystal size distribution in the massecuite. However, by removing slurry seeding and crystal development from the pan to the special seed preparation unit, newly formed nuclei could be dissolved and the sugar allowed to crystallize on the seed slurry. The automatic boiling system allowed a saving of 0.3% fuel on beet, a reduction of 20% in the boiling time and production of a sugar almost free from conglomerates and with a C.V. of 35.

**Operation experience with the application of a heat pump system in the evaporation station of a sugar factory.** P. Christodoulou. *Paper presented at 17th Gen. Assembly CITS*, 1983, 26 pp. — Details are given of a heat pump system installed in the evaporator station of Platý sugar factory in Greece. By this means, saturated vapour of 1.9 bar from the 2nd effect is compressed to a pressure of 3.4 bar using live steam of 25 bar, the mixture then entering the steam chest of the 1st effect. The result has been an increase in the evaporation capacity of the station as well as in the factory slice, which reached 9300 tonnes per day with the same evaporator heating surface as before (14,440 m<sup>2</sup>). Boiler fuel consumption has been reduced, while boiler feed water quality has not suffered from recycling of the vapour; at the same time, thermal pollution of the environment has been decreased as has the energy requirement for cooling of the low-temperature vapour. The economics are discussed.

**The kinetics of impure syrups applied to industrial sucrose crystallization.** V. Maurandi and A. Rossi. *Paper presented at 17th Gen. Assembly CITS*, 1983, 18 pp (French). — Investigations of the kinetics of intermediate-product boiling are reported, which showed close agreement, for massecuite of 88-91 purity, between data obtained experimentally and values given by empirical formulae derived from laboratory tests. The studies have also permitted evaluation of the order of magnitude of the fall in supersaturation consequent on the fall in effectiveness of the evaporation surface towards the end of a strike. With reference to the Ostwald-Weber diagram showing the probability of spontaneous nucleation, supersaturation in intermediate strikes was found to fall between the limiting value of pure sucrose solution (1.20) and that of low-grade massecuite (approx. 1.40) and averaged 1.26 with a standard deviation of 0.04.

**Industrial data processing in energy optimization within the manufacturing process.** G. Windal. *Paper presented at 17th Gen. Assembly CITS*, 1983, 17 pp (French). In order to demonstrate the applicability of the micro-processor to automatic control and optimization of sugar factory processes, the author describes a basic structure (the CHEOPS) that embodies data processing, distributed and hierarchical control, and automatic data gathering from the controlled processes for updating of the programs. Examples of processes controlled by this means include the pan and centrifugal station, De Smet and RT diffusion and beet yard operations; also mentioned is the management of technical data and production



of balances using the CLEOPATRA mini-computer system. (See also *I.S.J.*, 1983, 85, 312, 345.)

**Pan vapour recompression.** J. C. Giorgi. *Paper presented at 17th Gen. Assembly CITS*, 1983, 8 pp (French). Details are given of the pan vapour recompression system installed at Bucy-le-Long and Erstein sugar factories, where the 1st massecuite pan is a FCB horizontal, continuous unit and the recompressor is a 3-stage centrifugal type. The advantages of the system in terms of heat gain are discussed. Although the system is the same at both factories, conditions are different; at Bucy-le-Long the amount of vapour aspirated by the compressor is distinctly smaller than that produced by the pan, whereas at Erstein the installed power is very much higher than the theoretically required amount.

**Utilization of the heat content of carbonation vapours.** G. Witte and H. Schiweck. *Paper presented at 17th Gen. Assembly CITS*, 1983, 17 pp (German). — Because of their high water content, the waste gases from 1st and 2nd carbonation contain relatively large amounts of heat. Details are given of tests with an experimental plant installed at Rain sugar factory for condensation of 1st carbonation flue gas. Some of the vapour was withdrawn by radial fan and fed into the suction stream of a spiral heat exchanger for raw juice heating. It has been found that the level of heat loss in carbonation is governed by temperature and CO<sub>2</sub> utilization efficiency. Average CO<sub>2</sub> utilization was 71% (at a feed gas content of 35% CO<sub>2</sub> by volume), and the temperature of the vapour fed to the heat exchanger was approx. 85°C; raw juice was heated from 23.5° to 38.7°C, while other advantages were the reduction in odour emission that is normally associated with carbonation flue gas as well as recovery of ammonia for use as N source in biological treatment of effluent. The temperature of 2nd carbonation flue gas is calculated at 92°C.

**Study of a sugar factory with vapour compression.** T. Baloh. *Paper presented at 17th Gen. Assembly CITS*, 1983, 15 pp (German). — In a conventional sugar factory, two-thirds of the total fuel consumption is used to raise steam and one-third for pulp drying. One possibility of reducing consumption is to use exhaust gas from a turbine to dry the pulp and couple the turbine with a generator for production of electricity to be used in the factory. It is calculated that drying all the pulp to the same level as in a conventional factory but using turbine exhaust gas at 450°C will also allow production of 6.15 kWh per 100 kg beets sliced, which is double the normal power requirement of a factory. In view of this excess of electricity, the factory can be provided with electrically operated vapour compressors for both the evaporator and pan station, permitting a reduction in heating steam consumption from 23.45 to 3.6 kg/100 kg beets. The steam can be generated in a small low-pressure boiler. Such a system would eliminate the need for high-pressure boilers, condensation plant and water recycling plant.

**Possibilities of improving the energy economy in sugar recovery.** K. E. Austmeyer and U. Bunert. *Paper presented at 17th Gen. Assembly CITS*, 1983, 32 pp (German). With the aid of an energy flow diagram, the authors indicate the waste heat potential of boiler flue gas, the unused part of pan vapours, condensate and pulp dryer waste gas in a white sugar factory. Various possibilities of using this hitherto only partially tapped source of energy are described. They include the use of vapour

compression in the sugar house, and boiling on magma footing, pre-heating of combustion air for steam generation and pulp drying, as well as low-temperature pulp drying and use of flue gas, and recycling of pulp dryer exhaust gas or its partial condensation for sugar solution concentration. The effect of mechanical dewatering on energy saving is discussed, as is the possibility of using methane gas from effluent treatment as a minor supplementary source of fuel. The economics of the various schemes are discussed.

**pH values in juice purification.** O. C. Akyar and E. Kayimoglu. *Paper presented at 17th Gen. Assembly CITS*, 1983, 32 pp (German). — In two series of investigations (involving 20 and 27 raw sugar samples, respectively), the optimum end-points of preliming and 1st carbonation were determined and the corresponding pH and pOH values compared, while the relationship between temperature and pH for various juices during purification was determined. Results showed that the pH at 20°C corresponding to the optimum preliming and 1st carbonation end-points were not identical, although correlation was found between the pOH values at working temperatures. Titrimetric alkalinity was found to be inapplicable as a criterion for pOH and as a reference value for the optimum end-point. Values of pH difference per 10°C were somewhat higher than values given in the literature. In some cases, pH changes with temperature were found that deviated markedly from the mean at 20-40°C. The pOH values in preliming and 1st carbonation were approximately constant within the working temperature range. The visually established preliming optimum conditions, based on the mean pH of 158 beet samples, were found to be: pH<sub>20</sub> 11.55 ± 0.31 and pOH 2.69 ± 0.25.

**Residence time and juice coloration in an evaporator station.** K. Vukov, I. Körmendy and H. M. Loko. *Paper presented at 17th Gen. Assembly CITS*, 1983, 20 pp (German). — A nickel salt tracer was used to determine residence time in a Zsigmond rapid evaporator operating at relatively high temperatures (116°C in the two 1st effects, 109° in the two 2nd effects, 104.6° in the 3rd effect and 88°C in the 4th effect). Average times were found to be 3.2 min in the 1st effect, 5.3 min in the 2nd effects, 3.2 min in the 3rd effect and 5.4 min in the 4th effect. The thin juice entering the evaporator was of very poor composition and had a purity of only 86.8, although colour formation during evaporation was not excessive because of the short residence times. Sugar hydrolysis was 0.632% of the initial content; from this quantity and the invert sugar content as determined chemically, the amount of invert sugar degraded was found to be 0.392% on sucrose. Colour formation in the individual effects was well correlated with this quantity; some 3-6 minutes was the time required for complete development of high-molecular colorants.

**The accumulation of colouring matter in the sugar house process.** K. Hangyal and L. Paradi. *Paper presented at 17th Gen. Assembly CITS*, 1983, 7 pp (German). — The work described in the preceding abstract was extended to the sugar house. Results showed that colour formation in A-boiling fluctuated markedly, averaging 69%, whereas in B-boiling fluctuation was far smaller (9-29%). Colour formation in low-grade boiling was 2-3 times greater than in B-boiling, although coloration per unit time was about the same. Colour formation was also determined during the 1977/78 campaign in products from thin juice to molasses in all seven Hungarian factories. Results are discussed.

# LABORATORY STUDIES

**Denaturation conversions of albumins during raw juice liming.** L. P. Reva, G. A. Simakhina and V. M. Logvin. *Sakhar. Prom.*, 1983, (5), 40-43 (Russian). — Investigations of the reactions involving denaturation of albumins and their removal during liming showed that the process can be divided into three consecutive stages: (1) reversible denaturation at an alkalinity of 0.005-0.045% CaO, characterized by uncoiling of the polypeptide chains, rupturing of a large number of bonds and liberation of sulphhydryl groups; (2) aggregation of the albumin molecules at an alkalinity of 0.045-0.1% CaO — in a denatured state, these molecules have a greater capacity for interaction with the formation of sufficiently stable bonds and subsequent intensive settling; and (3) partial decomposition at >0.1% CaO of the coagulates — this is associated with the denaturing effect of  $\text{Ca}^{++}$  ions.

**Notes on fibre determination and some recent developments.** J. L. Clayton and R. A. Price. *Proc. Australian Soc. Sugar Cane Tech.*, 1983, 7-12. — A short account is given of the history of fibre determination in prepared cane in Queensland, and details are given of tests conducted in 1980 and 1981 using bags made of three different materials: cambric, cotton duck and polyester duck. Results showed that the cambric bags were lighter and easier to handle, exhibited better retention of insoluble solids and gave better replication than the other bags (the standard deviation being regularly about half that for the other fabrics). The only drawback was the need for a good spin dryer to remove the moisture from the cambric bag samples.

**Enzymic analysis of fermentable sugars in molasses.** D. A. Allan and F. E. Bush. *Proc. Australian Soc. Sugar Cane Tech.*, 1983, 195-198. — A method for determining fermentable sugars in molasses is described. Boehringer Mannheim kits are available for analysis of sucrose/glucose using invertase, and for determination of glucose/fructose using hexokinase, dehydrogenase and phosphoglucose isomerase, but molasses analysis by standard Boehringer Mannheim methods gave poor repeatability and an inflated value of total fermentable sugars concentration. It was therefore suggested to combine the enzymes from the two kits and determine total fermentable sugars as glucose. Recovery from a standard solution averaged 99.6%, while tests for repeatability showed values ranging from 44.1% to 45.8% (average 44.8%) for the same molasses sample. Results for distillery molasses samples averaged 43.8% fermentable sugars compared with 47.2% total sugars found by the Lane & Eynon method.

**Preservation of final bagasse by freezing.** P. J. Dibella and P. C. Ivin. *Proc. Australian Soc. Sugar Cane Tech.*, 1983, 199-204. — While assessment of mill extraction performance is best carried out by immediate analysis of prepared cane and bagasse samples taken from the

same rake of cane, shift samples need to be composited for factory pol accounting, for which the samples need to be preserved. Exploratory tests are described which were conducted at the Sugar Research Institute on the suitability of freezing as a means of bagasse preservation. Samples were maintained at  $-20^{\circ}\text{C}$  for about 24 hours, after which they were defrosted at ambient temperature ( $26-35^{\circ}\text{C}$ ) for about 1½ hours (frozen lumps being broken up by hand every 20 min to accelerate the process). An untreated sub-sample was analysed for Brix and pol by the standard disintegration technique and analysed for sucrose by HPLC, while another sub-sample was left under ambient conditions for 6 hours before analysis. Results of the tests, which were analysed statistically, showed that, although any small average difference between bagasse subjected to either form of treatment and untreated bagasse appeared to be within experimental error, with no significant difference for Brix, pol and sucrose % bagasse determined by HPLC between treated and untreated samples, those samples stored for 6 hours before analysis did show a significant fall in pol and sucrose purities, indicating that deterioration had occurred; no significant deterioration was found in the samples frozen straight away. Tests were also carried out to establish the effect of sample thickness on freezing rate; results showed that the temperature of a sample 35 mm thick was rapidly reduced to  $-7^{\circ}\text{C}$  within 1 hr, while samples 80 mm thick were still unfrozen after 8 hours, and a sample 55 mm thick took 4 hours to reach  $0^{\circ}\text{C}$ . It is thus evident that successful preservation of bagasse samples will depend on the use of very thin bags of bagasse in a freezer of suitable size. Under these conditions, preservation of the samples by freezing is a satisfactory alternative to the use of toxic chemicals as preservatives.

**Measurement of inclusions in sugar crystals using a density gradient column.** S. Y. Guo and E. T. White. *Proc. Australian Soc. Sugar Cane Tech.*, 1983, 219-224. The use of a density gradient column to estimate the amount of inclusion in sugar crystals is described. The uniform increase in the density of the contents of the column from top to bottom is brought about by mixing two liquids in varying proportions as the column is filled. A sugar crystal added to the column will eventually come to rest where its density equals that of the suspending liquid. Marker floats of known densities are added to the column to indicate the density of the fluid at various levels. Thus, the inclusion content  $v$  is given by  $(\text{sucrose density} - \text{crystal density})/(\text{sucrose density} - \text{liquor density})$ , where  $v$  = ratio of volume of inclusions to the total crystal volume. Carbon tetrachloride was chosen as suspending fluid in a 0.8 m glass column of 40 mm i.d. The system was maintained at a constant  $25^{\circ}\text{C}$ . Results of the experiments with laboratory-grown crystals of about 1 mm showed that the technique provided a useful means of fractionating samples according to inclusion content. Drawbacks mentioned include the smallness of the quantity of sample treated and the length of time per test (several hours) as well as the time needed to set up the column and calibrate the equipment. Care must be taken with the procedure in view of the smallness of the changes investigated, while good temperature control is essential.

**Continuous hydrolysis of concentrated sucrose solutions by immobilized invertase.** P. Monsan. *Paper presented at Symp. Int. sur l'Utilisation des Enzymes en Technologie Alimentaire* (France), 1982; through *S.I.A.*, 1983, 45, Abs. 83-356. — Studies on the effect of sucrose concentration on the initial rate of inversion by immobilized

### Laboratory studies

invertase showed that inhibition by the substrate (sucrose) was related to the structure of the sucrose molecule in increasingly concentrated solutions. Invertase was immobilized by covalent bonding to chemically modified maize cobs. In a fixed-bed reactor with 2M sucrose solution at 40°C, the half-life of the material was about one year. Tests were carried out in reactors of 0.1, 1.0 and 17.6 litre volumes. A pilot-scale reactor currently in operation in a cane sugar refinery is hydrolysing concentrated sucrose solutions. With a solution of initial concentration 69% by weight, its throughput at 52°C is 9.1 kg sucrose hydrolysed per hr, the degree of hydrolysis being 72%.

**Production-scale chromatography for the continuous separation of fructose from carbohydrate mixtures.** P. E. Barker, J. C. Gould and G. A. Irlam. *Inst. Chem. Engrs. Symp. Series*, 1982, **73**, D23-D38; through *S.I.A.*, 1983, **45**, Abs. 83-358. — Following laboratory experiments, a large-scale counter-current semi-continuous chromatographic refiner operating on the moving-port principle was constructed. A mathematical model of its operation was developed. Tests on the separation of fructose from mixed solutions are reported. With feed solutions containing 20, 40 or 60% solids by weight, comprising equal weights of fructose and glucose, products of fructose purity 99.9, 88 and 77, respectively, were obtained, but the product concentrations were only 0.64-1.79% w/v. With feed solutions containing 70% solids by weight, of which 69% was fructose, 22% dextran and 9% glucose (simulating effluent from dextran production), the purity of the fructose product was 99.9% at all flow rates tested, and the product concentrations were 3.2-5.1% w/v. Ways in which more concentrated products could be obtained are discussed. Throughput was up to 2.5 kg carbohydrates per hour, much higher than when the equipment was used in the batch mode.

**Accurate temperature-controlled polarimeter.** A. L. Cummings and R. J. Hocken. *Precision Eng.*, 1982, **4**, (1), 33-38; through *S.I.A.*, 1983, **45**, Abs. 83-420. At the US National Bureau of Standards, a photo-electric polarimeter was constructed for the purposes of calibrating polarimetric standards and of re-determining the value for the specific rotation of sucrose in solution. The polarimeter has full circle rotation capability with sensitivity of 0.6 arc-sec (3  $\mu$ rad) and in specific measurements an estimated accuracy of  $\pm 2$  arc-sec. The polarimeter uses both arc lamp and laser sources. Sample temperatures can be controlled to within  $\pm 0.5$  mK per day at, or around, 20°C. Six liquid, solid or gaseous samples may be simultaneously housed, and measurements on all six may be completed in a few min. Design details are explained, with diagrams of the complete system and the sample enclosure and cell, and circuit diagrams.

**Isolation and fractionation of sugar colorants by rapid chromatographic methods.** H. T. Cheng and C. R. Wang. *Rpt. Taiwan Sugar Research Inst.*, 1982, (98), 47-57. Details are given of high-performance gel permeation chromatography (HPGPC) and reversed-phase high-performance liquid chromatography (RPHPLC) methods for fractionation of colorants present in juices, syrups and sugars from carbonation and sulphitation factories. The colorants were isolated by adsorption on a Sep-Pak C<sub>18</sub> cartridge of Waters Associates. HPGPC was carried out on a column of  $\mu$ Bondagel E-125, which is an ether-

bonded silica gel column having a nominal molecular weight separation range of 2000-50,000; an ultraviolet detector was used at 436 nm. RPHPLC analysis of the colour fractions from fine syrup was carried out with 40% formamide in 0.3% acetic acid as solvent; the U.V. detector used a filter of 340 nm wavelength. Concentrated, sugar-free colorant solutions were obtained with 70.9  $\pm$  5.5% recovery. The methods were used to determine the effect of process changes on colour contents. Significant differences were found between the colorants from the carbonation and sulphitation factories.

**Thin-layer chromatographic determination of traces of chloridazon and lenacil in molasses.** E. Wislowska and B. Kostowska. *Chem. Anal. (Warsaw)*, 1981, **26**, (4), 727-730; through *Anal. Abs.*, 1983, **44**, 6F23. — The sample (50 g) was purified by extraction with organic solvents, then by chromatography on a column of silica gel (230-400 mesh); the column was washed with hexane, the lenacil (i) was eluted with hexane-ethyl ether (1:1) and chloridazon (ii) with methanol-ether (1:1). The combined eluates were evaporated at 40°C, the residue was dissolved in hexane, and this solution was used for thin-layer chromatography on silica gel G, together with standards, with benzene-acetone (1:1) as mobile phase. (i) and (ii), of R<sub>F</sub> 0.86 and 0.77, respectively, were located by exposure of the chromatogram to Cl vapour followed by spraying with KI-starch solution, and were evaluated visually. Down to 0.1  $\mu$ g of (i) or (ii) per spot could be determined by this method. The recoveries of (i) and (ii) were 90-95% and 80-85%, respectively. Detailed procedures for sample purification and TLC are given.

**Characterization of the main secondary components of the liquid sugars from cane molasses.** G. Palla. *J. Agric. Food Chem.*, 1983, **31**, 545-548. — Investigations of non-sugars in syrup obtained by cane molasses demineralization have been continued<sup>1</sup>; silica gel chromatographic fractions recovered from a wide range of liquid sugars have been examined, and the main constituents of the non-sugars found to be phenylpropanetriol glucosides (40-60%), phenyl glucosides (10-20%) and smaller quantities of other phenolic derivatives, while a fraction of high molecular weight was also detected. The glucosides most probably come from the lignin fraction of cane stalks. The phenylpropanetriols contributed to the colour of the liquid sugars examined, probably as their oxidation or dehydration products, generating a bright yellow colour (as did the phenyl glucosides) when exposed to air on silica plates. Syrups from beet molasses never contained more than 0.04% phenolics (on total sugar); the difference between beet and cane molasses is attributed to the absence of juice treatment with phosphoric acid and SO<sub>2</sub> in beet sugar manufacture, since acid media and high temperature accelerate lignin depolymerization with formation of phenolic material. Knowledge of the chemical structure of the colorants mentioned suggests the possibility of using anionic macroporous resins for syrup decolorization: until now, satisfactory adsorption has been obtained experimentally only by passing the diluted and well-demineralized syrup through resin that has been regenerated with ammonia, and work is in progress to improve the decolorizing process.

**Immobilization of beta-fructosidase to give invert syrup.** M. O. Mandel and V. A. Krosing. *Tallinna Polütehnilise Inst. Toimetised*, 1980, (499), 75-82; through *FSTA*, 1982, **14**, (12), 12L868. — Yeast beta-fructosidase was

<sup>1</sup> See Palla: *I.S.J.*, 1983, **85**, 216.

covalently bonded onto activated silochrome pretreated with gamma-aminopropyl triethoxysilane and *n*-benzoquinone. Two-stage application of the enzyme solution gave an immobilized enzyme preparation with activity up to 7000 units/g, total yield 45%. Treatment of the immobilized enzyme preparation with glutaraldehyde did not decrease enzyme activity, and thermal stability increased by up to 3 times. Maximum productivity of 1 kg immobilized enzyme preparation at 50°C and at a 100% conversion rate of 70% sucrose solution was 10-11 kg invert sugar (dry solids) per hr.

**Effects of oligosaccharides and polysaccharides on sucrose crystallization.** F. W. Parrish and M. A. Clarke. *Paper presented at 17th Gen. Assembly CITS, 1983, 17 pp.* — A review, with 44 references to the literature, is presented of work concerning the mechanisms of sucrose crystal growth, occlusion of non-sucrose compounds and the effects of oligosaccharides and polysaccharides on crystal habit. Crystallization in a laboratory vacuum pan, using an aqueous sugar solution seeded with powdered sucrose followed by centrifuging and washing of the isolated crystals, was compared with crystallization in the presence of added polysaccharides or their enzymolysis products. The products were examined by polarimetry, and photomicrographs obtained with an ISI scanning electron microscope, as well as image analyses. Results showed that sucrose yields were not significantly different, regardless of whether additives were present or not. Occlusion of starch and dextran was demonstrated by reaction with iodine and alkaline copper sulphate, respectively. While photomicrographs showed no significant differences with respect to axial ratios or conglomeration between crystals obtained from pure sucrose and from sucrose to which 1% starch had been added, crystals obtained from sucrose solutions to which 1% maltose had been added showed a high degree of conglomeration, although again there was no significant difference in the axial ratios. The crystals obtained in the presence of 1% starch showed, when examined with a polarizing microscope, adhering granules of ungelatinized starch on all faces. Determination of the temperature range for loss of bi-refringence of the starch granules gave values of 58-66°C for cane juice and >105°C for 70°Bx sucrose solution. The pan temperature was kept at 63°C, which is well below that required for starch gelatinization. Panose, isomaltose, maltotriose and indigenous sugar polysaccharide had no effect on yield or crystal axial ratios. The axial ratios of crystals obtained in the presence of a combination of starch and dextrans were similar to those obtained in the presence of dextrans alone.

**Growth kinetics of sucrose single crystals and twins.** G. Mantovani, G. Vaccari, G. Sgualdino, C. A. Accorsi, D. Aquilano, M. Franchini-Angela and M. Rubbo. *Paper presented at 17th Gen. Assembly CITS, 1983, 12 pp.* — For purposes of increasing knowledge on crystal growth, the following are necessary: (1) identification of the mode of growth of single crystals, (2) calculation of both surface tension  $\gamma_{hkl}$  and attachment energy  $E_{att}^{hkl}$  for each form of crystal, (3) plotting of growth isotherms  $R_{hkl}$  vs. supersaturation  $\sigma$  for each face so as to identify the growth mechanism, and (4) assessment of the effects of various solvents on crystallization kinetics and study of the specific adsorption of impurities on different forms of the same crystal growing in a given solvent. The authors summarize their work on each of these subjects. They have applied the Hartman & Perdok theory to crystal growth morphology, starting

from the structural data of Brown & Levy<sup>1</sup>, and projected a crystal structure divided into periodic bond chains involving a sequence of hydrogen bonds among the sucrose molecules. (A representative sketch of the structure is appended.) Experiments were conducted on growth of twin crystals in pure solutions and solutions to which raffinose had been added. A method is described which permits the growth rates of all forms of crystals to be determined simultaneously. While measurement of the solution flow rate relative to a growing face is limited by the fact that an already well-grown crystal having flat faces imparts a hydrodynamic pattern of behaviour to the solution, this drawback can be eliminated by using a spherical crystal; this approach has been applied to the growth of an initial seed in steady-state conditions and in stirred solutions, and successive stages of growth of a sucrose sphere in a steady state are illustrated.

**Mechanism of growth of sucrose crystals: a reassessment.** A. VanHook. *Paper presented at 17th Gen. Assembly CITS, 1983, 18 pp.* — While current understanding on crystal growth is that of a 1st order reaction under most ordinary conditions, with mass transport as the controlling factor at temperatures of 60°C and above, but of diminishing importance as the temperature falls and with surface integration then becoming the rate-determining factor, it is necessary to have a proper understanding of the form in which data should be presented. The influence of syrup concentration on growth rate should be given in terms of activity. Use of this hydrodynamic approach is illustrated by a comparison of supersaturations of a given sugar solutions as expressed in molal or sugar:water ratios, in mole fraction terms, in molar or sugar:volume ratios or in % by weight. The first three forms are almost linearly related at 30°C. Most growth rate data for sucrose are very close to 1st order kinetics for all ordinary conditions but undergo changes towards slower and higher order rates at low supersaturations and at certain temperatures. The inability of the author to find any higher order kinetics at low temperatures as theory would indicate, and Maurandi's emphasis<sup>2</sup> on the similar functioning of impurity and low temperatures on crystallization kinetics, show that more work needs to be done at low concentrations and temperatures. The various factors involved, viz. temperature, impurity (particularly raffinose), solubility and habit, as well as differences in crystal growth mechanisms, are briefly discussed with reference to the literature.

**The cation balance in sugar beet molasses ion exclusion.** H. Zaorska and K. Lisik. *Paper presented at 17th Gen. Assembly CITS, 1983, 21 pp (German).* — A balance of Na<sup>+</sup> and K<sup>+</sup> ions was calculated after 27 cycles of ion exclusion of delimed molasses using Lewatit TSW 40 cation exchange resin in a 4-column system. The sugar fraction obtained had a dry solids content of almost 14% while that of the feed molasses was 50.5%. The cation content of the sugar fraction was only 2.5%. The Na<sup>+</sup> in the active groups of the resin was found to displace the K<sup>+</sup>; in the 1st column the Na<sup>+</sup> and K<sup>+</sup> contents were about 20% and 79%, respectively, whereas in the 4th column the figures were almost the reverse, viz. 78% and 21%. Pre-treatment for Ca removal was necessary so as to prevent blockage of the active groups in the resin. Regeneration of the resin was unnecessary, thus avoiding the occurrence of heavily polluted effluent.

<sup>1</sup> *Acta Cryst.*, 1973, (B-29), 790.

<sup>2</sup> *Sucr. Belge*, 1982, 101, 207.

# BY-PRODUCTS

**Anaerobic fluidized bed experimentation with a molasses waste water.** B. Frostell. *Process Biochem.*, 1982, 17, (6), 37-40; through *S.I.A.*, 1983, 45, Abs. 83-430. An anaerobic fluidized bed system for waste water treatment was investigated on a laboratory scale for 165 days. Biomass was retained by attachment to a sand carrier and by external recycling of solids. Waste liquid from baker's yeast production on beet molasses was used as substrate. It had a COD of  $9.1 \text{ kg.m}^{-3}$ . After changes in the design of the fluidized bed system, stable operation could be obtained at high organic loads of 20-25 kg COD per  $\text{m}^3/\text{day}$  at  $30^\circ\text{C}$ . With an average load of  $22.2 \text{ kg.m}^{-3}$  COD per day during 17 days, a gas containing 56% methane was obtained, with 43% COD conversion to methane. The system had excellent stability under varying loads. The main drawbacks were the length of time required for start-up and the limited attachment of biomass which could be obtained.

**Fungal invertase as an aid for fermentation of cane molasses into ethanol.** Y. K. Park and H. H. Sato. *Appl. and Environmental Microbiology*, 1982, 44, (4), 988-989; through *S.I.A.*, 1983, 45, Abs. 83-451. — Cane molasses at 15.5° Bx was fermented to ethanol by *Saccharomyces cerevisiae* in the presence or absence of fungal invertase. At  $30^\circ\text{C}$  and pH 5.0, the invertase had no effect on ethanol yield, but at pH 3.5 it increased the yield. At  $40^\circ\text{C}$ , yields were much lower, but the decrease was partly counteracted by inclusion of the invertase.

**Utilization of bagasse by bacteria.** C. Sarkar and K. A. Prabhu. *J. Ferment. Technol.* (Osaka), 1982, 60, (4), 297-303; through *S.I.A.*, 1983, 45, Abs. 83-470. — Two bacterial strains were isolated from rumen liquid and liquid waste (bio-liquid) from a biogas plant using bagasse. The strains were capable of growing on alkali-treated bagasse, and were identified as *Cellulomonas* sp. In a basal salt medium containing 1% alkali-treated bagasse at pH 7-7.5, the cultures decomposed 50-55% of the bagasse in a 5-day shaken culture. A mixed culture of *Cellulomonas* sp. B1 and *Trichoderma* sp. decomposed 95% of the bagasse in 24 hours. The protein contents of the *Cellulomonas* sp. were 52 and 57%.

**Improvement of xylose production by acid hydrolysis of bagasse pith with low liquor ratio.** W. F. Yee, L. H. Wang, M. C. Hsie and S. L. Sang. *Rpt. Taiwan Sugar Research Inst.*, 1982, (98), 59-70 (Chinese). — Statistical analysis of xylose production from bagasse pith by acid hydrolysis showed that the most significant factors affecting yield were the quantity and concentration of  $\text{H}_2\text{SO}_4$  used, temperature, and time of pre- and post-hydrolysis. There was also remarkable interaction between  $\text{H}_2\text{SO}_4$  quantity and time of prehydrolysis. Under optimum conditions of 20% consumption of  $\text{H}_2\text{SO}_4$  in the pith mixture at an acid concentration of 25%, a temperature of  $75^\circ\text{C}$ , and 90 minutes and 150 minutes for pre- and post-hydrolysis, respectively,

yield was maximum at  $90 \pm 8.7\%$ . A preliminary design for continuous xylose manufacture in which the mixture was treated in a rotary kiln, followed by extraction of the xylose by spraying water over a bed of the mixture on an endless belt and dewatering the pith in a screw press proved feasible in a simulated experiment using counter-current extraction. For recovery of about one-third of the sulphuric acid in the hydrolysate, the latter was recycled to the extraction stage for adsorption of the acid by the pith.

**Application of good and poor pressed pulp silage in ruminant feeding.** R. Leitgeb, H. J. Müller and M. Haluschan. *Zuckerind.*, 1983, 108, 444-446 (German). Good beet pulp silage is crumbly, uniform and stable, while poor silage is very acid, smells strongly, has no solid structure but is wet and sticky. Ruminant feeding trials were conducted with good and poor pressed pulp silage as well as dried pulp. During the 84-day period, daily liveweight gains were 1393, 1373 and 1311 g for the dried pulp, good silage and poor silage, respectively, while the respective energy consumption per kg of liveweight gain was 2874, 2923 and 2887 starch units. However, no significant differences were found between the factors investigated, indicating that the feed value of pulp silage is not greatly affected by the fermentation processes.

**Evaluation system for pressed pulp silage quality.** M. Haluschan, R. Leitgeb and H. J. Müller. *Zuckerind.*, 1983, 108, 447-452 (German). — A scheme for evaluation of beet pulp silage is described which embodies five categories: very good, good, satisfactory, fair and poor. It covers factory handling properties as well as feed value for ruminants, and involves evaluation on the basis of colour, smell, structure and overall impression. Total acid content and acid composition are also included. While pulp in the first three categories may be fed to all animals, that in the last two categories should be fed in smaller amounts only to fattening cattle.

**Production of fructose from beet molasses.** P. S. Yip, R. F. Olson and S. E. Bichsel. *Zuckerind.*, 1983, 108, 467-472. — Pilot plant experiments are reported in which beet molasses was hydrolysed with invertase, and slaked lime then added at temperatures between 0 and  $5^\circ\text{C}$  in order to precipitate calcium fructosate during  $2 \pm 1$  hr residence in a 5-section continuous stirred tank. The fructosate was filtered off, neutralized with phosphoric acid and re-filtered. A raw fructose solution containing more than 90% fructose on total carbohydrate and having a purity  $>90\%$  was obtained. Purification with ion-exchange resin gives a quality comparable to that of 90% HFCS from maize. The residual molasses can be neutralized with phosphoric acid and sold as glucose molasses after concentration. In both stages of neutralization,  $\text{CaHPO}_4 \cdot 2\text{H}_2\text{O}$  is formed and is of value as a fertilizer.

**Quality of press mud cake (PMC) from various sugar factories in Maharashtra.** S. P. Patil, S. P. Kale and D. G. Hapase. *Indian Sugar*, 1983, 32, 779-783. — Filter cake samples from 21 sugar factories were analysed for nutrient components (organic carbon, nitrogen,  $\text{P}_2\text{O}_5$ ,  $\text{K}_2\text{O}$  and free lime) and their pH and electrical conductivity measured. Wide differences were found between factories but not between samples taken at monthly intervals at the same factory. The value of the filter cake as nutrient is particularly significant in view of the current need to import fertilizer.



# BREVITIES

**St. Kitts sugar production, 1983<sup>1</sup>.** — St. Kitts-Nevis produced 27,761 long tons of sugar (28,207 tonnes) in 1983 from a little more than 276,000 tonnes of cane. Of the total, 13,000 tonnes has been exported to the EEC and the United States.

**Greek sugar industry development<sup>2</sup>.** — In spite of a beet area reduced from 41,400 to 39,000 hectares, Hellenic Sugar Industry A/S. expects a greater sugar outturn in 1983/84 than in 1982/83, thanks to better weather. The beet crop is estimated at 2.5 million tonnes, against 2,406,000 in the last campaign, and sugar production is estimated at 300,000 tonnes, against 297,000 tonnes. Domestic consumption will be covered and a small surplus available for export; already some Balkan countries have expressed interest. A decision is awaited as to permission for building three by-products plants for the manufacture of 10,000 tonnes/year of alcohol from molasses, 6000 tonnes/year of citric acid, also from molasses, and 2000 tonnes/year of dried yeast.

**US Sugar Corporation share sale<sup>3</sup>.** — US Sugar Corporation, the Florida company which claims to be the largest raw sugar producer in the country, is planning to arrange a \$40 million co-ownership scheme backed by its majority shareholders. The proposal provides for the acquisition of about 70% of the company by an employees' trust fund, which would be funded by a loan provided by the company itself. Commenting on the scheme, a company spokesman said that public investors in the company were suffering from the lack of marketability of the stock. The buy-out offer, at \$68 a share, compares with a trading range over the past year of \$26 to \$42 and a book value of \$48. Independent advisers had calculated that the offer price gave a firm indication of the company's worth. The acquisition will also allow the Mott family, who own some 70% of the equity, to liquidate some of their holdings while retaining control of the company. Following the retirement of some of the shares acquired, the plan should leave the Mott family with a little over 50% of the common stock while the rest would eventually end up in the hands of the 2400 employees. US Sugar Corporation made net profits of \$19.3 million in the year to September 1982, at \$3.94 a share.

**Taiwan sugar production decline<sup>4</sup>.** — Taiwan's sugar production for the 1983/84 crop will be less than 600,000 tonnes, compared with 621,000 tonnes in 1982/83, according to a spokesman of Taiwan Sugar Corporation. Taiwan's production target is about 700,000 tonnes, down from the 800,000 tonnes target set for 1982/83. The shortfall from the 1982/83 target was due mainly to farmers' reactions to low world sugar prices. Sugar exports fell sharply during the first six months of 1983 to 123,000 tonnes sold to Japan and South Korea; this compares with 314,700 tonnes sold in the same period of 1982.

**Canadian investigation of US refined sugar sales<sup>5</sup>.** — The Canadian Sugar Institute has formally complained and requested an investigation by the Canadian revenue authorities of alleged dumping of refined sugar by the United States. The Institute says that a new US sugar program has led to major sugar imports, with a substantial price erosion and a loss of sales by Canadian refiners. It claims that US exporters have a substantial cost advantage over Canadian refiners through a system of duty and import fee drawbacks up to 8 or 9 cents/lb.

**Australian sugar season delay<sup>6</sup>.** — The after-effects of the long drought early this year and the widespread late rains along the whole of the Australian east coast sugar districts have resulted in one of the latest starts to harvesting and crushing for many years. Broadwater Mill was the first to start, on June 15, but by July 31 five of the 33 sugar factories had not started crushing and two of these, Isis and Maryborough, were not expected to start before early September. The rains improved prospects over what had been expected during the drought, but c.c.s. figures are lower than in the 1982 season.

## Indonesia sugar imports, 1982<sup>7</sup>

|              | 1982              | 1981           | 1980           |
|--------------|-------------------|----------------|----------------|
|              | tonnes, raw value |                |                |
| Australia    | 0                 | 0              | 121            |
| Brazil       | 0                 | 224,228        | 0              |
| China        | 0                 | 0              | 50,004         |
| Cuba         | 14,236            | 13,646         | 86,132         |
| EEC          | 88                | 307            | 11             |
| India        | 301,161           | 90,756         | 8,050          |
| Japan        | 17                | 13,050         | 12,600         |
| Korea, South | 96,682            | 217,418        | 94,545         |
| Malawi       | 0                 | 10,617         | 0              |
| Malaysia     | 0                 | 0              | 150            |
| New Zealand  | 1                 | 0              | 0              |
| Philippines  | 86,321            | 217,583        | 123,332        |
| Singapore    | 0                 | 0              | 26             |
| Switzerland  | 60                | 41             | 4              |
| Taiwan       | 0                 | 0              | 22,614         |
| Thailand     | 68,959            | 20,283         | 0              |
| USA          | 98                | 25,689         | 27             |
|              | <b>567,627</b>    | <b>833,625</b> | <b>397,616</b> |

**Thailand crop results, 1982/83<sup>8</sup>.** — Sugar cane output in Thailand in the 1982/83 crop (November/June) was 23,916,364 tonnes, a decrease of 21% from the previous year when the crop reached 30,263,797 tonnes. Sugar production in 1982/83 was 2,215,018 tonnes, comprising 227,703 tonnes of refined white sugar, 597,630 tonnes of white sugar, 3801 tonnes of brown sugar and 1,385,947 tonnes of raw sugar. Production in 1983/84 is estimated at 1.7 million tonnes, raw value, more than 26% below the previous season's output.

**EEC quota shortfall reallocation disputa.** — By the end of October no agreement had been reached concerning the reallocation of 12,000 tonnes of ACP quotas for which shortfalls had been declared. The UK wanted the tonnage to be shared by India, Zimbabwe and the Ivory Coast but the French wanted the full quantity to be given to the Ivory Coast.

**Bolivia sugar situation<sup>9</sup>.** — In 1983/84 Bolivia's sugar production is expected to be around 222,000 tonnes, of which 195,000 tonnes will be reserved for domestic consumption, 21,000 tonnes for export to the US and 4500 tonnes for ISA special stocks, leaving only about 1500 tonnes for export to other destinations. Production trend would appear to be downwards; it declined from 259,818 tonnes in 1981/82 to 228,345 tonnes in 1982/83 and is expected to decline further in 1983/84. Recently introduced legislation is unlikely to help reverse this situation. A recent government decree allocated production quotas which apportioned a major part to state-owned sugar factories. In addition, it imposed a state monopoly of commercialization, a ruling which created strong opposition from the producers. Meanwhile it is almost impossible to buy sugar on the local market because the retail price has been increased by 157% based on a study according to which labour cost in the cane fields is estimated at 800 pesos per tonne: one quintal of sugar bagged at the mill now costs 6247 pesos in La Paz and 6901 pesos in Santa Cruz, against the previous wholesale price of 3420 pesos.

**New Cuban distillery<sup>10</sup>.** — The first distillery to be built in Cuba of local design and construction was inaugurated in April in the Arquimedes Colina agro-industrial complex in Bayamo, Granma Province. It cost 3 million pesos and was completed in three months.

**Bolivia sugar factory rehabilitation project<sup>11</sup>.** — The Corporación de Fomento del Departamento de La Paz is seeking finance, estimated at around \$40 million, to revive the sugar factory and distillery project of San Buenaventura.

<sup>1</sup> F. O. Licht, *International Sugar Rpt.*, 1983, 115, 487.

<sup>2</sup> *Zuckerindustrie*, 1983, 108, 903.

<sup>3</sup> F. O. Licht, *International Sugar Rpt.*, 1983, 115, 504-505.

<sup>4</sup> *Reuter Sugar Newsletter*, August 1, 1983.

<sup>5</sup> F. O. Licht, *International Sugar Rpt.*, 1983, 115, 505.

<sup>6</sup> *Australian Sugar J.*, 1983, 75, 252.

<sup>7</sup> *I.S.O. Stat. Bull.*, 1983, 42, (8), x-xi.

<sup>8</sup> F. O. Licht, *International Sugar Rpt.*, 1983, 115, 559-560.

<sup>9</sup> *World Sugar J.*, 1983, 6, (3), 34.

<sup>10</sup> *Cuba Economic News*, 1983, 19, (134), 19-20.

<sup>11</sup> *Amerop Newsletter*, 1983, (118), 15.



## Brevities

**Japan beet sugar crop reduction<sup>1</sup>.** — Bad weather and temperatures below average since late May in Hokkaido are expected to reduce the 1983/84 sugar beet crop to about 3.5 million tonnes from 4.1 million tonnes in 1982/83. Beet sugar production is estimated at about 490,000 tonnes, white value, but will depend on weather conditions.

**New Yugoslavian sugar factory<sup>2</sup>.** — The Belgrade agro-industrial combine has put into operation one of the largest sugar factories in Yugoslavia. The new complex, which cost 5500 million dinars, is designed to produce 70,000 tonnes of sugar annually. There are now 21 sugar factories in Yugoslavia with a total production capacity of 800,000 tonnes per year. The government planned to increase the beet area by 33% in 1983 to 186,000 hectares, in order to reduce sugar imports; however, actual sowings were only 140,000 hectares.

**Ecuador sugar imports to rise<sup>3</sup>.** — Ecuador is expected to import 197,100 tonnes of sugar in 1983/84 (June/May), up from 77,200 tonnes in 1982/83, according to the US Agricultural Attaché in Quito. Sugar output in 1983/84 is forecast at 150,000 tonnes, from a cane crop of 2,030,000 tonnes. This is down from 253,700 tonnes of sugar produced from a 2,470,000 tonnes cane crop in 1982/83. Continuous rains in Ecuador's coastal region have resulted in floods and a 2-3 months delay for the 1983/84 sugar cane harvest, so that crushing was expected to start in late August or early September against the normal June start.

**Spanish sugar commission<sup>4</sup>.** — The Spanish government has taken steps to reorganize the sugar sector. Recently, the Ministry of Agriculture has established the Comisión Nacional Azucarera (CNA) which now exercises the control existing within the industry. The new commission is formed by ten directors, of whom five are chosen from among manufacturing companies and five represent the growers. The choice of the first five caused some difficulty since the Asociación de Industrias de Azúcar claimed all five seats for themselves, excluding the ACOR concern (Azucarera Cooperativa Onésimo Redondo) which has a large plant in Valladolid and plans to expand. In the end, one of the seats went to ACOR. Creation of the Commission will simplify matters for the sugar industry but will undoubtedly bring opposition too. It now takes over duties including decisions on production quotas, the most important matter involved.

**Pakistan sugar production capacity increase<sup>5</sup>.** — In the next five years, ten cane sugar factories are to be built, six in Sind Province, three in the Punjab and one in the North West Frontier Province.

**New Indonesian sugar factory<sup>6</sup>.** — A sugar factory in South Kalimantan, to be built with the assistance of a World Bank loan, will produce around 50,000 tonnes of sugar a year when it is completed in 1988, according to industry sources. The World Bank will lend Indonesia \$79 million to build the factory, and the project will help cut imports by 7% when it is operating. Indonesia's imports reached a record 848,000 tonnes in 1981/82 but are likely to have fallen sharply in 1982/83 because of a substantial increase in sugar output. Production is expected to be 1.8 million tonnes, raw value, in 1982/83 against 1.4 million tonnes, raw value, in 1981/82. Production is expected to rise to 2,500,000 tonnes by 1985, which should be sufficient to cover domestic requirements, estimated at 2,400,000 tonnes.

**Locust damage in the Philippines<sup>7</sup>.** — Locusts have damaged more than 1000 hectares of sugar cane plantations in Negros Occidental province, and the situation could deteriorate unless the government provided help, according to planters.

**Florida sugar production, 1982<sup>8</sup>.** — The six sugar companies of Florida crushed a total of 12,266,410 short tons of cane in 1982 to produce 1,260,577 tons of 96° raw sugar. This compares with 953,639 tons of sugar produced from 10,146,000 tons of cane in 1981.

**Sweden sugar importation<sup>9</sup>.** — In 1982/83 Sweden's domestic production was sufficient to cover requirements. However, during the current season, a substantial decrease of sugar production is expected, owing to unfavourable weather conditions, and Sweden will have to cover some 20% of its requirements by imports which, it has been announced, will be from developing countries.

**New technique for juice heater tube extraction<sup>10</sup>.** — Hydraulic equipment first used on North Sea gas rigs has been modified by the Production Engineering Research Association and adopted at Spalding sugar factory in the UK for extraction of heater tubes. With manual extraction, tube removal averaged 14 per hour but in difficult conditions could fall to 7 per hour. With the PERA equipment it is possible to remove a tube per minute under ideal conditions but experience at Spalding shows that an average of 22 per hour can be maintained.

**Indian sugar production plans, 1983/84<sup>11</sup>.** — India plans to produce between 7.5 and 8 million tonnes of white sugar (8.15-8.7 million tonnes, raw value) in the 1983/84 crop year which started on October 1. Production in 1982/83 is estimated at 8.24 million tonnes of white sugar (8.95 million tonnes, raw value).

**New Uganda project<sup>12</sup>.** — The Arab Bank for Economic Development in Africa is to provide Uganda with an \$8 million loan to finance the construction of a sugar complex, according to Bank officials. Under the agreement, the loan will be repaid over 16 years, including a 4-year grace period, at 8% interest.

**Brazil sugar company difficulties<sup>13</sup>.** — The Ometto Group, Brazil's largest private sugar producer, has sought court protection for six of its ten companies because they are unable to pay their debts. The lawyer for the group said that the sharp fall in the value of the Brazilian cruzeiro in 1983 meant that the companies could not earn enough to maintain payments to creditors. He said that the legal move, known as a "concordata", would enable the six to pay off their creditors over two years and keep operating. Press reports said that foreign debts of the group rose from US \$2.7 million in 1981 to \$31.5 million at the end of 1982. The lawyer said that the companies' difficulties had been aggravated by delays in remittances from Brazil's Sugar and Alcohol Institute which buys all the country's output.

**New bulk sugar terminal in Brisbane<sup>14</sup>.** — The Queensland Sugar Board is to build and operate a \$A 36.7 million bulk raw sugar terminal in Brisbane which will serve south-east Queensland and northern New South Wales. It is intended to be in full operation by June 1985 with a first year export of 180,000 tonnes. This is to be doubled by the year 2000. The terminal, with a storage capacity of 60,000 tonnes, will help cater for increased sugar production.

**China 1983/84 sugar supply estimate<sup>15</sup>.** — The US agricultural Counsellor in Beijing has estimated China's 1983/84 sugar production at 3,880,000 tonnes, white value, which is only slightly up from that of 1982/83. The season's production of sugar beet is expected to increase to 8,200,000 tonnes from 6,700,000 tonnes in 1982/83. At the same time, sugar cane output is expected to be down from the 1982/83 record of 37 million tonnes to 34 million tonnes, because of poor weather. China's sugar imports in calendar year 1983 are estimated at 2,200,000 tonnes; China has taken advantage of low world sugar prices to meet rising domestic demand.

## PERSONAL NOTES

We regret to report the death, earlier this year, of **Norman Adams**, at the age of 78. Mr. Adams joined the United Sugar Company at Bury St. Edmunds in 1927 after graduation and progressed from trainee to works manager of the factory which became part of the British Sugar Corporation Ltd. in 1936. In 1941 he moved to Spalding as general manager and in 1946 joined the headquarters staff in Peterborough as assistant to the Chief Technical Officer and as Technical Supervisor in 1948. In 1968 he was appointed to the Board as Technical Director before retiring in 1970.

<sup>1</sup> *World Sugar J.*, 1983, 6, (2), 37.

<sup>2</sup> F. O. Licht, *International Sugar Rpt.*, 1983, 115, 519.

<sup>3</sup> *Reuter Sugar Newsletter*, August 18, 1983; *Public Ledger*, August 23, 1983.

<sup>4</sup> F. O. Licht, *International Sugar Rpt.*, 1983, 115, 518.

<sup>5</sup> *Zuckerindustrie*, 1983, 108, 905.

<sup>6</sup> F. O. Licht, *International Sugar Rpt.*, 1983, 115, 467-468.

<sup>7</sup> *Public Ledger's Commodity Week*, September 24, 1983.

<sup>8</sup> *Sugar J.*, 1983, 46, (3), 23.

<sup>9</sup> F. O. Licht, *International Sugar Rpt.*, 1983, 115, 518.

<sup>10</sup> *British Sugar News*, 1983, (55), 3.

<sup>11</sup> F. O. Licht, *International Sugar Rpt.*, 1983, 115, 524.

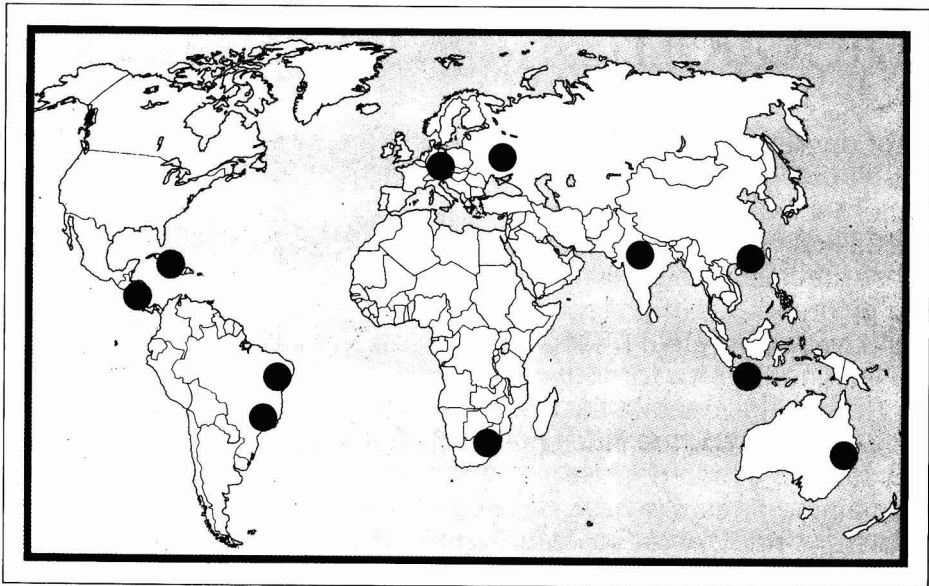
<sup>12</sup> *Public Ledger*, September 6, 1983.

<sup>13</sup> F. O. Licht, *International Sugar Rpt.*, 1983, 115, 542.

<sup>14</sup> *Queensland Newsletter*, October, 1983.

<sup>15</sup> F. O. Licht, *International Sugar Rpt.*, 1983, 115, 544.

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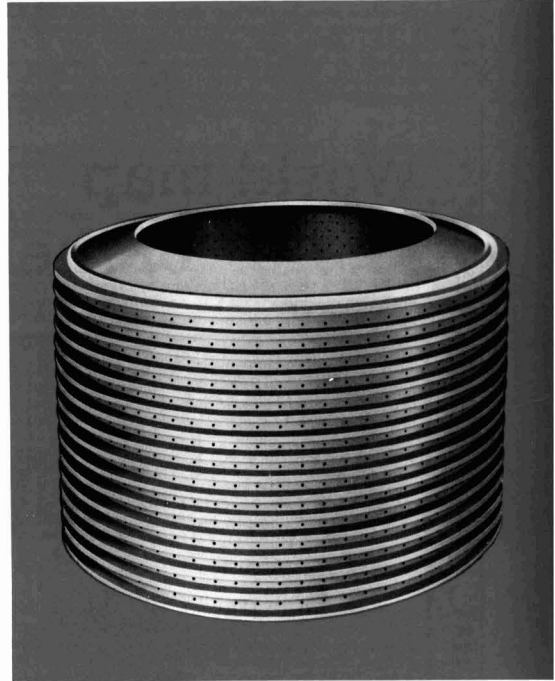
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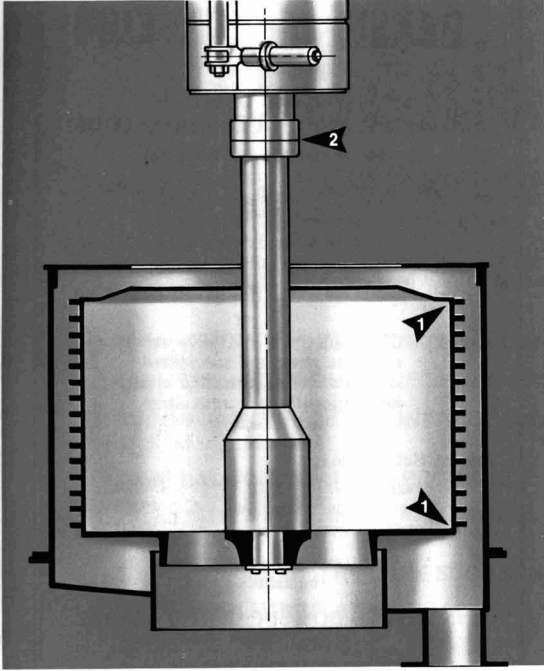
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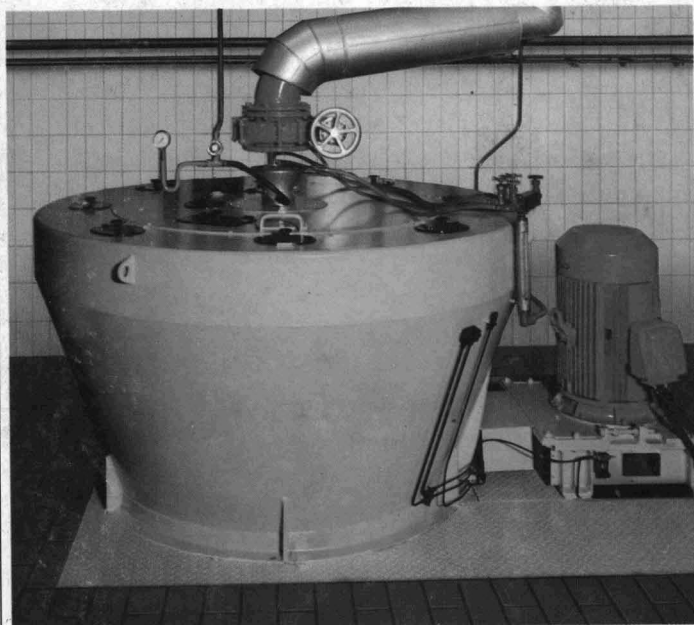
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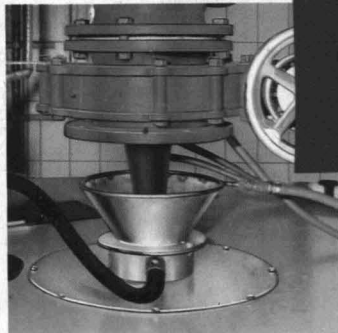


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