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## Notes and comments


be quantified. The Thailand quota in effect is subject to charges following overshipment in previous years. Honduras, one of the Annex II small exporters permitted to export up to 70,000 tonnes, expects to be able to export more and has submitted an application for an allocation from the hardship reserve.

## World sugar prices

The London Daily Price for raw sugar started the month of May at $£ 114.50$ ( $\$ 160$ ) per tonne but release of a large quantity of sugar by the EEC and reports from Thailand of a bumper crop brought the price down rapidly, as did news of Coca Cola's decision to allow use of $100 \%$ HFS in its fountain syrups, and new estimates by FIRS and the USDA of higher prod-

|  | Previous quotas in effect | Shortfall declaration | Current quotas in effect |
| :---: | :---: | :---: | :---: |
| Agentina | 626,450 | - | 626,450 |
| Australia | 2,829,874 | - | 2,829,874 |
| Austria | 113,250 | - | 113,250 |
| Bolivia | 114,410 | 64,865 | 49,605 |
| bazil | 2,798,095 | - | 2,798,095 |
| Colombia | 304,036 | - | 304,036 |
| Costa Rica | 86,335 | - | 86,335 |
| Cuba | 2,403,094 | - | 2,403,094 |
| Dominican Republic | 984,870 | - | 984,870 |
| Ecuador | 78,784 | 78,784 | 0 |
| Fji | 292,224 | - | 292,224 |
| Guatemala | 231,038 | - | 231,038 |
| Guyana | 125,480 | - | 125,480 |
| India | 700,400 | - | 700,400 |
| Tamaica | 83,773 | 48,211 | 35,562 |
| Malawi | 101,935 | - | 101,935 |
| Mauritius | 157,907 | - | 157,907 |
| Mexico | 70,000 | 70,000 | 0 |
| Mozambique | 78,951 | 28,951 | 50,000 |
| Wicaragua | 106,545 | - | 106,545 |
| Panama | 163,770 | 22,515 | 141,255 |
| Peru | 200,600 | 200,600 | 0 |
| Prilippines | 1,610,014 | 310,014 | 1,300,000 |
| ESalvador | 119,233 | - | 119,233 |
| South Africa | 878,080 | - | 878,080 |
| Swaziland | 251,768 | - | 251,768 |
| Thailand | 1,175,509 | - | 1,175,509 |
| Zimbabwe | 221,330 | $\underline{-}$ | 221,330 |
|  | $\overline{16,977,815}$ | 873,940 | $\underline{\underline{16,103,875}}$ |

uction than earlier forecast. The LDP fell to $£ 105$ by May 8 ; subsequently the price fluctuated rapidly but within a range of £105-£110 except for a sudden rise to £113 on May 22. News of increased activity in the Gulf War was a factor provoking price rises while news of cancellation of a shipment of Brazilian sugar to Venezuela was a depressing influence, as was the persistent awareness of the huge stocks overhanging the market. The month closed with an LDP of $£ 108$ (\$149) per tonne.

The LDP(W) was $\$ 177.50$ on May 1, representing a premium of $\$ 17.50$ over the LDP for raw sugar, and this premium gradually diminished during the month to reach only $\$ 8.50$ per tonne by May 23. After this it increased again to reach $\$ 12$ on May 29 but fell to $\$ 10$ on May 31 on which date the London Daily Price for white sugar was $\$ 159$ per tonne.

## Thailand sugar crop improvement ${ }^{2}$

Thailand's 1983/84 sugar production has improved substantially during recent months, and trade sources now expect it to exceed 2.3 million tonnes, raw value. By April 21, 22,570,000 tonnes of cane had been crushed, yielding $2,230,000$ tonnes of sugar, against the original estimate of $17,500,000$ tonnes of cane for the whole season.

## USSR sugar import needs ${ }^{3}$

In recent years the Soviet Union has become a major force in the international sugar market. Although it is one of the top producers, so high is the level of domestic consumption that it has become the world's largest importer. Clearly, therefore, the statistical position of the USSR can have an important effect on the evolution of market prices.

Unfortunately it does not submit full statistics to the International Sugar Organization and it is therefore difficult to

1 C. Czarnikow Ltd., Sugar Review, 1984, (1702), 96-97.

2 F. O. Licht, International Sugar Rpt., 1984, 116, 266.
3 C. Czarnikow Ltd., Sugar Review, 1984, (1696), 68.
build up a composite picture of the supply position of that country. Domestic industry magazines sometimes provide useful information, however, and this, together with the officially released details, can be used in an attempt to get a clearer picture of the situation.

Production of sugar from domestic beet was very poor in 1981/82, improved the following season, and in 1983/84 was, in terms of normal yields for that country, very good. Although full crop statistics have not yet been revealed, it is calculated that production last campaign amounted to around 8.75 million tonnes. Consumption has been increasing regularly and must by now be in the region of 13.1 million tonnes in terms of raw sugar so that, taking into consideration exports of around 200,000 tonnes and assuming that there will be no important change in the level of stocks, imports in 1983/84 will need to be of the order of 4.5-4.6 million tonnes.

This is well below the recent level and is the reason why the USSR has had so little impact in the market in recent months. For the health of the market it is perhaps as well that the availability of sugar from Cuba and the EEC, the two leading suppliers to the Soviet Union, has also fallen.

First indications are that sowings in the Soviet Union are behind hand compared with last year. How they will compare with a normal year remains to be seen but with the area roughly unchanged it might be realistic to forecast output at the average of the past three years or, say, around 7.5 million tonnes. Allowing for an increase in consumption of some 150,000 tonnes and exports once again at about 200,000 tonnes, that will leave a gap of the order of six million tonnes. Assuming once again that stock levels are left unchanged this will all have to be met by imports but would not be an unduly high tonnage, given the pattern of the past few years. It must be emphasized, however, that the figures have to be considered extremely tentative.

## World sugar balance, 1983/84

F. O. Licht GmbH recently published ${ }^{1}$

|  | 1983/84 | 1982/83 | 1981/82 |
| :---: | :---: | :---: | :---: |
|  |  | tonnes, raw value |  |
| Initial stocks | 37,943,000 | 32,501,000 | 24,445,000 |
| Production | 96,055,000 | 100,658,000 | 100,881,000 |
|  | 133,998,000 | 133,159,000 | 125,326,000 |
| Imports | 28,019,000 | 29,246,000 | 31,858,000 |
| Less Exports | -28,512,000 | -29,841,000 | -32,143,000 |
|  | 133,505,000 | 132,564,000 | 125,041,000 |
| Consumption | 96,878,000 | 94,621,000 | 92,540,000 |
| Final stocks | 36,627,000 | 37,943,000 | 32,501,000 |
| , "\% Consumption | - 37.81 | 40.10 | 35.12 |

its third estimate of world sugar movement in the six seasons up to and including September 1983/August 1984, and the more recent figures are reproduced above. Consumption is set to rise by $2.39 \%$ in 1983/84 bringing it to 800,000 tonnes more than production; final stocks are now expected to fall by 1.3 million tonnes but are still nearly 37 million tonnes - as a consequence of two years of very high output - although this represents a small fall to $37.8 \%$ of consumption, against $40.1 \%$ for 1982/83. The 1983/84 level of production reflects a remarkable recovery from what had been expected at the beginning of the season, not only in Europe but also, in the Philippines and Thailand.

## Indian sugar situation

Production of sugar in India in 1981/82 amounted to 8.4 million tonnes, white value, and another very large crop - 8.2 million tonnes - was produced in the following season. As an exporting member of the ISA India's opportunities for disposal of its large availabilities were limited and stocks rose dramatically. The government instituted measures to encourage consumption and discourage production; nevertheless, at the start of the 1983/84 crop forecasts were of the order of seven million tonnes.

As the season has progressed, however, it has become clear that the level of production is likely to be reduced further and estimates vary between 6.5 million and less than 6 million tonnes, while it seems probable that consumption will reach 7
million tonnes. This is not likely to cause any problem for India in meeting export commitments because of the high stocks; indeed, an application is before the I.S.O. for an increase in the 1984 export quota from the current level of 700,000 tonnes, raw value, to one million tonnes.

The fall in production is attributed to a slightly smaller cane area, lack of rain in northern India and to diversion of cane to gur manufacture.

## Australian sugar season, 1983/84 ${ }^{2}$

The 1983/84 sugar season saw a drop in sugar production for both Queensland and New South Wales, compared with the previous season's record output. Overall production for Australia is set at $3,170,745$ tonnes, 94 N.T., a drop of more than $10 \%$ from the $3,537,358$ tonnes of the previous crop. The fall reflected the effects of a severe drought in the early part of 1983 when the cane crop is normally in its major growth period. Good rains in May and June improved prospects so that estimates of the cane tonnage gradually rose and the total crushed amounted to $24,189,813$ tonnes, harvested from 307,243 ha, against $24,907,748$ tonnes of cane crushed in 1982/83, harvested from 318,693 ha. The erratic weather also showed its influence in the cane sugar content which was lower in all areas than in previous years. The tonnes cane per tonne sugar figure in Queensland averaged 7.54 which was the highest since 1978 and compared with the 6.95 obtained in the previous season and the record low of 6.66 of 1972.
1 International Sugar Rpt., 1984, 116, 249-255. 2 Australian Sugar J., 1984, 75, 530-531.

# Analysis of carboxylic acids formed by alkaline degradation of invert sugar 

A comparison between liquid and gas chromatography

By J. M. De Bruijn*, A. P. G. Kieboom*, H. van Bekkum* and P. W. van der Poel ${ }^{\dagger}$

## Introduction

Invert sugar is present in diffusion juices in amounts between 0.5 and $1.5 \mathrm{~g} / 100 \mathrm{~g}$ sucrose. Apart from its presence in sugar beets, invert sugar also finds its origin in the enzymatic hydrolysis of sucrose during the diffusion process. One of the aims of main liming is degradation of invert sugar to thermostable components, thus preventing or limiting pH drops in the evaporators. This destruction of invert sugar under alkaline conditions has been studied extensively ${ }^{1-8}$, but is not completely *lucidated yet with respect to the deficit in the mass balance ${ }^{4}$, and mechanistic and kinetic features. Two types of degradation products can be distinguished: carboxylic acids ${ }^{1-4}$ and non-acidic products of higher - molecular weight ${ }^{5-8}$, which are either coloured or considered as precursors for colour formation.
As a part of the Delft research program on carbohydrate chemistry, we have been engaged in the study of the various reactions of monosaccharides in aqueous alkaline media ${ }^{4}$. In this respect ionization ${ }^{9}$, mutarotation ${ }^{9}$, enediol anion formation ${ }^{10}$, isomerization ${ }^{11}$ and alkaline degradation ${ }^{4,11}$ have been investigated in order to obtain further insight into the mechanisms of these transformations. At present, a common investigation of the degradation reaction of invert sugar is being performed ${ }^{12}$ in order (i) to study the influence of reaction variables on product formation and (ii) to compare results from laboratory experiments with those from aprocedures on a technical scale. A rapid and quantitative analysis technique is a prerequisite for this investigation, in particular to follow the course of the reaction as a function of time.

- The quantitative analysis of the acidic part of the products formed is known to be difficult as the composition of the reaction mixture after alkaline degradation of invert sugar is rather complex. The mixture contains more than ten different carboxylic acids; among these lactic acid, glycolic acid, formic acid, acetic acid, $\mathrm{C}_{6}$-saccharinic acids (saccharinic, metasaccharinic, and isosaccharinic acid), and 2,4-dihydroxybutyric acid are the most

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abundant. Paper and thin-layer chromatography are not very suitable, since these analysis methods give only a qualitative or semi-quantitative picture of the composition of the reaction mixture. Therefore, quantitative analysis of the acidic products requires gas chromatography (GC), as studied by Oldfield et al. ${ }^{13}$ and Reinefeld et al. ${ }^{14,15}$, and/or high performance liquid chromatography (HPLC), as studied by Charles ${ }^{16}$ and Kubadinow ${ }^{17}$.

In this paper the scope and limitations of both GC and HPLC analysis techniques will be discussed on the basis of industrial juice samples as well as reaction mixtures from the alkaline degradation of invert sugar in laboratory experiments.

## Experimental

## GC analysis

Sucrose and non-acidic degradation products in juice samples were removed by means of an ion exchange clean-up procedure as described by Oldfield et al. ${ }^{13}$. Samples from model experiments were neutralized in the cold $\left(<4^{\circ} \mathrm{C}\right)$ with a weak cation exchange resin (BioRex 70 H -form, supplied by BioRad) in order to prevent lactonization of the saccharinic acids.

After freeze-drying of the samples, the carboxylic acids were dissolved in pyridine
and converted into their trimethylsilyl (TMS) derivatives by the method of Petersson ${ }^{18}$.
The samples thus obtained were analysed with a Varian Model 3700 gas chromatograph, equipped with a capillary CP Syl 5 column ( 25 m length, 0.23 mm i.d.), a splitter (splitter ratio 100:1), and a flame ionization detector (FID).
The temperature of the column oven was programmed as follows: 5 min at $75^{\circ} \mathrm{C}$, increasing to $280^{\circ} \mathrm{C}$ at a rate of $8^{\circ} \mathrm{C} / \mathrm{min}$, and another stationary period of 5 min at $280^{\circ} \mathrm{C}$. The temperature of injector and FID was $230^{\circ} \mathrm{C}$.
Identification of the trimethylsilylated carboxylic acids was performed by mass spectrometry using a Varian GC-MS system Mat 44 in which the GC part was identical with that described above.

## HPLC analysis

The samples either freed from sugars and neutral products or neutralized as described above were analysed on an "organic acid analysis" column from BioRad (HPX 87, 300 mm length, 7.8 mm

[^0]i.d.). This stainless steel column contains a sulphonated styrene-divinylbenzene copolymer as the strong cation exchange resin (cross-linkage 8\%; particle size 7-11 $\mu \mathrm{m} ;-\mathrm{SO}_{3} \mathrm{H}$ content $1.7 \mathrm{mmol} / \mathrm{ml}$ ).
A micro guard column (supplied by BioRad), containing the same material as the analytical column, was used to prevent column fouling.
The resin was always applied in the hydrogen form, which was shown to give the best results ${ }^{16,17,19,20}$ at $60^{\circ} \mathrm{C}$ using an $0.005 \mathrm{M} \mathrm{H}_{2} \mathrm{SO}_{4}$ aqueous solution ( 0.6 $\mathrm{ml} / \mathrm{min}$ ) as the eluent.
The equipment used was as follows: Waters Associates Chromatography Pump M-6000 A, Differential Refractometer RI 401 and Differential Refractometer Electronics unit, Rheodyne injector with a $100 \mu \mathrm{l}$ sample loop.

## Results and discussion

## Gas chromatography

Gas chromatographic analyses together
with mass spectrometric identification ${ }^{21-23}$ of a juice sample, taken from the evaporation unit of the CSM sugar factory at Breda, Holland, allowed the assignment of 17 peaks (Fig. 1).
The gas chromatogram shows an excellent separation of the various acidic products. Citric, malic and oxalic acid are already present in the sugar beet and have not been eliminated completely in the juice purification.
Pyrrolidonecarboxylic acid originates from saponification of glutamine. The other acids arise from alkaline degradation of invert sugar during the main liming process or from microbial destruction of sugar. The 1,4 -lactones of the $\mathrm{C}_{6}$-saccharinic acids are the result of the ion exchange clean-up procedure of the juice sample before derivatization, as mentioned in the experimental part. Performing this procedure, first of all the samples pass a strong cation exchange column, resulting in a sharp drop to pH 2 .


Fig. 1. Gas chromatogram of carboxylic acids (as their TMS-derivatives) of a thick juice sample from the evaporation unit

1. lactic acid
2. glycolic acid
3. oxalic acid
4. 2-C-Me-glyceric acid
5. glyceric acid
6. 2,4-dihydroxybutyric acid
7. malic acid
8. pyrrolidonecarboxylic acid
9. tetronic acid
10. 3-deoxypentonic acid
11. citric acid
12. $\mathrm{C}_{6}$-saccharinic acids
a. metasaccharinic acid
$\mathbf{a}^{\prime}$. metasaccharinic acid-1,4-lactone
b. isosaccharinic acid
$b^{\prime}$. isosaccharinic acid-1,4-lactone
c. saccharinic acid
$c^{\prime}$. saccharinic acid-1,4-lactone

At room temperature this causes partial lactonization of the $\mathrm{C}_{6}$-saccharinic acids before they enter the second column containing a strong anion exchange resin.

For comparison, Fig. 2 shows a typical example of a gas chromatogram of a reaction mixture from a laboratory experiment, in which glucose is completely degraded. On this chromatogram no $\mathrm{C}_{6}$-saccharinic acid lactones are present, because for neutralization the reaction mixture is treated in the cold with a weak cation exchange resin, as described in the experimental part, thus preventing lactonization.


Fig. 2. Gas chromatogram of acidic products (as their TMS-derivatives) from a laboratory experiment, in which glucose is degraded in alkaline medium. Reaction conditions: aqueous solution of 0.03 M glucose and 0.01 M KOH , $80^{\circ} \mathrm{C}, \mathrm{N}_{2}, 7 \mathrm{hr}$. During the reaction the pH was kept constant by the addition of 2 M KOH . Numbering of the peaks as in Fig. 1. Chromatographic conditions: see experimental part
Formic acid and acetic acid, normally formed upon alkaline degradation of monosaccharides, cannot be found in the gas chromatogram, because their TMS-
19 Kapp \& Ziegler: Chromatographia, 1976, 9, 148-150.
20 Turkelson \& Richards: Anal. Chem., 1978, 50, 1420-1423.
21 Petersson \& Samuelson: Acta Chem. Scand., 1967, 21, 1251-1256.
2 Petersson: Tetrahedron, 1970, 26, 3413-3428. 3 Idem: Org. Mass Spectr., 1972, 6, 577-592.
derivatives are too volatile and have the same retention as an unretained compound. For quantitative analysis of the acids, the response factors can be calculated by the method of Verhaar \& de Wilt ${ }^{24}$. However, the wide range of volatilities of the acid derivatives in combination with the splitting of injected samples make the quantitative analysis of acids unreproducible. This uncertainty of GC analyses can be prevented by oncolumn injection, or by using a packed column ${ }^{3,4,13-15}$. The disadvantage of using a packed column is the lower efficiency by comparison with a capillary column, whereas a splitterless injector system requires a more tedious injection procedure.

## - Liquid chromatography

HPLC analysis of a juice sample, also taken from the evaporator of the Breda sugar factory, showed ten separated peaks (Fig. 3). In order to identify these peaks


Fig. 3. HPLC separation of carboxylic acids of a thick juice sample from the evaporation unit. Numbering of the peaks as in Fig. 1, plus 13. formic acid, and 14. acetic acid.
Chromatographic conditions: see experimental part
we first carried out a preparative HPLC separation by injecting twenty times 0.6 mg of the juice sample on the same column. 12 fractions were collected after the detector, neutralized ( 2 M KOH ), freezedried, and trimethylsilylated. Subsequent GC-MS analysis of the fractions allowed us to assign the peaks in the liquid chromatogram.

The peaks of formic acid and acetic acid were identified by comparison with the retention times of the pure acids. For comparison Fig. 4 shows the HPLC separation of the main components involved in the alkaline degradation of invert sugar, while Fig. 5 shows a typical analysis of a laboratory experiment. As may be seen in the liquid chromatograms of the Figures 3, 4, and 5, not all of the acids are separated: the three $\mathrm{C}_{6}$-saccharinic acids possess the same retention time, the $\mathrm{C}_{6}$-saccharinic acids, glycolic acid and lactic acid show some overlap, and 2,4-dihydroxybutyric acid is not completely separated from formic acid and acetic acid.


Fig. 4. HPLC separation of some compounds involved in the alkaline degradation of invert sugar. Numbering of the peaks as in Figs. 1 and 3 , plus 15 . glucose, and 16. fructose. Chromatographic conditions: see experimental part


Fig. 5. Liquid chromatogram of a laboratory experiment in which glucose is degraded in alkaline medium. Reaction conditions: aqueous solution of 0.005 M glucose and 0.009 M KOH , $80^{\circ} \mathrm{C}, \mathrm{N}_{2}, 7 \mathrm{hr}$. During the reaction the pH was kept constant by the addition of 2 M KOH . Numbering of the peaks as in Figs. 1 and 3. Chromatographic conditions: see experimenal part

Quantitative analysis of the carboxylic acids was derived from the peak heights, which proved to be a reproducible method. The relative response factors of a number of carboxylic acids are summarized in Table I.

With HPLC it is possible to analyse samples from the laboratory experiments in a straight-forward manner: neutralization of the samples, as described in the experimental part, is the only prerequisite necessary. If these samples still contain invert sugar, analysis of both glucose, fructose and the carboxylic acid can be performed simultaneously, as shown by Fig. 4.

On the other hand, sucrose, present in juice samples from the sugar factory, cannot be directly analysed under the separation conditions described, because 24 J. Chromatogr., 1969, 41, 168-179.

| Table I. Relative response factors of carboxylic <br> acids, as derived from peak heights in liquid <br> chromatograms  | Relative <br> Carboxylic acid |
| :--- | :---: |
| response factor |  |
| 2,4-Dihydroxybutyric acid | 0.22 |
| Metasaccharinic acid | 0.42 |
| Formic acid | 0.51 |
| Isosaccharinic acid | 0.53 |
| Acetic acid | 0.61 |
| Pyrrolidonecarboxylic acid | 0.96 |
| Glycolic acid | 0.97 |
| Lactic acid | 1.00 |
| Glyceric acid | 1.42 |
| Malic acid | 1.44 |
| Citric acid | 1.69 |
| a Chromatographic conditions: see |  |
| experimental part. |  |

sucrose is partly hydrolysed in the column into invert sugar. This is shown in Fig. 6. Therefore, it is recommended to remove the large amount of sucrose by an ion exchange clean-up procedure ${ }^{13}$.


Fig. 6. Liquid chromatogram of sucrose showing hydrolysis in the column during elution.
Chromatographic conditions: see experimental part

## Comparison of GC and HPLC separation

The efficiency N of a chromatographic column may be defined by:

$$
\mathrm{N}=5.54\left(\frac{\mathrm{t}_{\mathrm{r}}}{\mathrm{w}^{1 / 2 h}}\right)^{2}
$$

in which $t_{r}$ is the retention time of a compound and $w^{1 / 2 h}$ is the peak width at half peak height. The GC capillary CP Syl 5 column has a much higher efficiency ( $\mathrm{N} \sim 1.5 \times 10^{6}$ ) than the HPLC HPX 87 column ( $\mathrm{N} \sim 1.1 \times 10^{4}$ ) as is demonstrated by the great difference in peak widths.

The capacity factor $k^{\prime}$ of a compound may be defined by

$$
k^{\prime}=\left(\frac{t_{\mathrm{r}}-t_{\mathrm{o}}}{\mathrm{t}_{\mathrm{o}}}\right)
$$

in which $t_{r}-t_{o}$ is the difference in retention time between an unretained and a retained compound. As shown in Table II the range in $\mathrm{k}^{\prime}$ values of the carboxylic acids is much higher for GC than for HPLC, so with CG the carboxylic acids can be separated over a relatively larger range of retention times.
Table II. $\mathbf{k}^{\prime}$ values of carboxylic acids for both GC and HPLC separation ${ }^{\text {a }}$

| Carboxylic acid | $k^{\prime} G C$ | $k^{\prime} H P L C$ |
| :--- | :---: | ---: |
| Formic acid | 0 | 1.43 |
| Acetic acid | 0 | 1.64 |
| Glycolic acid | 1.79 | 1.19 |
| Lactic acid | 1.71 | 1.28 |
| Glyceric acid | 3.40 | 1.01 |
| 2,4-Dihydroxybutyric acid | 3.81 | 1.51 |
| 2-C-Me-glyceric acid | 3.35 | 0.98 |
| Tetronic acid | 4.50 | 0.97 |
| Malic acid | 3.61 | 0.74 |
| Pyrrolidonecarboxylic acid | 3.65 | 2.22 |
| 3-Deoxypentonic acid | 4.98 | 1.29 |
| Metasaccharinic acid | 5.80 | 1.01 |
| Isosaccharinic acid | 5.84 | 1.04 |
| Saccharinic acid | 5.87 | 1.01 |
| Citric acid | 5.57 | 0.45 |

${ }^{\text {a }}$ Chromatographic conditions: see experimental part.

Both measures of separation quality N and $k^{\prime}$ show that GC gives by far the best separation results. On the other hand, the possibility of analysing both formic and acetic acid with HPLC is an advantage of this technique with respect to GC.

Table III summarizes and compares some characteristics of GC and HPLC analyses.


Fig. 7. Correlation of GC and HPLC for acidic (degradation) products. Chromatographic conditions: see experimental part
and the comparison of both analytical techniques discussed in this paper we may conclude the following:
The major advantage of HPLC is that it

| Table III. Comparison of GC and HPLC analysis of carboxylic acids ${ }^{\mathrm{a}}$ |  |  |
| :--- | :---: | :---: |
| Property | $G C$ | $H P L C$ |
| Sample preparation time | 1 day | $5 \mathrm{~min}(60 \mathrm{~min})^{\mathrm{b}}$ |
| Analysis time | 30 min | 20 min |
| Separation quality | ++ | + |
| Direct analysis of formic | - | + |
| acid and acetic acid | - | + |
| Quantification |  |  |
| a Chromatographic conditions: see experimental part |  |  |
| b $^{\text {Removal of sucrose in the case of an industrial sugar juice }}$ |  |  |

The capacity factors of some carboxylic acids by both gas and liquid
chromatographic separation are graphically represented in Fig. 7, in which the location of the different peaks of the carboxylic acids is indicated with the aid of simulated gas and liquid chromatograms.

With the help of this correlation chart
requires no time-consuming freeze-drying and derivatization of the samples. Thus, despite its lower separation capacity, HPLC is a very rapid, convenient, and reproducible method for the quantitative analysis of acidic products from alkaline degradation reactions. On the other hand, GC analysis may be preferred for special
purposes, for instance, when the ratio of isosaccharinic acid and metasaccharinic acid has to be investigated.

## Acknowledgement

Thanks are due to N. H. M. de Visser and M. A. M. de Schutter for helpful discussions. Acknowledgements are due to Dr. G. J. Verschoor for carrying out some preliminary experiments.

## Summary

A comparison is made between GC and HPLC analysis of carboxylic acids formed by alkaline degradation of invert sugar. GC analyses of the trimethylsilylated carboxylic acids have been performed on a capillary column with temperatureprogramming. HPLC analyses of the tarboxylic acids were carried out on a strong cation exchange resin in the hydrogen form. Despite the higher resolution of GC, HPLC has been adopted for the authors' research since it has proved to be a convenient and reproducible method for routine analysis of carboxylic acids: there is no need for time-consuming freeze-drying and derivatization procedures and volatile products like acetic and formic acids are quantitatively detected.

Analyse des acides carboxyliques formés par dégradation alcaline du sucre inverti. Comparaison entre chromatographic liquide et gazeuse.
On a comparé l'analyse par chromatographie gazeuse (GC) et par chromatographie liquide à haute わerformance (HPLC) des acides carboxyliques formés lors de la dégradation alcaline du sucre inverti. Les analyses par GC des acides carboxyliques triméthylsilylés ont été exécutées sur une *olonne capillaire avec témperature programmée. Les analyses par HPLC des acides carboxyliques ont été effectuées sur une résine échangeuse cationique forte sous forme hydrogène. En dépit de la plus haute résolution de la GC, l'HPLC a été adoptée par les auteurs pour leurs recherches parce que cette dernière s'èst avérée une méthode convenable et reproductible pour une analyse de routine des acides carboxyliques. Le séchage à
basse température et la formation de dérivés, qui prennent beaucoup de temps, ne sont pas nécessaires dans ce cas et les produits volatils, tels que les acides acétique et formique, sont détectés quantitativement.

Analyse der durch alkalischen Abbau von Invertzucker gebildeten Carbonsäuren ein Vergleich zwischen Flüssigkeits- und Gaschromatographie.

Gaschromatographie und HPLC wurden bei der Analyse der durch alkalischen Abbau von Invertzucker gebildeten Carbonsäuren verglichen. Die Gaschromatographie der Trimethylsilycarbonsäuren wurde in einer Kapilaräule mit Temperaturprogrammierung durchgeführt, und die HPLC-Analysen der Carbonsäuren erfolgte an eienem starken Kationenaustauscherharz in der $\mathrm{H}^{+}$-Form. Trotz der höheren Trennschärfe der Gaschromatographie, verwendeten die Autoren bei ihren Untersuchungen die HPLC, da sie sich als geeignete und reproduktionsfähige Methode für Routineanalysen von Carbonsäuren erwiesen hat; man braucht weder zeitaufwendige Gefriertrocknung noch

Derivatisierungsverfahren, und flüchtige Substanzen wie Essig- und Ameisensäure können quantitativ ermittelt werden.

## Análisis de ácidos carboxilicos formados por degradación alcalina de azúcar invertido. Comparación entre cromatografía líquida y gaseosa.

Se han comparado cromatografía gaseosa (GC) y cromatografía líquida de funcionamiento alto (HPLC) en el análisis de ácidos carboxilicos formados por degradación alcalina de azúcar invertido. Analisises por GC de ácidos carboxilicos trimetilsililados se han cumplido en una columna capilaria con temperatura programada. Analisises por HPLC de los ácidos carboxilicos se han cumplido con una résina de cambio de cationes fuertes en la forma $\mathbf{H}^{+}$. A despecho de la resolución mejor de la GC, los autores han adoptado la HPLC para su experimentación porque se ha resultado un método conveniente y reproductible para análisis rutinario de ácidos carboxilicos; no hay necesidad de secar a temperaturas bajas o de formar derivados, que requieren mucho tiempo, y productos volátiles como ácido acético y ácido fórmico se miden cuantitativemente.

## Brevities

Cuban efforts to meet sugar production target ${ }^{1}$
Rains, industrial breakdowns, low sugar extraction and absenteeism have plagued the 1984 cane sugar season in Cuba and efforts are being made to recover the shortfall of more than $1,100,000$ tonnes below target output at May $1^{2}$. The industry is aiming at an output at least one million tonnes greater than the 7.2 million tonnes of the 1983 crop and the goal set before the harvest began was 8.5 million tonnes. This would be the second biggest crop in Cuban history after the 1970 record of 8.6 million tonnes. In that year crushing continued long after the normal season end at the start of the wet season and this year also it was announced that the original harvest end date of April 30 was no longer valid and that crushing would continue to May 17; several industry officials have admitted that it might have to continue further in many regions of the country, and this may affect replanting, weeding and fertilizing for the 1985 crop.

## EEC-ACP sugar price negotiations ${ }^{3}$

At a conference between Ministers of the EEC and ACP countries in May the latter were advised that no possibility was foreseen for the
increase of guaranteed prices of ACP sugar on the EEC market. As no price increases were granted to domestic producers, the Commission considers that ACP producers are not discriminated against by a freeze on prices. Moreover, the EEC is not ready to agree to requests by ACP countries for a transfer of unutilized preferential sugar quotas from one country to another.

## New bagasse paper factory in Indonesia ${ }^{1}$

A new paper mill using cane waste as raw material has begun production at Probolinggo, approximately 100 km east of Surabaya in East Java. It is an enlargement of an existing 100 tonnes/day plant using rice straw and bagasse, and was built at a cost of $\$ 250$ million by a West German firm and two Austrian firms. A further expansion is scheduled for completion in 1985 which will produce approximately 300 tonnes of newsprint per day using pulp made from bagasse.
1 F. O. Licht, International Sugar Rpt., 1984, 116, 201-202.
2 C. Czarnikow Ltd., Sugar Review, 1984, (1701), 89.

3 F. O. Licht, International Sugar Rpt., 1984, 116, 220.
4 World Sugar J., 1984, 6, (9), 41.

# Dehydration of exhausted cossettes 

By Jan Dobrzycki and Stanislaw Wawro<br>(Technical University, Lodz, Poland)

## Introduction

The idea of osmotic dehydration of exhausted cossettes by means of molasses is investigated in this paper in order to obtain fundamental information only about the velocity of the process and the limits of dehydration obtainable in reasonable time. The authors realize how many technical and economical difficulties can arise on a factory scale but they have left this problem beyond the scope of this communication.

The osmotic dehydration of beet pulp consumes very little energy in comparison with thermal dehydration by drying. On the other hand diluted molasses obtained during the counter-current dehydration could be utilized in some factories for fermentation or in the desugarizing plant.

## Experimental

## Diffusion in the beet tissue

A flat slice 15 mm thick was cut from the beet perpendicularly to the axis of the root, plasmolysed, and digested for 12 hours in hot water. From the exhausted beet tissue a cylinder 40 mm diameter $\times 15 \mathrm{~mm}$ was cut out and placed tightly in a tube with one end open (Fig. 1). The free


Fig. 1. Tube 1 with sample 2


Fig. 2. Components of osmotic dehydrated beet pulp
end of the cylinder was held in contact with warm molasses flowing in excess with a velocity of about $0.3 \mathrm{~m} / \mathrm{s}$. After a predetermined time of diffusion the sample
was cut into discs 1 mm thick by means of a special device perpendicularly to the axis
Paper presented to the 17th Gen. Assembly CITS, 1983.

Table I. Penetration of molasses into the tissue of exhausted cossettes (molasses RDS $\mathbf{8 3 \%}, \mathbf{5 0 ^ { \circ }} \mathbf{C}$ )
mass of slice, g
$b$ water added for digestion, g
c RDS of digestate, \%
d marc content in the slice, \%
M molasses content in the slice, \%
RW residual water remaining in the slice, \%
CDS calculated dry substance of "net" dehydrated pulp, \%
After 60 minutes diffusion

| Slice | After 60 minutes diffusion |  |  |  |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| number | $a$ | $b$ | $c$ | $d$ | M | RW | CDS |  |
| 1 | 0.1305 | 0.9522 | 3.116 | 12.64 | 30.7 | 56.7 | 18.2 |  |
| 2 | 0.1334 | 0.9660 | 1.965 | 7.79 | 19.3 | 72.9 | 9.7 |  |
| 3 | 0.1858 | 0.9358 | 1.839 | 5.43 | 13.2 | 81.3 | 6.3 |  |
| 4 | 0.1608 | 0.9639 | 1.069 | 5.0 | 8.9 | 86.1 | 5.5 |  |
| 5 | 0.1772 | 0.9276 | 1.029 | 4.85 | 7.65 | 87.5 | 5.25 |  |
| After 90 minutes diffusion |  |  |  |  |  |  |  |  |
| 1 | 0.0957 | 0.9084 | 3.925 | 13.27 | 48.4 | 38.3 | 25.7 |  |
| 2 | 0.1452 | 0.9737 | 3.616 | 11.01 | 32.7 | 56.3 | 16.35 |  |
| 3 | 0.1647 | 0.9347 | 2.586 | 5.58 | 20.4 | 74.0 | 7.0 |  |
| 4 | 0.1483 | 0.9998 | 1.818 | 5.05 | 16.65 | 78.3 | 6.0 |  |
| 5 | 0.1275 | 0.9154 | 1.419 | 4.86 | 14.0 | 81.1 | 5.65 |  |





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of the cylinder.
From each disc the peripheral ring was rejected and the central slice of diameter 15 mm subjected to analyses. After the addition of $b$ grams water the slice (mass $a$ grams) was digested and the refractometric
dry substance (RDS) of the digestate ( $\mathrm{c} \%$ ) was measured. Then the slice was sweetened-off with water and dried to determine its marc content ( $d \%$ ).

From these measurements the following values were calculated:



Molasses percentage in the dehydrated slice

$$
\begin{aligned}
& \mathrm{M}=\frac{\frac{b}{a}+1-0.1 d}{\mathrm{RDS}_{\mathrm{mol}}} 100 c \\
& \mathrm{~g} / 100 \mathrm{~g} \text { dehydrated pulp }
\end{aligned}
$$

Residual water from the original pulp
$\mathrm{RW}=100-d-\mathrm{M}$
$\mathrm{g} / 100 \mathrm{~g}$ dehydrated pulp
Amount of "net" dehydrated pulp (Fig. 2)
RW $+d \quad \mathrm{~g} / 100 \mathrm{~g}$ dehydrated pulp Calculated dry substance content CDS in the "net" dehydrated pulp i.e. after subtraction of molasses components

$$
\begin{aligned}
& \mathrm{CDS}=\frac{100 d}{d \div \mathrm{RW}} \\
& \mathrm{~g} / 100 \mathrm{~g} \text { "net" dehydrated pulp }
\end{aligned}
$$

The results of one experiment are shown in Table I and Fig. 3 illustrates the penetration of molasses dry matter into the beet tissue as function of the depth after various times. The diffusion is disturbed by the counter-current diffusion of water from deeper regions and by shrinkage of the tissue under the influence of external osmotic pressure.

## Batch dehydration

Some 200 cylindrical cossettes, of diameter $5 \mathrm{~mm} \times 25$ or 35 mm , were plasmolysed, desugarized with hot water and then placed in warm molasses used in excess and stirred gently. After a predetermined time of contact, samples of 20 cossettes each were removed, wiped with filter paper and analysed.

From the total dry substances content $e(\%)$ determined in a drying oven, the marc content $d(\%)$ in treated samples and from molasses RDS the composition of the samples was calculated:

Molasses content in dehydrated pulp

$$
\mathrm{M}=\frac{e-d}{\mathrm{RSD}_{\text {mol }}} 100
$$

( $\mathrm{g} / 100 \mathrm{~g}$ dehydrated pulp)
Residual pulp water RW and dry substance CDS in the "net" dehydrated pulp was calculated as above.

Figs. $4-7$ show the composition of 100 kg of exhausted cossettes dehydrated by molasses used in excess.

It can be seen from Figs. 4-7 that in four experiments the main part of the original water is eliminated during the first 20-40 minutes. The osmotic
shrinkage reduces the volume to about $1 / 4$ of the initial volume of exhausted beet tissue. After 2 hours the degree of dehydration reaches $60 \%$ DS in the "net"


Fig. 7
dehydrated pulp and after 4 hours more than 70\% DS.

## Conclusions

After prolonged contact with molasses the exhausted beet cossettes undergo osmotic dehydration and lose most of their water content. The product consists of dehydrated pulp with molasses penetrated into the tissue and covering its surface.

The dehydration can be carried to a dry substance content similar to that of molasses. The dehydration is accompanied by shrinkage to about $1 / 4$ of the initial pulp volume.

The investigation is only a preliminary step to further technical and economic analysis of the method.

## Summary

The paper deals with the possibility and effectiveness of osmotic dehydration of exhausted cossettes by means of final molasses. In contact with beet pulp warm molasses penetrates the tissue, expels water and causes considerable shrinkage of pulp.

## Déshydratation des cossettes épuisées

On a étudié la possibilité et l'efficacité de déshydratation osmotique des cossettes épuisées, par l'action de la melasse. En contact avec les cossettes la mélasse chaude pénètre le tissu, chasse l'eau et cause une contraction considérable des cossettes.

Entwässerung von ausgelaugten Schnitzeln ,
Die Möglichkeit der osmotischen Entwässerung von ausgelaugten Schnitzeln im Kontakt mit Melasse wurde untersucht. Warme Melasse dringt in das Schnitzelgewebe hinein, treibt Wasser heraus und verursacht bedeutende Schrumpfung der Schnitzel.

## Deshidratación de cosetas agotadas

Este artículo trata de la posibilidad y la efectividad de deshidratación por osmosis de cosetas agotadas con melaza final. En contacto con pulpa de remolacha, melaza caliente penetra los tejidos, la agua se expulsa y la pulpa sufre encogimiento notable.

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# Cane sugar manufacture 

## Progress in selected Sugar <br> Technology Department studies

K. Onna, R. Tamaye, D. Hsu and
B. Somera. Proc. 41st Conf. Hawaiian

Sugar Technologists, 1982, 130-132.
Progress in certain studies by staff of the HSPA Sugar Technology Dept., most of which are being continued, is reported: Calculated vs. analysed fibre \% bagasse: During a milling test, figures for fibre \% bagasse were obtained by direct analysis and by calculation as 100 - (moisture \% bagasse + estimated refractometric solids $\%$ bagasse); the analysed fibre \% bagasse value was lower than the calculated value by 2.7-4.0 units, and the difference was significant at the $95 \%$ confidence level. Most of the difference was due to the presence of soil and to the error in estimating refractometric solids \% bagasse from pol \% bagasse and last mill juice purity, while other contributory factors included loss of comminuted fibres during analysis.
Mixing efficiency of maceration water: Pol extraction is promoted by rupture of the juice cells and the fibrovascular system and by good mixing of free liquid (free pol in feed blanket plus imbibition water) with the generated free pol. The two processes were studied at the last mill; pol and moisture extraction values and displaceability index ( $\%$ free pol) were used to estimate the amounts of free and trapped pol in the feed blanket, extracted juice and bagasse. Mixing efficiency was then calculated in three different forms: (i) pol extraction/moisture extraction,
(ii) ( 100 pol extracted/free pol in feed)/moisture extraction, and (iii) [(100 pol extracted) /(free pol in feed + free pol generated)] divided by moisture extraction. The three expressions give widely differing values, and their validities are discussed. The need to define mixing efficiency before making any measurements is underlined.
On-line measurement of bagasse moisture: Failure to recalibrate a Quadrabeam Model 475 moisture analyser and permitting the distance between bagasse sample and sensor to vary resulted in a poor
correlation between meter reading and bagasse moisture during a one month's test. The system focuses two light beams on the sample - one is scattered, while the other is both scattered and partially absorbed by the moisture in the bagasse, and the relative intensities of the two scattered beams are used to indicate the moisture content. Another problem was the excessive speed with which the digital display changed and the extent of the change, which was too large for the meter signal to be used to control milling.
Cane juice preservative: Heavy metal compounds are, to date, the only chemicals found to stabilize juice samples; mercuric chloride is the one most commonly used, while lead acetate and subacetate are also effective preservatives, although they can affect measurements of refractometric solids. However, the chemicals mentioned are highly toxic, and there is a problem of safe disposal of treated samples. Several organic biocides were therefore tested as possible substitutes, but none was as effective as the heavy metal compounds without presenting a disposal problem. Electrodialysis: The economics of ash removal from syrups by electrodialysis were estimated for two plant sizes; for a medium factory producing about 60,000 short tons of sugar per year, treatment of a $30 \%$ RDS syrup containing an average $0.9 \%$ carbonate ash to reduce the ash content by $40 \%$ would give an internal rate of return (IRR) of $14 \%$, while the IRR for a larger factory producing about 150,000 short tons of sugar annually would be $22 \%$ under identical conditions of syrup and electrodialysis; for a syrup of $50 \%$ RDS and containing $20 \%$ more ash than the industrial average, a 40\% reduction in ash would yield IRR values of $25 \%$ and $35 \%$ for the medium and large factories, respectively. The data are based on extrapolation from a laboratory study with syrups of $25 \%$ RDS.
Saturation temperature cell: Details are given of a modified version of the saturation cell for measurement of molasses saturation temperature ${ }^{1}$. Test results have been encouraging, and a prototype was constructed in 1982.

The model comprises two optically clear glass plates which are electrically conducting at the surface; the sample is sandwiched between two microscope cover glasses and in turn sandwiched between the two glass plates. A thermocouple is inserted in the sample to measure its temperature. When electricity is passed across the face of the plate, heat is generated and rapidly transferred to the sample. An infrared light-emitting diode and a photodetector automatically establish the transmission minimum and hence the saturation temperature.
Tackmeter: Molasses tackiness or stickiness is the resistance to pull exerted by molasses adhering to two surfaces separating at a specified rate, and difficulties in boiling are frequently attributed to it. A "tackmeter" was constructed from a double-pan analytical balance, and comprised a sample plate, a tared sample finger connected to one arm of the balance, a set of pull weights and a timing system. The gap between the finger and plate is set to a desired value and a sample placed between them. When the balance arm arrestors are released, the timer is automatically started and the pull weight causes the finger to rise and pull the sample into a thread; when the thread reaches a predetermined length, the corresponding position of the balance arms triggers a photo-electric circuit and thus stops the timer. The tackiness is calculated from several runs using different weights. The results of an actual test are reported.

## Centrifugal washing and separator efficiency tests at the Oahu Sugar Company factory: summary of results to date

B. J. Somera. Proc. 41st Conf. Hawaiian Sugar Technologists, 1982, 133.
The higher raw sugar pol of 98.8 decided by the Hawaiian sugar industry in 1978 necessitated greater washing in the centrifugals. A double washing technique was introduced at Oahu Sugar Co. whereby the normal amount of wash water was split into two halves, giving a lower-purity
1 Wright: I.S.J., 1978, 80, 40-44.
molasses fraction and a higher-purity one; for separation of the fractions, a Western States swing-spout separator was installed in one centrifugal for test purposes, and the intention was to use the fractions for boiling of higher- and lower-purity massecuites, respectively. Results of tests showed that, for the same amount of wash water, single washing produced higherpurity sugar than double washing, although crystal dissolution and molasses purity rise were greater. With sufficient increase in the amounts of wash water for double washing to give the same sugar purities as obtained with single washing, crystal dissolution and molasses purity rises were still lower than with single washing; crystal dissolution with double washing appeared to approach a minimum when the time ratio of first to second wash was about 3:2. In the separation tests, the relative quantities of high-purity fractions obtained from the separator were much greater than those from the curb because of the mixing that took place in the separator chamber. As a result, the average difference between the purities of the two separator fractions ( 6.2 units) was much smaller than that between the corresponding curb samples (19.7 units).

## Effect of trash grinding on cane calculations

R. R. Tamaye. Proc. 41st Conf. Hawaiian Sugar Technologists, 1982, 140.
Two processing schemes involving trash are considered: (1) where the trash is prepared and milled together with the cane, and (2) where the trash is milled separately and the expressed liquid used as additional imbibition for cane milling. For the first scheme, in which the fibre load on the mill will be greater as will the amount of liquid to be expressed (because of entrained water from the cleaning plant), the easiest method of calculating mill performance is probably that based on the weight of analysis of mixed juice and analysis of prepared cane and bagasse: 100 $\left(\frac{\text { pol \% bagasse x fibre \% prepared cane }}{\text { pol \% prepared cane x fibre \% bagasse }}\right)$ $\times 100$. This method is applicable at about
half the sugar factories in Hawaii, whereas at the others more reliable estimates of trash in prepared cane and of fibre in trash will be required, necessitating periodical sampling and analysis of the cane entering the mill, at least until establishment of a data base which might allow the use of less rigorous methods of estimation. Since the moisture from the cleaning plant cannot be distinguished from that within the cane, the absolute juice factor will have to be determined empirically (since the absolute juice in prepared cane will not be equivalent to absolute juice in net cane). With the second scheme, extraction is given by: $\frac{\text { pol in mixed juice }}{\text { pol in cane }+ \text { pol in trash }} \times 100$, where cane and trash milling are considered as a single operation. However, if calculations are also to be made for the two milling operations separately, more measurements will be needed, including the weight and analysis of the liquid expressed by the trash mill, weight and/or analysis of two of the three bagasse streams (cane, trash and cane + trash) and analysis of the incoming cane and trash; the equations used will depend on the basic data available.

## A review of the causes of the spontaneous decomposition of massecuites and molasses

## R. T. Webb. Proc. 41st Conf. Hawaiian Sugar Technologists, 1982, 141-143.

A brief survey is presented of reports of, and theories on causes of, massecuite and molasses spontaneous decomposition. From analysis of the various accounts, it is suggested that the decomposition reactions occur continually during juice processing but that the degree and rate of decomposition are not normally noticeable until several of the following conditions occur: (1) the material is concentrated to $75^{\circ} \mathrm{Bx}$ or more after two or more boilings, (2) reducing sugars are present at a critical concentration which is governed by the type and amount of other impurities present, (3) amino-acids are present at a critical concentration which may be a
function of reducing sugars content, (4) the material is sufficiently hot for the exothermic reaction involved to be initiated or accelerated, (5) heat from the reaction cannot escape as fast as it is generated, and (6) there is a sufficient mass of reactants present to accelerate the reaction. Since the only controllable item under normal processing and storage conditions is (4), storage temperature is considered to be the major and primary control criterion for molasses; in Hawaii, $40.5^{\circ} \mathrm{C}\left(105^{\circ} \mathrm{F}\right)$ is regarded as the highest acceptable storage temperature for final molasses, since at this temperature the chemical reactions are slow enough to be controlled by circulating and cooling the molasses with pumps or compressed air. The author is of the opinion that production or economic constraints that require the inert storage for long periods of hot, high-density, impure products in insulated vessels provide ideal conditions for initiating the explosive decomposition of massecuite or molasses.

## Dextran investigations at Hilo Coast Processing Company

C. Morks. Proc. 41st Conf. Hawaiian Sugar Technologists, 1982, 144-147.
Determination of dextran in 1st expressed juice and mixed juice at factories owned by Hilo Coast Processing Co. (HCPC) showed that the amount of dextran entering the factories with the harvested cane was on average nearly four times as high as the additional dextran formed in the milling station. Raw sugar analyses for polysaccharides and dextran are tabulated for 27 shipments received at Crockett from Hawaii in 1982, and show a maximum dextran content of 149 ppm , while most of the contents were well below 100 ppm , and eleven values were in single figures. However, while these results would indicate that Hawaiian raw sugar is well within the limit of 250 ppm dextran above which some refiners exact a penalty, it is pointed out that most Hawaiian raw sugar cargoes are mixed and that the dextran content rose with increase in the proportion of sugar emanating from Hilo, $45-50 \%$ of
which sugar was produced by HCPC. The dextran levels were found to be very much higher at Pepeekeo, Papaikou and Wainaku factories. Despite remedial measures, the problems have remained, and it is considered important to improve the harvesting procedures, since enzymic treatment will reduced the problem in processing. A major problem is the high rainfall in the Hilo Coast region - this coupled with any delay in cane delivery has been found in Australia to lead to high dextran levels.

## Material selection for pumping applications

H. Gellert. Proc. 41st Conf. Hawaiian Sugar Technologists, 1982, 154-156.

Details are given of types of white iron used for abrasion resistance, with particular reference to their use in slurry pumps. Mention is made of a pump made of $28 \%$ chrome-iron which was used to recirculate cane cleaner water; after 8 years' operation there was no detectable loss of metal from the casing or impeller, while a chromium oxide, ceramic-coated shaft sleeve installed after the first season looked like new. Comparison of the economics of a pump made of chrome-iron with one made of NiHard 1 chrome-nickel (widely used in the Hawaiian sugar industry but very brittle and of low tensile strength) over 8 years shows that the total costs of the former pump are less than half those of the latter pump when allowance is made for replacement of various components.

## Plate-type exchangers

N. L. Lucas. Proc. 41 st Conf. Hawaiian Sugar Technologists, 1982, 157-159.
Applications of plate-type heat exchangers at two Hawaiian sugar factories are described. At Lihue, they are used to cool low-grade massecuite to $125^{\circ} \mathrm{F}$ and to reheat it to $130-135^{\circ} \mathrm{F}$ before spinning. The heat exchangers, complete with hollow elements and rotating stirrer arms, are mounted in batch crystallizer shells. At Paia, the heat exchangers are used for massecuite reheating. Performances at both
factories are discussed. At Lihue heat transfer is excellent, and the cooling time of $61 / 2$ hours compares with more than 12 hours in coil-type crystallizers. Some design problems have occurred. At Paia heat transfer has been excellent with no measurable purity rise during reheating with water at $134-136^{\circ}$ F. Again, some design problems have been encountered. A short résumé of crystallization technology demonstrates the requirements of good crystallizer operation and the advantage of good heat transfer.

## Economics of hydraulic cranes at Hilo Coast Processing Company

## J. F. Roney. Proc. 41st Conf. Hawaiian

 Sugar Technologists, 1982, 190-193.At HCPC, about 7.2 million gallons of soil-laden water from the cane cleaning plant is pumped to land containment every day. The soil is removed by settling in 22 ponds, while the effluent is discharged into the sea. When the ponds are filled with mud they are dredged by excavator (hydraulic crane) or by cable crane with a clamshell bucket. The performances and economics of the two types of crane are compared and tend to demonstrate the superiority of the hydraulic crane.

## Some microbial aspects and control measures of deterioration of harvested canes

R. L. Samaniego and A. T. Angeles. Crystallizer, 1983, 6, (2), 11, 16-17.
The findings and observations of a number of authors on harvested cane deterioration causes, effects and reduction are summarized.

## The implications of an early milling campaign on productivity

R. R. Covar and L. J. Tolentino. Crystallizer, 1983, 6, (3), 7, 9, 14-15.
While some sugar factories in the Philippines in the past have offered financial incentives to growers to supply cane early, there are a number of factors, particularly weather and cane immaturity,
that discourage farmers from following the practice; if there is inadequate cane, a factory starting operations early will be faced with the problem of higher unit processing costs and a lack of fuel in the form of bagasse. The situation is discussed with the aid of graphs and calculations.

## Finger scraper

G. Lustre akd J. Panganiban. Crystallizer, 1983, 6, (3), 11.
The advantages of the finger-type scraper over the conventional type of mill scraper are discussed on the basis of tests during 1981-82 in which a finger-type scraper was installed on the 1 st mill top (Lotus) roller and had a beneficial effect both on milling and on the life of the roller. Since then, the remaining scrapers on the mill have gradually been replaced. The finger-type scraper can be made in a factory machine shop.

## Bacterial fermentation - a cause of sugar loss in the mill

R. A. Johnson. BSES Bull., 1983, (4), 15.

While about 50 different micro-organisms occur in green cane, the only ones of any apparent importance that can survive burning of the cane are Leuconostoc mesenteriodes and Lactobacillus spp. These survive by colonizing growth cracks in the cane and are well placed to enter the stalk at harvesting, either through fractures produced by the harvester blades or through the cut ends of the billets. A Bacillus sp . is the commonest soil microorganism to be picked up by the harvester. Both L. mesenteroides and Lactobacillus spp. produce lactic acid as a major or sole product of fermentative sucrose metabolism - most Lactobacillus spp. produce lactic acid only (at an approximate rate of one part acid to two parts sucrose destroyed), while $L$. mesenteroides produces both lactic acid and dextran; the Bacillus sp. is responsible for the production of a complex mixture of proteins and amino-acids. $L$. mesenteroides is the predominant bacterium in conventional milling trains,
where it prefers lower temperatures and so grows in cooler pockets of the juice stream, while the other two micro-organisms mentioned prefer higher temperatures and form small colonies at the "hot"end of the train. However, massive infections by all three types, particularly Lactobacillus spp., may occur in diffusion if the operating temperatures are allowed to fall within the optimum range for their growth $\left(60-70^{\circ} \mathrm{C}\right)$. The normal life cycle of a bacterial colony has four distinct phases: a lag phase of little growth during which the - bacteria adapt themselves to the environment, an exponential phase of rapid growth and population increase, a stationary phase where the growth rate falls because of decrease in nutrient - availability and accumulation of poisonous metabolic waste products, and a death phase in which the population returns to its original level. In diffusers, under suitable temperature conditions, bacteria may be regarded as in a permanent exponential phase, since there is a continuous supply of nutrients in the form of shredded cane and continuous removal of fermentation waste products, e.g. lactic acid. Ion chromatography and microscopy revealed that sucrose losses in a diffuser in a Queensland factory were due to fermentation by Lactobacillus spp. The problem was quickly solved by installing an extra heater at an appropriate position in the juice stream.

## Measures of cost reduction in the - sugar industry

P. S. Narayana.SISSTA Sugar.J., 1983, 9, (3), 6 pp .

Factors relating to the economics of sugar \& production in India are discussed with the aim of demonstrating how to increase profitability in both cane agriculture and processing.

[^1]the crown wheel was removed from the discharge roller operated as a self-setting mill and gave better compression of the cane blanket, higher juice flow over the discharge roller and a marginally lower bagasse moisture content. Future possibilities are discussed, as is the question of the importance of the feed:discharge opening ratio compared with the escribed volume of the discharge roller.

## Cane juice demineralization by ion-exchange

G. Kasinathan. SISSTA Sugar J., 1983, 9, (3), 3 pp .

Clarified juice ion-exchange treatment is described and its advantages listed. Effluent disposal, the benefit of regeneration with HCl rather than $\mathrm{H}_{2} \mathrm{SO}_{4}$, and ammonia recovery are among the aspects examined.

## Evaporators - a few design and operational aspects

S. K. Ghosh. SISSTA Sugar J., 1983, 9, (3), 10 pp .

Factors discussed include material used for the heating surfaces, tube pitches, removal of incondensable gases, the adverse effect on heat flow of dropwise and filmwise condensation and of the juice film at the internal wall of the tube, the advantages of long-tube evaporators, and entrainment.

## Stabilizing the process of sugar manufacture by introducing automation and instrumentation

P. V. L. Narasimhan. SISSTA Sugar J., 1983, 9, (3), 5 pp .

Instrumentation and automatic control of cane mills, liming and pH control, juice heater temperature control, continuous on-line Brix measurement, evaporator and vacuum pan control, control of molasses dilution and of crystallization are discussed.

## Microprocessor control of low-grade station

M. Blain, N. E. Woodford and
N. G. Skippen. Sugar J., 1983, 46, (5), 13-16.
See I.S.J., 1983, 85, 334-337.

## Determining the optimum locations and sizes of sugar cane processing plants

G. Chadwick. Sugar y Azúcar, 1983, 78, (11), 31-32, 35.

A plant size and location model that minimizes processing and transport costs by means of non-linear separable programming is described. The model is formulated as a matrix, an example of which is reproduced and its use explained. Possible applications of the model are indicated; the author has used it to evaluate the effects of transport cost subsidies on the South African sugar industry.

## Fly ash collection for bagasse-fired boilers at Shakarganj Mills Ltd., Jhang

M. Tufail. Proc. 14th Ann. Conv. Pakistan Soc. Sugar Tech., 1977, 192-203.
Descriptions are given of different devices available for fly ash collection, and the system used at Shakarganj Mills Ltd. is described. This incorporates separation of bagasse particles in the superheater zone, which acts as a settling chamber for the heavier particles, while the semi-burnt particles are re-injected into the furnace. An inertial separator removes dust particles from the flue gases entering the air preheater, while final cleaning of the gases is effected by a pair of small-diameter cyclones.

## Continuous cooling crystallization

R. A. Khan. Proc. 14th Ann. Conv. Pakistan Soc. Sugar Tech., 1977, 226-231.
Advantages of connecting six Blanchardtype crystallizers in series for continuous cooling of low-grade massecuite at the author's sugar factory are indicated by comparison of results of one season's continuous operation with those of the previous season's batch operation.

## Beet sugar manufacture

## Vessel for beet cossettes prescalding with saturated steam

I. A. Oleinik, A. V. Sadych,
A. A. Ivashkevich, N. G. Solovov and
I. S. Tkach. Sakhar. Prom., 1983, (11), 21-23 (Russian).
Details are given of a prescalder in which saturated steam is brought into direct contact with the cossettes. Its use in combination with a sloping-trough diffuser is outlined. The unit is in the form of a vertical cylinder separated into three horizontal sections in each of which steam at $191^{\circ} \mathrm{C}$ and a pressure of 0.105 MPa is fed through perforated screens. Cossettes at an hourly throughput of 125 tonnes and a density of $500 \mathrm{~kg} \cdot \mathrm{~m}^{-3}$ form a $160-\mathrm{mm}$ layer on the screens. Blades attached to a central axis and rotating at an optimum of 600 rpm transfer the cossettes from section to section and finally into the diffuser. The rated performance of the prescalder is a temperature increase from $15^{\circ} \mathrm{C}$ to $68-70^{\circ} \mathrm{C}$ during 20 seconds' residence. Comparison with conventional prescalding using juice showed that the new system gave a juice purity 1.2 units higher, and the albumin, total N and ash contents in the exhausted cossettes were also higher.

## Behaviour of albumins in lime treatment of raw juice

L. P. Reva, G. A. Simakhina and
V. M. Logvin. Sakhar. Prom., 1983, (11),
24-25 (Russian).

Juice samples from beet stored for different periods and having purities of 85.8 and 76.4 , respectively, were subjected to preliming at $60^{\circ} \mathrm{C}$ for 10 min , liming at $85^{\circ} \mathrm{C}$, carbonatation and filtration. The concentrations of albumins and their degradation products were determined at the different process stages by precipitating the high-molecular fraction with trichloroacetic acid and filtering, and determining the albumins in the residual fraction by the colorimetric method described earlier ${ }^{1}$. Results showed that $90-97 \%$ of the high-molecular albumins were removed by purification, while
$50-60 \%$ of the degradation products (having a M.W. below 6000) were removed. However, prolonged storage of beet led to increase in the albumin degradation products and hence to reduction in albumin removal efficiency as well as increased solubility of predefecation mud in main liming.

## An automatic control system for a direct-flow film evaporator

B. A. Eremenko, K. F. Gerbut,
A. I. Tsenzura, P. Yu. Lavrov and
V. P. Rudnev. Sakhar. Prom., 1983, (11), 34-36 (Russian).

Details are given of the automatic control scheme for the quintuple-effect film evaporator at Yagotin sugar factory, which regulates juice feed to each effect, thick juice Brix (on the basis of dilution with sulphitation juice when the Brix is too high), steam pressure in the 2nd effect, and juice level in all effects, as well as condensate feed and adjustment of juice concentration with water in the feed tank before the evaporator. Provision is also made for automatic injection of make-up juice when feed is inadequate.

## The situation regarding and prospects of introducing thyristor sets in sugar factories

M. V. Moskaev. Sakhar. Prom., 1983, (11), 36-37 (Russian).
The benefits of thyristor drives are briefly indicated, and applications to sugar factory equipment and possible future trends described.

## The heat economy of diffusiondefecation system.

A. I. Khomenko. Sakhar. Prom., 1983, (11), 42-47 (Russian).

A comparative evaluation is made of the heat economy of diffusion and juice purification systems using different types of diffuser; the merits of slopingtrough diffusers are particularly extolled. It is shown that recycling 1st carbonatation juice instead of mud to preliming has an adverse effect on diffusion while also
increasing reheat steam consumption, since pan vapour or condensate is not suitable for reheating a juice mixture having a temperature greater than $60^{\circ} \mathrm{C}$. Means of reducing steam consumption are indicated.

## Calculation of pressure reduction in the vacuum system of a sugar factory

V. N. Gorokh, B. F. Us and K. O. Shtangeev. Sakhar. Prom., 1983, (11), 47-48 (Russian).
It is shown that calculation of pressure fall in a vacuum system having a considerable number of local resistances should allow for the effect of steam compressibility. Relevant equations are presented for calculation of pressure loss in a vacuum system and highly expanded steam lines where steam flow velocities are high.

## Effect of cossette liming on diffusion juice

E. Zaragosa, J. Randall and W. Camirand. J. Amer. Soc. Sugar Beet Tech., 1982, 21, 383-394.
Following earlier studies on the effect of liming on beet tissue ${ }^{2}$, investigations were conducted on the effects of liming on raw and thin juice properties. Results showed that treatment of cossettes with up to $1.5 \% \mathrm{CaO}$ as dry lime or up to $4 \% \mathrm{CaO}$ as lime slurry had a positive effect in reducing juice colour from at least 220 ICUMSA units to generally 12.1-20.2 units, giving a juice that was clear, light and straw-coloured in contrast to a greyishblack, turbid juice from untreated cossettes. The colloid content was also reduced by liming, so that it would be possible to treat raw juice, after settling, by ion exchange without need for carbonatation. A slightly lower purity of thin juice from limed cossettes was attributed to methanol and acetate generated by the liming reactions, but methanol should volatilize during evaporation, while acetate has negative melassigenic properties. Juice Brix, pH and lime salts content were little affected by
1 Reva \& Simakhina: I.S.J., 1979, 81, 92.
2 Camirand et al.: ibid., 1982, 84, 374.
cossettes liming.

## An impeller pump for undiluted carbonatation mud and other highly viscous fluids of high abrasive particle content

J. Torzecki, M. Dziubinski, H. Fidos and J. Sek. Gaz. Cukr., 1983, 91, 145-147 (Polish).

Details are given of a Polish PW2 impeller pump, the prototype of which has undergone large-scale testing in a sugar factory. Designed to handle materials such as undiluted carbonatation mud of up to $1700 \mathrm{~kg} / \mathrm{m}^{3}$ density, it has a rated hourly delivery of $20 \mathrm{~m}^{3} / \mathrm{hr}$ at 1410 rpm ; the motor is rated at 13 kW .

## Tests on the effectiveness of new types of industrial filter cloth for a sugar factory

J. Haszczynska. Gaz. Cukr., 1983, 91, 147-151 (Polish).
Twelve different filter cloths were tested at four Polish sugar factories in rotary vacuum filters and filter-thickeners; seven were made of polyamide fibres, one was a polyamide-cotton mixture, two were of polyester and two of polypropylene. Full details are given of the properties of the cloths and of their performances over periods extending from 8 to 22 days.

Risk of dust ignition and explosion from mechanical sparks in a sugar - factory
S. Kabat. Gaz Cukr., 1983, 91, 152-154 (Polish).

It is stated that 20 out of 47 cases of dust -ignition and explosion in the world's food industry ( 7 out of 14 in Poland) during the period 1960-79 were caused by sparks resulting from friction or impact. Conditions under which sparks may be emitted and the risk of fire and explosion heightened are discussed generally, and the specific case of sugar and beet pulp dust examined. The chief areas of fire and explosion hazard are indicated, and guidance is given on preventive measures.

## Erection of the sugar factory at Ropczyce

S. Ginal. Gaz. Cukr., 1983, 91, 155-158 (Polish).
A dj scription is given of the factory erected at Ropczyce in the southeast corner of Poland. Planned for a daily slice of 6000 tonnes of beet, with facilities for post-campaign processing of stored thick juice, the factory is the largest in the Polish sugar industry and represents the largest investment of any food enterprise to date in Poland. Details are given of the general layout of the buildings and of the equipment and its suppliers (including many manufacturers of specialized equipment outside Poland). Information is given on the processing scheme, which includes conventional 3-massecuite boiling.

## Assessment of the storage properties of sugar beet varieties

J. Trzebinski and Z. Sadoch. Gaz. Cukr., 1983, 91, 208-211 (Polish).
Results are discussed of storage tests on $\mathrm{AJ}_{3}$, Polycama, Monohill and PN-mono 1 beet varieties, samples of which were stored for up to 60 days at $4^{\circ} \pm 2^{\circ} \mathrm{C}$, and their weight and sugar losses determined. Results are tabulated.

## Methods of beet handling in British Sugar factories

D. Horsley. British Sugar Beet Rev., 1983, 51, (4), 24-27.
While much of the beet crop is delivered to UK sugar factories by haulage contractors who seek to minimize the turn-round times of their trucks, the processors are interested in safe, controlled unloading so as to minimize damage to the beets. Not only does damage to beet mean losses in sugar but also contamination of flume water, which must be considerably purified before it meets the stringent requirements for discharge into rivers. The amount of beet delivered by tipper and non-tipper trucks is generally in the ratio $65: 35$, with variation from area to area. The beet is washed from the non-tippers during the day and fed directly into the factory, while
that from the tippers is unloaded into storage for use at night and at the weekend. A small excess of beet from the non-tippers is accumulated in a wet store during the day and then fed to process as soon as possible after the end of the day's beet reception so as to minimize the loss of sugar resulting from leaching. Too high a proportion of nontippers would create problems in the form of time taken to unload the beets (a non-tipper may remain at least 30 minutes in the beet yard compared with only some 10 minutes for a tipper) and increased wet-stored beet and hence sugar losses. The installation of tipplers at three factories has overcome the difficulty, but drawbacks include high installation and maintenance costs and the high rate of breakage suffered by the beets as they fall into the hoppers. Elevated roads built in the late 1940's and early 1950's were designed to be used by tippers, the beets falling into the silo storage area below; recent studies have shown that tipping of beet onto beet already in the silo offers the best means of minimizing beet breakage. In the meantime, these roads have proved to be limited in their application and have needed much structural repair, so that alternative means were sought with inauguration of the major factory expansion program. These have included the polar silo to which beets from large tipping hoppers are transferred by belt conveyor. They suffer from high installation and maintenance costs and a considerable degree of beet breakage. A flat concrete pad constructed at Brigg and first used in the 1981/82 campaign has a capacity extended in 1982 to 3000 tonnes. A self-propelled piling vehicle stacks the tipped beets. The system has a number of advantages, and tests showed that the amount of beet breakage was not significantly different from that experienced with the elevated road at the factory, while it was less than with a wet silo. The pile should be no higher than 3 m so as to avoid damage caused by crushing. A test has been carried out to establish the rate at which beet can be unloaded onto the pad.

## Sugar refining

## Simulation of a sugar refinery

L. Hernández C. CubaAzúcar, 1983, (Feb, special number), 34-39 (Spanish).
A simulation program has been developed for calculating material and energy balances in a sugar refinery. It has been developed for a particular technology but allows a certain degree of flexibility for e.g. inclusion or not of raw sugar affination, heating of treated liquor, etc., and can be used in the analysis of bottlenecks, calculation of capacities, etc.

## Elimination of colouring matter on calcium phosphates

R. Fajardo G., J. Hernández, H. Estacio C., C. Noriega P. and L. D. Bobrovnik. Centro Azúcar, 1982, 9, (3), 97-112 (Spanish).
X-ray, U.V. and visible spectroscopy and gel filtration were used in the study of adsorption of colouring matter from raw sugar solution on calcium phosphate precipitates. From analysis of the isotherms concerned it is concluded that adsorption is irreversible and also that it is not selective, since the same types of colouring matter were found in both raw and clarified liquor.

## The new sugar house at Tirlemont refinery

Anon. Zuckerind., 1983, 108, 855-860 (German).
See Braeckman: I.S.J., 1984, 86, 54.

## Equations for calculations of sugar solution viscosity

L. A. Sapronova. Sakhar. Prom., 1983, (10), 34-38 (Russian).

Data from the literature on the viscosity of white sugar solutions of 46 and $75.6 \%$ solids content and pure sucrose solutions of 30 and $65 \%$ solids content at $20-70^{\circ} \mathrm{C}$ in $10^{\circ}$ intervals have been used (together with a few values obtained by the author) in the construction of a graph of log viscosity vs. molar concentration. Two equations have been derived for
calculations in the refinery, one equation applying to unsaturated solutions and the other to saturated solutions.

## Entrainment separators for pans and evaporators

D. M. Humm. Proc. 4lst Conf. Hawaïan Sugar Technologists, 1982, 120-129.
After brief discussion of factors contributing to entrainment, the author describes separator design principles and the operating characteristics of centrifugal and baffle-type separators as well as mesh pads. Details are then given of a test program at Crockett refinery involving zigzag baffle-type separators and wire mesh pads (centrifugal separators being omitted because of their high cost of installation in the domes of existing pans and because of a fear that their high pressure drop could overload the vacuum system). A Technicon Monitor IV AutoAnalyzer was used for continuous measurement of condensate sugar content. Results indicated that during the concentration stage in boiling, $<2 \mathrm{ppm}$ sugar occurred in the condensate compared with $10-30 \mathrm{ppm}$ when no separator was used; however, high sugar contents were occasionally noted during the final boiling stages when the massecuite level was too high and re-entrainment took place. A distance of at least 4 ft between massecuite and baffles is therefore recommended for high-purity strikes, but at Crockett this requirement would interfere wih normal boiling and cause a reduction in pan capacity, so that installation of normal baffle-type separators was considered inadvisable. A dense-bed zigzag baffle-type separator reduced the condensate sugar to 5 ppm or below (in most cases $<2 \mathrm{ppm}$ ) under a wide range of conditions, including very high steam rates. Two types of wire mesh pads gave a condensate sugar consistently $<2 \mathrm{ppm}$. Baffles installed in the domes of remelt pans reduced the condensate sugar to $<2 \mathrm{ppm}$ in most cases, compared with $3-5$ ppm without baffles when remelt strikes were boiled and 20.50 ppm when thin sweet-water was being concentrated. Baffles installed in the three evaporators
markedly reduced condensate sugar. Guidelines are given for selection and installation of entrainment separators.

## Automated sugar refinery has sweet smell of success

W. R. Willis. Control Eng., 1983, 30, (7), 138, 140; through S.I.A., 1983, 45, Abs. 83-1551.
Everglades Sugar Refinery lnc., Florida, USA, has a throughput of 350 million 1 b sugar/year. The refinery process includes carbonatation and bone char treatment. Five General Electric Series Six Model 60 programmable controllers have been installed in the past 18 months, to replace large numbers of relays, timers and switches, and more are to be put in. Operation is very satisfactory, and only five people per shift are required for supervision.

## Vibratory screen for refined sugar

J. Hluze. Listy Cukr., 1983, 99, 227-231 (Czech).
A description is given of a Czechoslovakian vibratory screen for refined sugar, and its design theory is analysed. The dynamics of the system with and without damping are evaluated mathematically.

## The quality of water used at refineries for process purposes

N. I. Yakimenko and S. A. Brenman. Sakhar. Prom., 1983, (11), 26-28 (Russian).
The chemical composition of raw water used for refining processes can add considerably to the non-sugars removal problem unless the water is first demineralized. If the water is exceptionally hard and has a high mineral content it is preferable to use condensate or apply demineralization. However, no standards have yet been laid down for the quality of water used in Soviet refineries nor has any outline scheme been established for its treatment, although the authors do recognize that the requirements will be at least comparable to those of drinking water.

## Laboratory studies

## Biometry of particles of bagasse and pith

M. Antigua. Revista ICIDCA Suppt., 1983, (2), 7 pp (Spanish).

A method, suitable for occasional research or assay purposes but not intended for routine control, involves the screening of bagasse with Nos. 5, 12 and 100 screens to separate fibrous and parenchymatous material from pith, weighing and then separating the components of the different fractions using tweezers and a micro-film -eader, and measuring the lengths, etc., determining standard deviations of the dimensions, etc.

## Energy of activation of crystal nucleation

R. Ts. Mishchuk, L. G. Belostotskii and S. I. Sagan'. Sakhar. Prom., 1983, (10), 28-30 (Russian).
Knowledge of the crystal nucleation process as obtained from investigations conducted by various authors going back to 1932 is summarized, and the activation energy shown to be a function of temperature and Brix, with the probability of new crystal formation increasing with temperature rise. With increase in supersaturation, the probability is displaced to the region of lower concentration.

## Growth studies of yeast in refined syrups at various levels of solids and nutrients

M. Bolinder. J. Amer. Soc. Sugar Beet Tech.. 1982, 21, 345-350.

Investigations conducted on yeast growth in syrups are reported. Prepared blends of \& sucrose with $50 \%$ invert syrup, 62 DE corn syrup and high-fructose corn syrup were inoculated with Saccharomvces rouxii ATCC 22027 and an unidentified yeast isolated from fermenting corn syrup; yeast nitrogen base (YNB), containing all the essential nutrients and vitamins required for yeast cultivation (but no carbohydrate) was also added. Yeast growth under controlled conditions was monitored and found to be closely related to the solids
and nutrient levels, no growth occurring in any blends having a solids content greater than $77 \%$; at lower solids levels, yeast growth was more rapid in the presence of added YNB. In experiments using dilutions of $50 \%$ invert syrup of $68.77 \%$ solids to which YNB was added, yeast growth decelerated with increase in solids content, from 4 days required to develop 1 ml of gas at $68 \%$ solids to more than 34 days at $77 \%$ solids. The time required to develop 0.5 ml of gas was governed by the amount of YNB added to a highly refined HFCS, extending from 2 days with 100 and 500 ppm YNB to more than 5 months in the presence of up to 10 ppm YNB. Water activity ( $A_{W}$ ), approximately given by (moles of water)/(moles of water + moles of solute), is considered a reliable guide to the risk of syrup spoilage by yeast, since it defines the effective concentration of water as a reactant. From the results of the experiments, it is concluded that a syrup may be considered safe from deterioration if the $\mathrm{A}_{\mathrm{W}}$ is $<0.80$ or if it contains $<60$ ppm nitrogenous nutrients.

## Conversion of refractometric dry substance into real dry substance for Quentin molasses

G. Sgualdino, G. Vaccari and
G. Mantovani. J. Amer. Soc. Sugar Beet Tech., 1982, 21, 395-401.

The dry solids content of beet molasses as determined refractometrically by the $1: 1$ dilution method has a value that is always higher than the dry solids value obtained by Karl Fischer titration (considered the true value); while the difference is generally about 1.5 units and this value could be used as an approximate correction factor for normal molasses, in the case of molasses that has been treated by the Quentin ion-exchange process the difference varies considerably as a function of the $\mathrm{Mg}^{++}$content. Experiments were carried out to establish a suitable correction factor; they involved adding KCl and $\mathrm{MgCl}_{2}$ to molasses samples in increasing amounts in the range $2.10 \%$ anhydrous salt on molasses, and determining the dry solids by refractometry and Karl

Fischer titration. Regression analysis of the results yielded an equation: $y=0.01643 x$ $+1.6387(r=0.992)$, where $y$ is the difference between RDS and the Karl Fischer value and $x$ is the Mg content (meq \% RDS). For molasses purity, the correction is given by $y=0.0123 x$ $+1.1363(r=0.977)$, where $y$ is the difference between the Karl Fischer and refractometric purities.

Methods of dextran determination to aid cane sugar recovery and quality
P. J. Kemp. Proc. 41st Conf. Hawaiian Sugar Technologists, 1982, 148-153.
Descriptions are given of methods for dextran determination, including the CSR alcohol haze method of Nicholson \& Horsley ${ }^{1}$ and as adapted by Chen et al. for use with a spectrophotometer (employing a wavelength of 700 nm and a cell path length of 2.35 cm ), the SPRI method in which the dextran is selectively precipitated with alkaline copper sulphate ${ }^{2}$, and a modification of this developed by Steimel in which use of a centrifuge is omitted. Brief mention is also made of the procedure using an enzyme electrode developed by Riffer at California \& Hawaiian Sugar Company ${ }^{3}$. Use of biocides to prevent formation of dextran and of enzymes to remove it is briefly discussed.

## Use of indigenous kieselguhr as stationary phase in thin-layer chromatography. Separation of sugars

E. Moraru, T. Hodisan and C. Sarbu. Rev. Chimie, 1983, 34, (2), 144-147; through Abs. Roman. Sci. Tech. Lit., 1983, 19, (2), Abs. XIX 11.

The results obtained in the separation of certain sugars (galactose, sucrose, maltose, fructose, raffinose, glucose and lactose) using indigenous kieselguhr as a stationary phase are presented. The comparative results obtained with Silicagel R demonstrated the possiblity of its use in thin-layer chromatography.
1 J. Agric. Food Chem., 1959, 7, 640-643. 2 Roberts: I.S J., 1983, 85, 10-13. 3 ibid., 131-136.

## Some aspects of colour formation in sugar manufacturing

A. Abou-Elela, F. A. Adam and
M. T. El-Haty. Sugar News (Philippines), 1983, 59, 132-137.
Various analytical methods were used in investigations of colour formation during raw sugar manufacture. Cane juice samples from two Egyptian factories were used, and experiments were also conducted on reactions of glucose, glucose-glycine, fructose and fructose-glycine in the presence of lime and/or amino-acids. The roles played by quantity of lime, temperature and period of liming on browning are examined, and the effects of juice quality and amino-acid concentration on melanoidin formation discussed.

## Sugar analysis made easy with HPLC

W. P. P. Abeydeera. BSES Bull., 1983, (4), 6.7.

High-performance liquid chromatography is explained, its advantages over conventional methods of sugar analysis are listed, and its applicability to determination of sucrose inversion rates and of the rates of decomposition of fructose and gluccose during factory processing (so as to minimize their losses and thus increase the potential recovery of molasses sucrose) is indicated. (See also I.S.J., 1983, 85, 300-306.)

## Analysis of sugar for tetrachlorodibenzo-p-dioxin.

N. C. A. Weerasinghe, J. L. Meehan, M. L. Gross and J. Gaines. J. Agric. Food Chem., 1983, 31, 1377-1378.
Both Silvex and 2,4,5-T (used as herbicides in cane fields) contain tetrachlorodibenzo-$p$-dioxin (TCDD), and a pilot study was undertaken on samples of white and raw sugar, molasses and animal fodder (containing low-grade molasses) from a Louisiana sugar factory to see if any TCDD occurred in the manufacturing process. The factory received cane grown in Silvex-treated fields. TCDD is highly toxic to animals and may be hazardous to human
health. Results of gas chromatography/ high-resolution mass spectrometry showed that no TCDD was detected in any of the samples at a detection limit of $0.5-1 \mathrm{ppt}$ (average $0.7 \pm 0.3 \mathrm{ppt}$ ) at an average recovery of $63 \pm 13 \%$. However, TCDD was readily detected in method validation samples to which 0.30 and 0.60 ppt had been added; mean accuracy was $67 \%$. It is considered that the number of samples analysed was insufficient for the study to be conclusive regarding TCDD occurrence, and future investigations should be carried out with a larger number.

## Studies on floc formation of raw sugar in Taiwan

H. T. Cheng, H. C. Tseng, W. F. Lin and C. S. Ting. Rpt. Taiwan Sugar Research Inst., 1983, (100), 65-79 (Chinese).

Research was carried out to determine the nature of the material in granulated raw sugar that causes floc in bottled beverages. The various types of organic, inorganic and microbial floc and haze that appear in bottled beverages containing cane sugar are defined, differentiated and discussed. Numerous investigations of floc were aimed at predicting whether a particular sugar would flocculate. A rapid method for testing raw sugar flocculation has been developed and some 4000 raw sugar samples have been checked; however, no raw sugar in Taiwan has formed a cottony floc. Because of different soil and climatic conditions, considerable difference was found in floc formation. Polysaccharide and protein constituents in raw sugar were found to be important factors in floc formation. Details are given of raw sugar floc properties, isolation and analysis.

## Some thermodynamic heat coefficients of sugar solutions

D. E. Sinat-Radchenko. Sakhar. Prom., 1983, (12), 20-22 (Russian).
Values of the isobaric thermal expansion coefficient (characterizing the change of specific volume and used in calculations of heat transfer under conditions of free convection and forced laminar flow of sugar solutions) were obtained as a function of temperature in the range
$0-140^{\circ} \mathrm{C}$ and dry solids up to $90 \%$. Graphed results showed that the curves tended to converge with increase in the solids content, as did curves of elasticity determined at constant volume and temperatures in the range $0-90^{\circ} \mathrm{C}$. Also determined was the coefficient of isothermal compressibility, characterizing the reversible relative reduction in volume with pressure rise and applicable to calculations involved in concentration by reverse osmosis.

## Contribution to complete calculation of the crystallization rate of sucrose

D. Schliephake and B. Ekelhof. Zuckerind., 1983, 108, 1127-1138 (German).
A series of experiments was conducted on determination of the crystallization rate in sugar solutions of $60,70,80,90$ and 100 purity. The apparatus used was as described earlier ${ }^{1}$ in which crystal growth took place in an upward flowing solution, whereby the crystals fell at an accelerating velocity as their size increased. The height of the suspension fell as the crystals grew and was measured by an ultrasonic system in which the transmitted signal was made up of the sound absorbed by the suspension and that reflected by the crystals, and thus represented a measure of flow rate. Samples of the crystals were taken at intervals and photographed on a glass plate; enlargement of this photograph permitted the projection surfaces of individual crystals to be determined using a particle analyser. Details are given of the literature sources used to establish solubility (and hence supersaturation), dry solids, viscosity and diffusion coefficient of the test solutions. The theoretical fundamentals of crystallization kinetics and the method used to calculate the rate coefficients of mass transfer and of the crystal surface reaction are explained, and non-dimensional representation of mass transport demonstrated. For representation of crystallization rate as a function of purity, supersaturation and temperature, three-dimensional graphs have been constructed, and the results are discussed. 1 Orlowski \& Schliephake: I.S.J., 1973, 75, 393.

## By-products

## Contributions to explanation of microbiological and chemical relationships in pressed pulp ensilage. I. Investigations on micro-organisms in pressed pulp

## F. Hollaus, W. Braunsteiner and

 N. Kubadinow. Zuckerind., 1983, 108, 1049-1058 (German).Factors affecting microbial growth and fermentation during ensilage of beet pulp are discussed, including pH , temperature and creation of anaerobic conditions (after
consumption of atmospheric oxygen introduced with the pulp). Details are given of methods used to identify the microorganisms in pressed pulp; those found included Clostridium thermohydrosulfuricum, Bacillus stearothermophilus, B. coagulans, Lactobacillus delbrückii, $L$. brevis, C. butyricum, C. pasteurianum, Escherichia coli and Klebsiella pneumoniae. Counts were also made of yeast cells that had formed at various points in one of the sugar factories from which the samples had emanated. The quantities of lactic acid degraded by yeast are indicated. The significance of the micro-organisms and of the use of specific disinfectants in diffusion for optimum ensilage is discussed.
Improvements brought about by continuous dosing of thermolabile dithiocarbamate instead of batch dosing of formalin in the diffuser, use of an optimum ensilage temperature, addition of molasses to the pulp and injection of $L$. delbrückii starter culture are considered. (See also
-I.S.J., 1984, 86, 24.)

## Densification: cuber versus typical pellet mill

G. B. Nelson, G. C. Nelson and H. White. Proc. 41st Conf. Hawaiian Sugar Technologists, 1982, 98-99.
The first author, chairman of Papakube Corporation, describes a unit developed by his company for extrusion of solid waste materials, including bagasse, in dense rods measuring $1 \frac{1}{4}$ inches square which are cut into lengths ranging from $1 / 2$ to 3 inches. Comparison is made with typical pellet mills.

## Biogas from filter mud

C. S. Abrigo. Crystallizer, 1983, 6, (2), 10, 12.

It is stated that an average $1 / 2$ million tonnes of filter-cake is generated annually by the 42 sugar centrals in the Philippines and could serve as substrate for methane fermentation. A study is reported in which 3 kg of diluted filter-cake to which 200 ml of activated starter had been added yielded almost 12 litres of biogas over an 8 days' digestion period, about half of the gas being produced in the first two days. Filtration of the medium after water dilution had almost no effect on the subsequent fermentation process. The calorific value of the biogas was only 12.9 kcal (where the medium had been filtered) and 12.5 kcal (without filtration) compared with theoretical values of 46.3 and 44.7 kcal , respectively, for a gas containing methane alone as combustible component. Possible reasons for the low methane content in the gas are discussed, and some recommendations are made.

## Recent dj velopments in final molasses utilization

J. D. Layoso. Crystallizer, 1983, 6, (2), 11, 19.
The possible use of cane molasses as fertilizer, nematicide, animal fodder, food additive and as stabilizer/strengthener for concrete is discussed on the basis of research conducted by various authors.

## Effect of storage time on Egyptian cane molasses quality

A. Abou El-Ela. Taiwan Sugar, 1983, 30, 151-154.
The effect of storage time on molasses quality was investigated; $200-\mathrm{kg}$ samples of molasses from Komombo raw sugar factory were stored in an open pit for 24 months, and analysis carried out every 5 months for total solids, total sugars, sucrose, aldoses, total N, cationic N, acidity, viscosity, unfermentable reducing substances and alcohol. Paper chromatography was used for separate and identify amino-acids and
sugars present in both fresh and stored molasses. Tabulated and graphed results indicate the changes that take place with storage time, resulting in a loss of fermentation efficiency. (In Egypt, cane molasses is used as raw material in a number of fermentation industries.)

## Proteins from carbohydrates: some perspectives

B. Revuz. Ind. Alim. Agric., 1983, 100, 697-701 (French).
After a brief introductory review of fodder yeast manufacture and particularly single-cell protein manufacture from e.g. beet and cane molasses and vinasse, the author outlines the three stages of yeast manufacture (preparation, fermentation and recovery and purification) and discusses certain important factors: costs, the high pollution load of the waste, the competition bj tween protein manufacture and energy recovery for the various raw materials, and the price difference between SCP and soya cake (the latter having risen in price by $200 \%$ over the last 25 years by comparison with a $500 \%$ increase in the price of SCP). In a subsequent section, the article deals with protein manufacture from manioc using Candida tropicalis to synthesize an endocellular amylose of high enzymic activity which acts on the amylose and amylopectin components of the starch, and with protein manufacture from various materials on which Aspergillus niger is cultivated.

## Alcohol from sugar beet - an Austrian sugar industry project

W. Dübell. Zuckerind., 1983, 108, 1169-1172 (German).
A project for alcohol manufacture is described which is designed for a daily output of 120,000 litres from green syrup or thick juice and which would be integrated with normal sugar factory operations. Vinasse would be concentrated and either sold as fertilizer and/or animal fodder (replacing molasses as additive to beet pulp) or burnt to yield heat which would cover a considerable part of the process energy used.

## Patents

## UNITED STATES

## Continuous centrifugal

V. Hentschel, H. Kurland, H. Schaper,
J. Schueller, G. Warner and E. Zeichner, assrs. Braunschweigische
Maschinenbauanstalt, of Brunswick, Germany. 4,308,075. October 10, 1980; December 29, 1981.

The original mother liquor separated from a massecuite in the continuous conicalbasket centrifugal 1 is of different purity from wash liquids applied higher up the basket. Suitable seals 12,15 can be fitted beneath the screen to ensure that liquids of different purity do not mix and are discharged in different zones. Slide valves 17 are then used to govern whether these liquids mix (broken arrowed lines) or are directed into separate chambers for collection (solid arrowed lines).


## Cane mill

J. P. Georget, of Denain, France, assr.

Fives-Cail Babcock. 4,310,361. December

11, 1980; January 12, 1982.
See UK Patent Application 2,026,896 ${ }^{1}$.

## Apparatus for separating cane components

S. E. Tilby and B. Vukelic, assrs. Intercane Systems Inc., of Windsor, Canada. 4,312,677. February 12, 1980; January 26, 1982.

Billet cane is brought by a metering carrier in the form of a chain-driven bucket elevator to a hopper which feeds a transport conveyor in the form of a bed made up of roller chains mounted on slats which lie between adjacent links and protrude above the surface so that they form a series of tracks along the conveyor to align the billets. These are carried to a delivery conveyor where they pass beneath guide rolls to form a single, aligned layer which is fed into the inlet of splitter station 22 to be split longitudinally in half by blade 116 . Each billet half is then conveyed to a pith milling station and an epidermis removing station where these components are separated and recovered
for later processing.
The splitter station 22 is of the known type with feeder rolls 108 and pairs of rollers 150,152 with pointed tines on their surfaces to grip the billet halves and draw them to the pith removal stations 24 . Here, as the billet halves are gripped and held against the stationary surface by the tracks 162 which pass around the pressure rollers, the pith is scooped from the inside by rollers 160 fitted with cutting blades. It and some juice falls onto the chute 184 anc so is removed, while the depithed billet halves pass downwardly between the belts 200 and 202 to stations 26 where the toothed rollers 222 and tracks 224 serve in a similar manner to those in the depithing section; here, however, the epidermis is scraped off the outer surface of the billets and directed along ducts 244 on each side while the remainder of the billets pass downwards between tracks 224 and rollers 246. They are caused to travel down through shredder discs 252 and chipper wheels 254 before falling into chute 260 and so out of the separator.

1 I.S J., 1983, 85, 187.


[^2]
# A fermentation inhibitor in sugar beet 

By D. V. Vadehra, R. Hashia, P. Mehta, N. C. Chandan and J. K. Gupta<br>(Department of Microbiology, Punjab University, Chandigarh, India)

## Introduction

The non-renewable nature of petroleum, combined with growing world-wide demand, has stimulated a great deal of interest in alternative resources of energy, particularly for internal combustion engines. Many alternatives have been suggested, of which fuels from biomass represent one of the opportunities. Ethanol produced by fermentation is being evaluated in many countries as a liquid fuel for automobiles and as a chemical feedstock.

- Of the many temperate climate crops that can be fermented only sugar beet, potatoes and maize have been suggested and used for industrial alcohol. The use of sugar beet for the production of alcohol - was first described by Mariller ${ }^{1}$.

Traditionally, beets are scalped at harvest; the leaves and the top of the root down to the lowest leaf scar, which is the most metabolically active part of the root, are removed and this in turn improves the storage ${ }^{2}$.
The extraction process in existing beet sugar factories employs a diffuser. If one is producing sugar beets for power alcohol it may be more economical to modify or replace the diffuser operation with a simpler shredding extraction method. The present work was undertaken to evaluate simpler methods for the production of sugar beet juice and its fermentation to ethanol.

## Materials and methods

Beet seed of variety Ramonskaya, procured from Punjab Agricultural University, Ludhiana, was grown on a local farm during October to April. The beets *, were cut into small longitudinal cossettes measuring approx. $6 \times 3 \mathrm{~cm}$. The juice was extracted by shearing and cutting of the pieces with the help of a standard carrot juicer. Excessive pulp was removed by pressing the juice through a muslin cloth. The pH was adjusted to 4.5 with 1 N sulphuric acid. For the preparation of juice from peeled sugar beets, the upper skin portion of the beet was removed with a sharp knife before juice extraction.

D. V. Vadehra


Ravinder Hashia

## Fermentation studies

A culture of Saccharomyces cerevisiae (strain $K_{2}$ ) procured from Haryana Agricultural University, Hissar, was used throughout this study. The culture was maintained at $4^{\circ} \mathrm{C}$ on slants comprising $0.5 \%$ yeast extract, $0.5 \%$ peptone, $1.0 \%$ dextrose and $1.5 \%$ agar. $100-\mathrm{ml}$ aliquots of seed medium $(0.5 \%$ yeast extract, $0.5 \%$ peptone and $1.0 \%$ sucrose), were sterilized in 250 ml Erlenmeyer flasks and inoculated with a 24 hr -old yeast slant culture. The flasks were incubated for 24 hr at $28 \pm 2^{\circ} \mathrm{C}$ on a rotary shaker. The fermentation broth was inoculated with $10 \%$ of the seed culture (approx. $10^{7}$ cells $/ \mathrm{ml}$ ) and incubated at $28 \pm 2^{\circ} \mathrm{C}$ for 48 hr . The synthetic medium used for comparative studies contained $10 \%$ sucrose, $0.1 \%$ $\left(\mathrm{NH}_{4}\right)_{2} \mathrm{SO}_{4}$, and $0.1 \%\left(\mathrm{NH}_{4}\right) \mathrm{H}_{2} \mathrm{PO}_{4}$. The medium was autoclaved at 10 psi after adjusting the pH to 4.5 with $1 \mathrm{~N}_{2} \mathrm{SO}_{4}$.

Physico-chemical treatments of beet juice
(a) Filtration and centrifugation: The floccular material in beet juice was removed by filtration (ordinary filter paper) and centrifugation (4000 rpm, 30 min ). The clear juice obtained was fermented to alcohol.
(b) Heat:

The beet juice was heated at temperatures ranging from 40 to $100^{\circ} \mathrm{C}$ for 10 to 120 min . The precipitated flocculum was removed by filtration.
(c) Chemical:

Alum, calcium carbonate, calcium oxide, lead acetate, potassium ferrocyanide and Triton X-100 were each added to beet juice at a concentration of $2 \% \mathrm{w} / \mathrm{v}$. The
mixture was vigorously stirred and left for 8 hr at room temperature, after which the sedimented flocculated material was removed by filtration.
The pH of the juice was adjusted to 4.5 with $1 \mathrm{~N} \mathrm{H}_{2} \mathrm{SO}_{4}$ prior to fermentation.
The floccular material obtained by the various physico-chemical treatments was added to portions of the synthetic medium at a concentration of $2 \%$ (wet weight) and fermentation was carried out for 48 hr .

## Analytical procedures

The cell count at various time intervals was determined by standard pour plate technique. Initial and residual total sugar was estimated by the phenol/sulphuric acid method ${ }^{3}$. The ethanol content was determined by the potassium dichromate reduction method ${ }^{4}$.

Fermentation efficiency and specific fermentation rate were calculated as follows:
Fermentation efficiency $=$

$$
\frac{\text { Actual ethanol recovery }}{\text { Theoretical recovery }} \times 100 \%
$$

where theoretical ethanol recovery $=$ total
fermentable sugar $\times 0.51^{*}$. Specific
fermentation rate $\mathrm{V}_{\mathrm{e}}=1 / \mathrm{ExdE} / \mathrm{dt}, \mathrm{hr}^{-1}$, where $\mathrm{E}=$ ethanol concentration, $\mathrm{g} / \mathrm{l}$.

## Results and discussion

The beet juice prepared from peeled and whole beet was compared for its ability to act as a substrate for S.cerevisiae. The alcohol yield as weil as the fermentation efficiency were higher by about $75 \%$ in juice from peeled beet than in juice prepared from whole beet (Table I). These results indicate that the method for the extraction of juice may be critical in the fermentation efficiency and that the peel contains an inhibitor. This hypothesis was tested by adding graded amounts of macerated peel to the synthetic medium.

[^3]| Table I. Alcohol fermentation of peeled and unpeeled beet juice |  |  |
| :--- | :---: | :---: |
|  | Alcohol yield, |  |
| g/litre | Fermentation <br> efficiency, $\%$ |  |
| Origin of juice | 20.54 | 30.07 |
| Unpeeled whole beet | 35.55 | 49.79 |
| Peeled beet |  |  |

The results in Figure 1 show that the pulped beet root caused inhibition of the alcohol production in synthetic medium, the extent of inhibition being concentration-dependent. Further, the nature of the pulp added had a considerable effect on the yield. Pulp from the peel and from the whole unpeeled beet had greater inhibitory properties, particularly at higher concentration, than the pulp obtained from beet of which the peel had been removed prior to pulping. At a pulp concentration of $10 \%$ the efficiency was only $3.5 \%$ and $12 \%$, respectively, compared with an efficiency of $30 \%$ in the control. These results confirmed that the higher concentrations of inhibitor are located in the superficial layers of the sugar beet. The inhibitory effect, though concentration-dependent, was obvious even at a concentration of $2 \%$, when fermentation efficiency was reduced by about $33 \%$. Growth studies of S.cerevisiae in peeled and unpeeled whole beet juice did not show any significant variation. The maximum cell count of approx. $4.8 \times 10^{8}$ cells $/ \mathrm{ml}$ was achieved in 24 hr with juice from both peeled and unpeeled beet, indicating that the inhibitory properties are specific for


Fig. 1. Effect of added sugar beet peel and pulp on alcohol fermentation in synthetic medium
alcohol production and not the microbial growth.
Since peeling of sugar beet on a large scale would be a cumbersome and uneconomical process, various physicochemical treatments were tried for improvement of the alcohol yield from the juice of unpeeled beet. The results, given in Table II, show that filtration or centrifugation of the juice improved the alcohol yield by $87.5 \%$ and $150 \%$, respectively. It appears that the inhibitory principle is equally distributed in the coarse
boiling time did not further increase the efficiency.

By plotting $\log _{e} E$ against time, which is a constant term (Figure 3), the specific fermentation rate $\left(V_{e}\right)$ of boiled and unboiled beet juice was calculated. The specific fermentation rate and the fermentation efficiency which together characterize the fermentability were plotted and the results are given in Figure 4. Good fermentability corresponds to $\mathrm{V}_{\mathrm{e}}$ of $0.4 \mathrm{hr}^{-1}$ and a fermentation efficiency of nearly $90 \%$. The results clearly show that boiling increased the fermentation efficiency considerably $(90 \%)$ and thus the alcohol yield. However, boiling did not show any significant influence on the fermentation rate.

Similar experiments were also conducted by Larsen et al. ${ }^{5}$ who reported that heating *

| Table II. Effect of filtration and centrifugation on the alcohol yield and efficiency of beet juice fermentation |  |  |
| :---: | :---: | :---: |
| Treatment | Alcohol yield, g/litre | Fermentation efficiency, \% |
| Filtration | 35.55 | 54.44 |
| Centrifugation ( $4000 \mathrm{rpm}, 30 \mathrm{~min}$ ) | 47.40 | $73.25$ |
| Unpeeled whole-beet juice | 18.96 | 29.30 |

as well as the finer fragments of the pulp.
The effect of heat treatment of juice on its fermentability was studied and the results (Figure 2) show that heat had a remarkable influence on alcohol yield. The optimum time of treatment varied with the temperature, and a conversion efficiency of $90 \%$ was achieved when juice was boiled for 30 minutes, although extending the


Fig. 2. Effect of heat treatment of beet juice on fermentation efficiency
of beet juice extracted by the diffusion process often increases the yield of alcohol as well as the vigour of fermentation. The presence of unwanted micro-organisms which compete for nutrients and produce many toxic compounds is well known to cause a considerable drop in fermentation efficiency. However, our results on the growth of S.cerevisiae in boiled and unheated beet juice did not show any significant variation, which indicates that the contribution of microbial load, if any, is minimal and the low yield of alcohol from unsterilized beet juice is due to the presence of an inhibitor. The increase in fermentability of beet juice has also been correlated with the denaturation of the metabolically active proteins which otherwise interfere with the ethanol fermentation.

Preclarification of molasses by various chemicals and polyelectrolytes is well known to increase the fermentation efficiency ${ }^{6}$. Several chemical procedures
5 Dev. Ind. Microbiol., 1981, 22.
6 Dobronravov \& Zhuravleva: Sakhar. Prom., 1972, 46. (1), 15-17.


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Fig. 3. Plot of $\log _{\mathrm{e}} \mathrm{E}$ against time of boiled $(\Delta-\Delta)$ and unheated $(O-O)$ beet juice


Fig. 4. The specific fermentation rate (Ve) and fermentation efficiency of (a) unheated and (b) boiled beet juice
have been reported in the literature ${ }^{7,8,9,10}$. The chemical and preferred concentration depend on the nature and amount of floccular material present. Some of these treatments were tried with beet juice and the results are given in Table III. All the chemicals studied increased the
fermentation efficiency remarkably, but the maximum increase ( $183 \%$ ) was seen with alum, followed by that with calcium carbonate $(148 \%)$. It appears that these chemicals either neutralize the activity of the inhibitor or cause its aggregation and thus easier removal by filtration.
$\left.\begin{array}{|lcc|}\hline \text { Table III. Effect of chemical pretreatment on the alcohol yield and efficiency of beet juice } \\ \text { fermentation }\end{array}\right]$

The presence of the inhibitory principle and its apparent association with floccular material was confirmed by adding the floccular material obtained by various physico-chemical methods to synthetic medium (Table IV). The improvement with a particular treatment and the extent of inhibition caused by the floccular material showed a positive correlation. The flocculum obtained from boiled beet juice decreased the efficiency by only $\mathbf{8 \%}$ against $53 \%$ with the flocculum from alum treatment, indicating the inhibitor to be heat-labile.

The isolation and characterization of the inhibitor is being studied in our laboratory.

## Acknowledgement

The authors are grateful to Punjab Tractors Ltd., Mohali, for providing a research grant and other facilities. They are in particular grateful to Mr. Chandra Mohan, Managing Director, and Mr. G. S. Rihal, Manager R \& D, for their invaluable guidance and constructive criticism.

## Summary

The low yields of alcohol from whole sugar beet juice of variety Ramonskaya is due to the presence of a heat-labile. inhibitor. The inhibitory substance does not affect the growth of yeast cells but has a specific affect on alcohol production. It can be denatured or removed by heat treatment, while a number of chemicals also neutralize the inhibitory effect. Alum and calcium carbonate were the most promising of these chemicals.

## Un inhibiteur de fermentation dans la betterave sucrière

La faible rendement en alcool obtenu aux dépens de betteraves sucrières entières de la variété Ramonskaya est due la présence d'un inhibiteur thermolabile. Cet inhibiteur n'a pas d'effet sur la croissance des cellules de levure mais a un effet

[^4]| Table IV. Influence of floccular material obtained by various physico-chemical <br> treatment of beet juice on the alcohol fermentation of synthetic medium |  |  |  |
| :--- | :---: | :---: | :---: |
| Source of | Alcohol <br> floccular | Fermentation <br> efficiency, | Relative decrease in <br> efficiency over <br> material |
| Fillitre | $\%$ | control, $\%$ |  |
| Filtration | 11.46 | 22.46 | 19.77 |
| Centrifugation | 8.69 | 17.03 | 38.88 |
| Boiling | 13.00 | 25.55 | 8.35 |
| Alum | 6.72 | 13.16 | 52.79 |
| Calcium carbonate | 7.90 | 15.49 | 44.44 |
| Lead acetate | 7.90 | 15.49 | 44.44 |
| Calcium oxide | 7.90 | 15.49 | 44.44 |
| Potassium ferrocynide | 9.48 | 18.58 | 33.35 |
| Triton X-100 | 8.69 | 27.04 | 38.90 |
| Control | 14.22 |  | - |
| *Conc. Added $=2 \%$ wet weight |  |  |  |

spécifique sur la production d'alcool. Il peut être dénaturé ou enlevé par traitement thermique. Un nombre de produits chimiques neutralisent également l'effet inhibiteur. L'alun et le carbonate de calcium étaient les produits les plus prometteurs.

## Ein Fermentationsinhibitor in Zuckerrüben

Die niedrigen Alkoholausbeuten aus Säften ganzer Zuckerrüben der Sorte Ramonskaja sind auf die Anwesenheit eines hitzeempfindlichen Inhibitors zurückzuführen. Der Inhibitor wirkt nicht auf die Wachstum der Hefezellen; er hat
jedoch einen besonderen Einfluss auf die Alkoholerzeugung. Die Substanz kann durch Erhitzen denaturiert oder abgebaut werden, während zahlreiche Chemikalien auch die Inhibition neutralisieren. Von diesen Chemikalien zeigten Aluminiumkalium-sulfat und Calciumcarbonat die besten Ergebnisse.

## Un inhibidor de fermentación en

 remolacha azucareraEl rendimiento pobre de alcohol del jugo de remolachas azucareras enteras de la variedad Ramonskaya es debido a la presencia de un inhibidor termosensible. La sustancia inhibidora no afecta el crecimiento de las celulas de la levadura sino tiene un efecto especifico sobre producción de alcohol. Es posible desnaturar o eliminar la sustancia por tratamiento térmico, mientras que varias sustancias químicas neutralizan el efecto inhibidorio también. De estas sustancias, las más prometedores fueron alumbre y carbonato de calcio.

## Brevities

## Brazil sugar exports limitation ${ }^{1}$

The Export Director of the Brazilian Sugar and Alcohol Institute has stated that only 2.47 million tonnes of sugar will be exported during June 1984/May 1985, compared with 3.3 million tonnes scheduled for the current crop year. This is in response to low world market prices; cane not utilized for sugar will probably be used for the manufacture of fuel alcohol. Heavy exports during the early months of this year make it necessary to limit the availability of sugar for shipment from the new crop so as to ensure that Brazil's ISA export quota of just under 2.8 million tonnes is not exceeded. This program will presumably also limit the export availability during the early months of 1985 when, it is to be hoped, a new International Sugar Agreement relying on stocking procedures rather than quotas will be in force for major producers like Brazil.

## Texas sugar crop, 1983/84

The Texas cane harvest which began on October 11, 1983 and ended on March 30, 1984, was the eleventh of Rio Grande Valley Sugar Growers Inc. and, with the exception of the first year, the least productive. The culprit was the worst freeze of the century which struck on December 24 and produced five days of below-zero temperatures which went down to $-8^{\circ} \mathrm{C}$. Until the frost, sugar recovery had averaged 6.91 tonnes/ha but by January 9 the effects of the freeze showed in rapidly deteriorating cane, with $48 \%$ of the crop remaining in the field. After January 24 no sugar was produced; pol in cane fell to 8.51 and purity to below 70, and molasses was obtained until completion of the crop at a steady rate of 83 litres/tonne of cane. The high acidity of the juice caused corrosion
problems in the mill and means adopted to ameliorate these resulted in accelerated scaling. Figures from before the freeze showed 48,501 tonnes of $96^{\circ}$ sugar made from 592,530 tonnes of gross cane, harvested from 7221 ha; after the freeze a further 4637 tonnes of sugar were made from 400,502 tonnes of cane grown on 6690 ha ; in addition to the permanent storage of 11.4 million litres, a further reservoir was excavated and lined with plastic and this eventually stored a further 17 million litres of molasses.

## Italian beet area, 1984 ${ }^{2}$

According to the President of the Sugar Beet Consortium, the 1984 sugar beet area in Italy is estimated at 210,000 hectares, only 5000 ha down from the 1983 level, despite earlier forecasts predicting planting declines. Sugar production should reach 1.2-1.3 million tonnes, white value, should normal weather continue, against $1,244,000$ tonnes last year and only $1,179,000$ tonnes from the drought-striken 1982 campaign. However, the Italian sugar industry is in need of restructuring if national demand, amounting to $1,600,000$ tonnes/annum, is to be met. The President considers that Italian beet growers should aim for a beet area of 270,000 hectares, which would be sufficient to satisfy national requirements and would avoid further deterioration of the country's food trade balance. This year, Italy will have to import 600,000-700,000 tonnes of sugar at a cost of 500-600 million lire. At a national sugar beet growers convention, he said that the sugar industry would need to be restructured to achieve the goals of 270,000 -ha area and 1.6 million tonnes sugar output. Farmers should take advantage of the new state holding company, formed to aid financially-hit sugar companies; under this plan, both the state and private
investors, such as farmers, will be able to buy into the ailing firms, creating mixed state and private sector companies. The President added that the Montesi commodities group had reduced its debt to farmers for past beet deliveries to 15,000 million lire from 130,000 million originally owed.

## Brazilian/Latin American sugar technologists meeting

The 3rd National Congress of STAB, the Brazilian Sugar Technologists Association, to be held during August 19-24 in São Paulo, is to be a joint meeting with ACTALAC, the Civil Association of Sugar Technologists of Latin America and the Caribbean.

End of sterling contracts on the London sugar futures market ${ }^{3}$

On April 30, £116 per tonne was paid on the London Terminal Market for raw sugar for delivery in May. That transaction was the final one on the No. 4 Contract and also the final sterling trade in a London Futures Exchange for sugar. The London Market reopened after the war in January 1957, and for many years prices were designated in sterling, and this has continued until the end of April. For a number of reasons it was eventually felt that the contracts should be dollar-based and, from June 1983, there has been a dollar-based contract for raw sugar in London and from July 1983 a similar one for white sugar.
1 C. Czarnik ow Ltd., Sugar Review, 1984, (1695), 63.

2 F. O. Licht, International Sugar Rpt., 1984, 116, 261.
3 C. Czarnikow Ltd., Sugar Review, 1984, (1699), 81-82.

# The influence of sodium ions on sucrose solubility in beet molasses 

By L. Blomberg, E. Swietlicka and J. Tjebbes<br>(Svenska Sockerfabriks AB)

A sugar factory's revenue from the sugar which ends up in the molasses is in Sweden only about one third of that derived from white sugar. As the portion of the sugar taken up in molasses can be diminished, we are thus experiencing an unnecessary loss of revenue. The magnitude of this loss at a given factory is influenced by both the capacity of the equipment to reach a state of equilibrium for the molasses and the solubility of sugar at equilibrium. This solubility in turn is a function of the composition of the non-sugar in the folasses. Within the Swedish industry there are a few factories where non-sugars regularly cause the sugar solubility in molasses to be around $10 \%$ higher than in the others.
The composition of the non-sugar portion of the molasses can to some extent be influenced by factory operations. The sodium content can, for instance, be changed through addition of soda lye before the evaporation for pH control or through thin juice softening via ion exchange or addition of soda ash. It is, therefore, of great interest for us to obtain a clear picture of how minor changes in the sodium content can affect the solubility of sucrose in Swedish molasses.

In this investigation the same standard methodology is used as that applied to the weekly evaluation of exhaustion of factory molasses. More specifically, the changes occuring in the solubility of sucrose, as a response to an increase of the sodium content have been measured. These jincreases have been achieved by additions of mixtures of sodium salts to the molasses. Some characteristics of the molasses samples used are recorded in Table I.
Swedish molasses normally has a sodium content of between 8 and 15 moles Na per tonne of beet, about 8 moles originating from beet, and the rest coming from additions in the manufacturing process. The amounts added in this investigation corresponds to from 7 up to 14 moles Na per tonne of beet.

## Earlier work

There exists a rich literature covering the

topic "Moles extra sucrose in molasses per mole extra sodium ion" in which estimations of this factor vary from 0.2 to 1.0 moles/mole.

Dedek ${ }^{1}$ has made a critical review of his own and others' determinations of molar concentrations of sucrose and potassium plus sodium at equilibrium both in technical molasses and in artificial solutions.

He concluded that: "Artificial solutions and technical molasses cease to crystallize at an equimolar proportion of sucrose to potassium and sodium. This proportion is the only reliable criterion for a totally exhausted sample of molasses".

The observation of the nearly equimolar proportion of sucrose to alkali in molasses has been confirmed by many authors ${ }^{2,3,4}$. In Sweden, as probably in many other countries, technical improvements in machinery and control in the after-product station have caused a gradual lowering of the sugar/alkali proportion in molasses to about 0.7 or 0.8 The above-mentioned conclusion of Dedek must be interpreted as meaning that an extra mole of sodium in the molasses binds an extra mole of sucrose.

The influence of sodium on the loss of sugar via molasses is discussed in many papers, but in most cases without being explicitly expressed as a formula of "moles extra sucrose per mole $\mathrm{Na}^{\prime}$.

Vavrinecz ${ }^{5}$ has gathered newer investigations of sucrose solubility in solutions of different sodium salts. Those investigations were done with rather low non-sucrose/water proportions, Nsa/W < 1 , normal technical molasses having a $\mathrm{NSa} / \mathrm{W}>2$.

Devillers et al. ${ }^{4}$ and Krieger ${ }^{6}$ have studied the correlations in French and Hungarian molasses, respectively, between sugar on the one hand and potassium, sodium and nitrogen on the other. The correlations indicate a much lower dependence between sucrose and sodium than 1 mole/mole.

Schneider et al. ${ }^{7}$ allowed a number of molasses to reach equilibrium at $40^{\circ} \mathrm{C}$ and thus determined a connection between the purity and a factor:

$$
\frac{\% \mathrm{NSa}-\%(\mathrm{~K}+\mathrm{Na})}{\text { moles }(\mathrm{K}+\mathrm{Na})}
$$

From other, similar measurements Reinefeld et al. ${ }^{8}$ arrived at a correlation between sucrose in molasses and the chemical composition, usually called the

## Paper presented to the 17th Gen. Assembly

 CITS, 1983.Z. Ver. Deutsch Zuckerind., 1927, 77, 495.

2 Carolan: I.S.J., 1949, 51, 277.
3 Asselbergs et al.: Zucker, 1960, 13, 574.
4 Devillers et al.: Sucr. Franç., 1976, 117 437.

5 Zeitsch. Zuckerind., 1965, 90, 449.
6 Zucker, 1977, 30, 601.
7 ibid., 1961, 14, 208, 234, 307.
8 ibid., 1974, 27, 2.

Braunschweig formula:
Sucrose in molasses $=$
$0.343 \times(\mathrm{K}+\mathrm{Na})+0.094 \times \mathrm{N}_{\mathrm{BI}}-0.31$ in which sucrose is given in $\%$ on beet and $\mathrm{K}, \mathrm{Na}$ and $\mathrm{N}_{\mathrm{B} 1}$ are expressed in millimoles per 100 g of beet. According to this formula an extra mole of sucrose is retained in molasses per extra mole Na .
Quentin ${ }^{9}$ and Partale et al. ${ }^{10}$ describe how the sucrose solubility is affected when ion exchange resins are used for exchanging cations in molasses with $\mathrm{K}, \mathrm{Na}$, Ca or Mg . In these studies it is shown that the solubility is markedly diminished when K or Na is replaced by Ca , but without explicitly stating how many fewer moles sucrose in molasses are dissolved per mole Na exchanged for Ca .

Finally, in a paper of 1974 Schneider \& Perschak ${ }^{11}$ discuss the increase in molasses sugar caused by increasing sodium content. The findings in the earlier work ${ }^{7}$ are interpreted, and the authors reach the conclusion that an extra mole of sodium ion will cause an increase in sucrose of 0.2 moles.

## Experimental

As stated above, in Sweden the degree of exhaustion in molasses from all the sugar factories is regularly evaluated. This is done by use of a method developed from those described by Wiklund ${ }^{12}$, Wagnerowski et al. ${ }^{13}$ and Partale et al. ${ }^{10}$. In this method sucrose solubility in the samples is determined at $45^{\circ} \mathrm{C}$ and $\mathrm{NSa} / \mathrm{W}$ $=2.8$. This temperature and concentration have been chosen as standard conditions for the massecuites before centrifugation. At the time of this decision, around 1975, these conditions were considered as optimal. Although technology has been improved since then, we have kept the same standard conditions for the sake of comparison. Normally the technical molasses still show a higher content of sucrose as they have not reached equilibrium and have not been cooled sufficiently.

For this investigation we have used samples of eight technical molasses from the 1978 and 1982 campaigns which were produced by the sugar factories at Örtofta
and Jordberga, with a daily slice of 6000 and 5000 tonnes of beet, respectively. Neither factory uses thin juice softening or a Quentin process. To the samples were added aqueous solutions of mixtures of sodium chloride, acetate and lactate in equal molar concentrations. These counterions were selected owing to their dominance among the anions of beet molasses, and changes in composition of the non-sugars were thereby minimized. Compared with the figures given by Vavrinecz ${ }^{14}$ for the average composition of beet molasses, the contribution from these anions were increased by the additions from 15 to, at the highest, $19 \%$ of the non-sugars.
The sucrose solubility and the sodium content were determined for all samples, both original and enriched. Atomic absorption spectrometry was used for the determination of sodium. In the results only these analytically determined concentrations of sodium ion have been used, thus ascertaining that no undetected precipitation of sodium salts had taken place during the exhaustion analysis. In the factory samples the sodium ion content is normally little more than 100 millimoles per 100 g NSa and this was increased through the added salt solutions by $50-100$ millimoles per 100 g NSa . The analytical determinations show a standard deviation of 4 millimoles per 100 g NSa .
In order to obtain a value for the solubility of sucrose at $\mathrm{NSa} / \mathrm{W}=2.8$ six solubility experiments are performed. They are done in duplicate at three different $\mathrm{NSa} / \mathrm{W}$ values obtained by the addition of water. To the six samples sucrose crystals are added corresponding to an increase in sugar of $40 \%$ of the sample. The samples are then kept rotating in closed stainless steel vessels at $70^{\circ} \mathrm{C}$ for 12 hours. Afterwards they are run through a pressure filter, also at $70^{\circ} \mathrm{C}$, and the syrup thus obtained is analysed. The following determinations are made: total solids (TS), pol, raffinose (enzymatically by $\alpha$ galactosidase and NAD according to Schiweck \& Büsching ${ }^{15}$ ), invert sugar (by dinitrophthalic acid ${ }^{16}$ ) and sodium. The content of sucrose ( Sa ) in $\mathrm{g} / 100 \mathrm{~g}$ is
calculated by the formula:

$$
\begin{aligned}
\mathrm{Sa}=\mathrm{Pol}-1.852 & \times \text { Raffinose } \\
& +0.33 \times \text { Invert }
\end{aligned}
$$

From the values for Sa and TS the sucrose solubility can be obtained. It is normal practice to compare the solubility for sucrose in molasses with that in pure water by the coefficient of saturation, C . At a given temperature, $t^{\circ} \mathrm{C}$, and at the current $\mathrm{NSa} / \mathrm{W}$-ratio the coefficient of saturation is defined by

$$
\mathrm{C}=\frac{\text { Sucrose solubility in sample }}{\text { Sucrose solubility in water }}
$$

C is a linear function of the $\mathrm{NSa} / \mathrm{W}$ ratio and not dependent upon temperature in the interval of interest for molasses. The six values obtained from the experiments are thus plotted against the corresponding $\mathrm{NSa} / \mathrm{W}$ ratio and a straight line is drawn using the least-squares-fit method. The value for C at $\mathrm{NSa} / \mathrm{W}=2.8$ is thus obtained from this graph. Normally there is found a standard deviation in this $\mathrm{C}_{2.8}$-value of 0.01 .

## Results

The measured changes in $\mathrm{C}_{2.8}$ resulting from the addition of sodium salts can be expressed as "moles more sucrose in molasses per mole extra sodium ion". For simplification an amount of 100 g NSa has been used as a reference for calculation.

At a $\mathrm{NSa} / \mathrm{W}$ ratio of $2.8,100 \mathrm{~g} \mathrm{NSa}$ is dissolved in 100/2.8 $=35.71 \mathrm{~g}$ water. At a temperature of $45^{\circ} \mathrm{C}$ the solubility of sucrose in water is $2.4589 \mathrm{~g} / \mathrm{g}$. The amount ${ }^{\text {. }}$ of dissolved sucrose at saturation and a coefficient of saturation $=\mathrm{C}_{2.8}$ is then, in g per 100 g of NSa :
$35.71 \times 2.4589 \times \mathrm{C}_{2.8}=87.82 \times \mathrm{C}_{2.8}$.
If then the coefficient of saturation is increased by the addition of sodium salts by $\Delta \mathrm{C}_{2.8}$ an increased amount of sucrose
9 ibid., 1957, 10, 408.
10 ibid., 1970, 23, 155.
1 ibid., 1974, 27, 542.
12 Socker Handl., 1946, 2, 65.
13 Zeitsch Zuckerind., 1962, 87, 664.
14 ibid., 1974, 99, 23.
15 "Sugar Analysis-ICUMSA Methods", Ed. Schneider. (ICUMSA, Peterborough) ' 1979 , p. 71.

16 Emmerich: Zucker, 1967, 20, 603.

The influence of sodium ions on sucrose solubility in beet molasses
will be dissolved at saturation. The increase will be:

$$
87.82 \times \Delta \mathrm{C}_{2.8} \mathrm{~g} / 100 \mathrm{~g}
$$

or, if amounts of sucrose are given in moles $/ 100 \mathrm{~g}$,

$$
\frac{87.82}{342.3} \times \Delta \mathrm{C}_{2.8}=0.2566 \times \Delta \mathrm{C}_{2.8}
$$

If this increase has resulted from an increase of sodium ions by $\Delta \mathrm{Na}$ moles $/ 100$ g NSa, a value of

$$
0.2566 \times \frac{\Delta \mathrm{C}_{2.8}}{\Delta \mathrm{Na}}
$$

expresses the increase of "moles more sucrose per mole extra sodium ion".
For example, an increase in $\mathrm{C}_{2.8}$ of 0.078 with an increase in sodium ions of 0.100 moles $/ 100 \mathrm{~g}$ NSa means that we have Obtained an increase of 0.20 moles of sucrose in molasses per mole of sodium ions.
The experimental results from this part of the investigation are reported in a compressed form in Figure 1, which shows the changes in $\mathrm{C}_{2.8}$ compared with increasing contents of sodium ions. In the figure, two theoretical lines are drawn corresponding to an increase in sucrose solubility by 1.0 and 0.2 moles more sucrose per mole extra sodium ion respectively. It is clearly seen that for the investigated molasses, the value is lower than 0.2 .
When sodium hydroxide is added to the
thin juice there will also be an increase of sugar in molasses caused by the increase in non-sugars. If the addition corresponds to an increase of 0.1 mole $\mathrm{Na} / 100 \mathrm{~g} \mathrm{NSa}$, the increase in the amount of non-sucrose will be $2.3 \mathrm{~g} / 100 \mathrm{~g}$, the atomic weight of Na being 23 . This addition corresponds to $2.3 / 2.8=0.82 \mathrm{~g}$ of water at a constant $\mathrm{NSa} / \mathrm{W}$-ratio of 2.8. At a normal coefficient of saturation of $\mathrm{C}_{2.8}=1.3$, this extra water will hold sucrose in an amount of

$$
\frac{0.82 \times 2.4589 \times 1.3}{342.3}=0.0077 \text { moles } \mathrm{Sa}
$$

This corresponds to $0.0077 / 0.100=0.08$ expressed as "moles more sucrose per mole extra sodium ion".

The total increase of sugar in molasses caused by extra sodium ions is the sum of the two described effects. It can thus be estimated to be less than 0.3 moles $/ \mathrm{mole}$ in Swedish molasses when caustic soda is used for pH control. Of course, as Schneider ${ }^{11}$ also underlines, this increase must be compared with the loss in sugar yield caused by invert formation.

Within the thin juice softening process calcium ions, $\mathrm{Ca}^{2+}$, are replaced by sodium ions, $\mathrm{Na}^{+}$. In order to estimate how this exchange will influence the sucrose solubility in the molasses comparison must be made of pairs of molasses where part of the calcium in one


Fig. 1. Changes in $\mathbf{C}_{2.8}$ after salt addition


Fig. 2. Differences in $\mathrm{C}_{28}$ between Na -enriched and Ca -enriched molasses

|  | Nomenclature and symbols |
| :---: | :---: |
| TS | $=$ Total solids by refractometer, $\mathrm{g} / 100 \mathrm{~g}$ |
| Pol | $=$ Sugar, uncorrected, by polarimeter, ${ }^{\circ} \mathrm{S}$ |
| Sa | $=$ Sucrose, i.e. Pol corrected for raffinose and invert, g/100 g |
| W | $=$ Water, obtained as $100-\mathrm{TS}, \mathrm{g} / 100 \mathrm{~g}$ |
| NSa | $=$ Non-sucrose, obtained as TS $-\mathrm{Sa}, \mathrm{g} / 100 \mathrm{~g}$ |
| $\mathrm{NSa} / \mathrm{W}$ | $=$ Non-sucrose-water ratio |
| $\mathrm{L}_{\mathrm{t}}$ | $=$ Solubility of sucrose in water at a temperature of $t^{\circ} \mathrm{C}^{17}, \mathrm{~g} / \mathrm{g}$ |
| C | $=$ Coefficient of saturation for sucrose in molasses |
| $\mathrm{C}_{2} 8$ | $=\quad \mathrm{C}$ at a $\mathrm{NSa} / \mathrm{W}=2.8$ |

Melasse gesetzt worden. Die Löslichkeit der Saccharose in Originalmelasse und Na bereichterte Melasse ist mit Hilfe unserer Standardmethode für die
Melasseentzuckerungver-messung bestimmt worden. In den untersuchten Melassen hat Na die Löslichkeit mit weniger als 0.2 Mol Saccharose per Mol zugesetzte Na erhöht. Wenn Ca in der Melasse gegen Na ausgetauscht wurde, zeigte es sich, dass die Löslichkeit der Saccharose mit 0.3-0.4 Mol Saccharose per Mol Na erhöht wurde.

## L'influence des ions de sodium sur la solubilité de saccharose dans la mélasse de betterave

Il y a dans la littérature des estimations comment la teneur du Na -Ion influe la quantité du sucre de la mélasse. On a constaté qu'une augmentation de la teneur du Na d'un mol cause une augmentation du sucre de la mélasse de $0.2-1.0 \mathrm{~mol}$ saccharose. Dans notre analyse ça nous avons fait usage des quelques mélasses
suédoises. Nous avons ajouté des mélanges des Na -sels en forme de chloride, acétate et lactate à la mélasse. La solubilité de la saccharose en mélasse originale et en
mélasse enrichissée du Na a été fixé par notre méthode générale pour l'épuisement de la mélasse dans la fabrique. En les mélasses examinées le Na a augmenté la solubilité de la saccharose de moins que 0.2 mol par mol Na qui a été ajouté. Dans le cas le Na remplace le Ca il s'est trouvé que la solubilité de la saccharose a augmenté de $0.3-0.4 \mathrm{~mol}$ saccharose par mol Na ajouté en remplaçant le Ca .

## La influencia de los iones de sodio sobre la solubilidad de sacarosa en melaza de remolacha

En la literatura se encuentran estimaciones de la magnitud de la influencia de iones de sodio sobre la cantidad de azúcar en melaza que varian entre 0.2 y 1.0 moles de sacarosa por mol de sodio. Se han añadido a varias melazas suecas soluciones de sales de sodio lactato, acetato y cloruro -y el efecto de la mezcla sobre la solubilidad de sacarosa se ha determinado por el método usualmente empleado para determinación de agotamiento. En las muestras investigadas, los iones de sodio han producido un aumento de solubilidad de sacarosa de menos de 0.2 moles por mol. En el caso donde el sodio sustituye el calcio, se encuentra que la solubilidad de la sacarosa es aumentado de $0.3-0.4$ moles por mol de Na añadido.

## Brevities

Hazards control in handling and storage of granular foods

An international symposium is to be held on this topic in Paris during April 24-26, 1985, the eight sections including those concerned with dust explosions, spontaneous fires, particularly in dryers, biological heating during storage, etc. A call has been made for communications and further information is available from APRIA (Association pour la Promotion Industrie Agriculture), 35 rue du Général Iooy, 75008 Patis, France.

## No change to EEC sugar plan ${ }^{18}$

The European Commission says a review of the world market for sugar shows that there is no need to revise the EEC's 5 -year production quota program for sugar before it expires at the end of June 1986. According to a review of the quota program completed halfway through its life-span, the system has led to a drop in the area
under sugar beet in the Community to the lowest since 1975 and has achieved its main objectives of generating enough revenue from producers to finance surpluses. Revenues are projected to exceed spending by 41.3 million E.C.U. in the current year.

## Chinese sugar crops, $1983{ }^{19}$

The output of sugar beet in China rose by $36.8 \%$ ד in 1983 to $9,182,000$ tonnes, according to the State Statistical Bureau in Beijing. This no doubt reflects the economic changes which the Chinese authorities have recently introduced, enabling villagers to benefit as a result of increased output. On the other hand, there was a fall in sugar cane output by $15.6 \%$ to $31,141,000$ tonnes, as a consequence of adverse weather conditions.
17 Vavrinecz: Zeitsch. Zuckerind., 1962, 87, 481.

18 Public Ledger, May 5, 1984.
19 C. Czarnikow Ltd., Sugar Review, 1984, (1701), 90.

## Brevities

## Gyyana sugar industry strike ${ }^{1}$

Six of Guyana's ten sugar factories were closed on March 5 by a strike called by unions in protest at delays in payment of wage increases ordered by the court, and seeking the withdrawal of controversial labour legislation before
Parliament. The strike came to an end two weeks later when the government met their demands.

## HFS use in Coca Cola ${ }^{2}$

It was reported early in May that Coca Cola Co. has raised the maximum level of high fructose syrup allowed in its fountain drinks from $75 \%$ of sweetener content to $100 \%$. It has been estimated that if this new limit is utilized to the
full there would be a loss of outlet to sugar amounting to some 50,000 tonnes a year.

Louisiana 1983 sugar production ${ }^{3}$
A total of $6,429,251$ short tons of cane crushed in the Louisiana sugar factories during the 1983 Bison yielded 595,878 tons of raw sugar and $31,597,102$ US gallons of $80^{\circ} \mathrm{Bx}$ molasses.

## Dominican Republic sugar crop delay ${ }^{4}$

Heavy rains caused a reduction of crushing rate in the Dominican Republic's sugar factories and put the crushing of the 1983/84 cane crop behind schedule. Nevertheless, sugar production by the factories of C.E.A. is expected to reach the projected level of 800,000 tonnes, against 772,000 tonnes in $1982 / 83$, while the production of the private sector is expected to reach around 500,000 tonnes. Most of the country's production will be sold to the US under quota.

## New pelleted pulp plant in Denmark

A/S De Danske Sukkerfabrikker is to install a new, modern drying plant at its Saksk $\phi$ bing sugar factory, to commence operations in the 1985 campaign. The capacity of the plant will be about 15 tonnes of pellets per hour. Output will either be sold in Denmark, where it will replace imports, or will be exported. The cost of the hant is about 45 million krone and will be financed from the proceeds of a recent increase in share capital, as will be a new steam boiler at the Assens factory and a new storehouse and packing plant in Copenhagen.

## Burundi-Cuba agreement ${ }^{5}$

Under an agreement signed in Havana in September 1983, Cuba is to provide technical assistance and training in a number of fields including the sugar industry.

## Chile sugar factory to re-open ${ }^{6}$

The Rapaco plant is to be put back into operation in 1984 by the Chilean national sugar company IANSA which operates four other plants. As a consequence, white sugar production is expected to rise to 300,000 tonnes from 222,000 tonnes last year. Domestic consumption
is estimated at between 330,000 and 350,000 tonnes and the balance will be covered by imports expected to come from Argentina, Bolivia and Peru.
Call for irrigation expansion in north-east Brazil ${ }^{7}$
After a drought from January to August and only intermittent showers in the rest of the year, the 1983 cane crop in the Campos region of Rio de Janeiro state was a disaster and the 1984 crop is expected to be the same. This could have been avoided if an kdequate irrigation system were available, however, since there are many permanent waterways in the region. Cane yield is 35-40 tonnes per hectare but this can be raised to 120 tonnes per hectare by means of irrigation, against 80 tonnes per hectare in São Paulo state, the leading sugar producing area in Brazil.

## Uganda sugar factory temporary closure ${ }^{8}$

Madhvani Sugar Company recorded losses of 600 million Uganda shillings ( $\$ 2,000,000$ ) in 1983 and has announced a "temporary stoppage for a period of about 12 months". The company is stated to be finding it difficult to continue running the plant and has approached the government (which owns $51 \%$ of the equity) for financial help. The company's sugar complex at Kakira, Jinja, produced 131,000 tonnes of sugar in $1971 / 72$, before it was expropriated during the Amin regime, but then fell to 1000 tonnes per year. A $£ 65$ million rehabilitation program was agreed in 1982 and the Madhvani Group were charged with restoring production capacity to 90,000 tonnes/year; however, production in 1983/84 is estimated at only 6000 tonnes.

## Cuban bagasse paper plant ${ }^{9}$

Trial production, using wood pulp imported from the USSR, has begun at the Jatibonico Bond Paper Complex in Cuba. The plant is to change to bagasse as its raw material and when at full capacity will produce 60,000 tonnes of good quality bond paper per year.

## Ethiopia sugar project delay ${ }^{10}$

It is reported that progress on the Fincha sugar scheme in Ethiopia has been delayed following reports of the withdrawal of Libyan financial support for the project, although this withdrawal has not been confirmed.
Sugar beet transplanting in China ${ }^{11}$
In recent years a method of cultivating sugar beet has been developed whereby seeds are sown in separate pots in hot-houses and are then transplanted into the fields at a later stage. This enables the beet growing season to be extended and also ensures a greater field population. The system originated in Japan but there have been field trials in the United Kingdom and elsewhere. This approach clearly has the greatest potential where labour costs are low, and last season experiments were carried out on 220 hectares in the Heilongjiang Province in China and resulted

Taiwan sugar exports, $1983^{12}$
1983
1982

|  | tonnes raw, value - |  |
| :--- | ---: | ---: |
|  | 60,471 | 130,349 |
| Japan | 82,653 | 127,736 |
| Korea, South | 11,740 | 65,764 |
| Saudi Arabia | 30,347 | 56,013 |
| USA | 958 | 474 |
| Other countries | $\underline{186,169}$ | $\underline{380,336}$ |

in improvements in both beet yields and sugar content. The experiment is to be extended to 2670 hectares this season.

## Mechanical cane harvesting in Cuba ${ }^{13}$

The number of cane harvesters in operation in the 1983/84 crop was expected to reach 3700 units and to harvest $62 \%$ of the cane crop. The remaining cane would be cut by some 24,600 manual workers.

## Poland sugar production, 1983/84 ${ }^{14}$

In its best campaign since World War II, the Polish sugar industry processed 17 million tonnes of beet in the 1983/84 campaign to produce $1,970,000$ tonnes of sugar.

Indonesian manufacture of Australian sugar machinery ${ }^{15}$

Discussions have been held between Indonesian and Australian business executives on the manufacture in Indonesia of sugar factory equipment under licence from Australian firms or on a joint-venture basis. The Indonesian government hopes to build 18 new sugar factories with a daily capacity of 4000 tonnes of cane, with the help of foreign partners. A seminar had been held in February which initiated discussions and was attended by representatives of leading Australian companies engaged in the manufacture of cane harvesters and other field equipment, transport systems, milling and processing machinery and firms involved with consultancy services to the cane sugar industry.

1 World Sugar J., 1984, 6, (10), 34.
2 C. Czarnikow Ltd., Sugar Review, 1984, (1700), 86.

3 Sugar J., 1984, 46, (10), 23.
4 Reuter Sugar Newsletter, March 2, 1984.
5 Cuba Economic News, 1983, 18, (137), 5.
6 F. O. Licht, International Sugar Rpt., 1984, 116, 226.
7 Sugar y Azúcar, 1984, 79, (4), 9.
8 F. O. Licht, International Sugar Rpt., 1984, 116, 227.
9 Cuba Economic News, 1983, 18, (137), 16. 10 F. O. Licht, International Sugar Rpt., 1984, 116, 227.
11 C. Czarnikow Ltd., Sugar Review, 1984, (1697), 74.

12 I.S.O. Stat. Bull., 1984, 43, (2), 9.
13 Cuba Economic News, 1983, 19, (138), 3.
14 Zuckerind., 1984, 109, 358.
15 World Sugar J., 1984, 6, (11), 36.

## Trade notices

## Micro-processor-based refractometer

Electron Machine Corporation, 1500 West Ocala Street, Umatilla, FL 32784, U.S.A.
The MPR-83 refractometer can be used in any process line where a product is blended, concentrated or where the dissolved solids content needs to be monitored and/ or controlled. The instrument offers flexibility in both on-line and sample measurements, instant direct alphanumeric display and capability for interfacing with printers, remote displays and central computers. The reading is unaffected by colour, turbidity, foam, entrained air or solids, and the mechanical structure of the sensing head eliminates error introduced by vibration or temperature change of components. Options include alarm setpoints, automatic cleaning, etc. as weil as various installation options.
model illustrated, the VB 250/500, which has a capacity up to $1250 \mathrm{~m}^{3} / \mathrm{hr}$ at heads up to 90 metres. It is available in cast iron,


## New vertical rotor pump

Tangie Engineering Ltd., Blandford Heights Industrial Estate, Blandford, Dorset, England.
The VB series of vertical, single-stage, radially split casing, long coupled inline pumps has been expanded with the latest
bronze or stainless steel and various seal arrangements are available. Unlike the smaller CV close-coupled valves, the VB pumps have a space coupling which, when removed, allows the complete rotating assembly to be removed, including the bearing housing, without disturbing the pipework connexions or vertically mounted motor. Reassembly is simple as
the bearing housing and rotating parts are self-aligning.

## Bagasse board-making plant

Compak Systems Ltd., Beaumont Street, Gainsborough, Lincs. DN21 2EP, England.
A new process has been developed for the manufacture of board from bagasse or similar residues. It provides boards in thickness from 4 to 40 mm and with density from 300 to $650 \mathrm{~kg} \cdot \mathrm{~m}^{-3}$ or more, according to requirements, with excellent physical characteristics, impact and tensile strength. The boards can be sawn, cut, nailed, stapled or glued and laminates of every kind may be applied, in many cases as an integral part of the process. The Compak plant is designed for simple oper- $\%$ ation with minimum dependence on training or craft skills. In many cases it can be run by as few as 5 people per shift and a $650 \mathrm{~m}^{2}$ site will accommodate an entire plant, including raw materials storage, steam raising plant and, where needed, conversion machinery for boxmaking, pallet construction, etc. Single-shift output is about 1000 tonnes a year.

## High-pressure slurry pumps

Holthuis B.V., P.O. Box 249, 5900 AE Venlo, Holland.
A series of piston diaphragm pumps has been designed for the pumping of large quantities of slurries at high pressures. The pump capacities range from 5 to $700 \mathrm{~m}^{3}$ / hr and pressures from 5 to 175 bar . The diaphragms and valves have a minimum lifé of 6000 hours and 3000 hours, respectively. Among the applications is the pumping of sludge in the sugar industry.

PUBLICATIONS RECEIVED

## Materials handling

TBMA Holding B.V., Noordwijkerhout, Westeinde 68a, Holland; TBMA (UK) Ltd., 11a Stafford Street, Stafford, Staffs. ST16 2BP, England.
A brochure from TBMA describes various items for use in materials handling, including rotary, slide and diverter valvés, blowers, filters, cyclones, air conveyors, silos and tanks and equipment for their discharge.

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    1 Pigman \& Anet: in "The Carbohydrates", Vol. IA, 2nd Edn. Eds. Pigman \& Horton, (Academic Press, New York) 1972, pp. 165-194.
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    18 Carbohyd. Res., 1974, 33, 47-61.

[^1]:    Self-setting mill
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[^3]:    * One $g$ of glucose according to the GayLussac equation gives 0.51 g of ethanol

    1 Chimie et Industrie, 1943, 12. 146.
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