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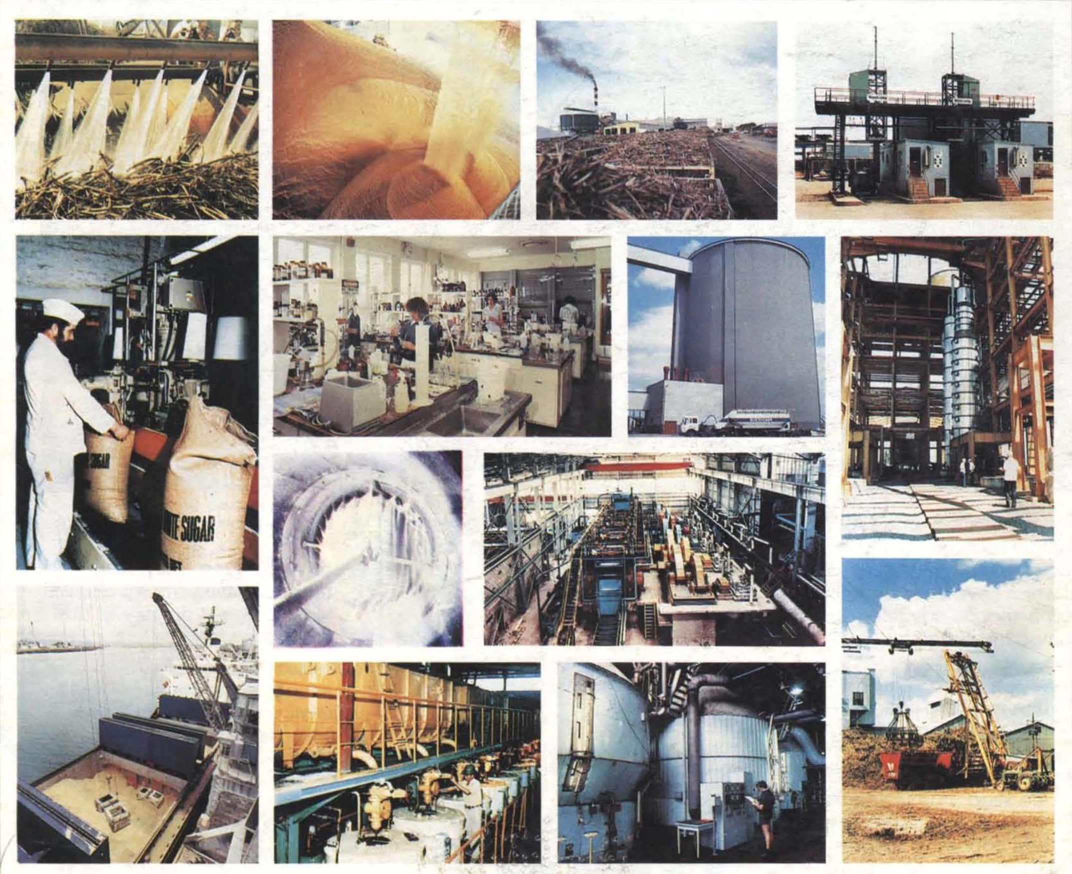

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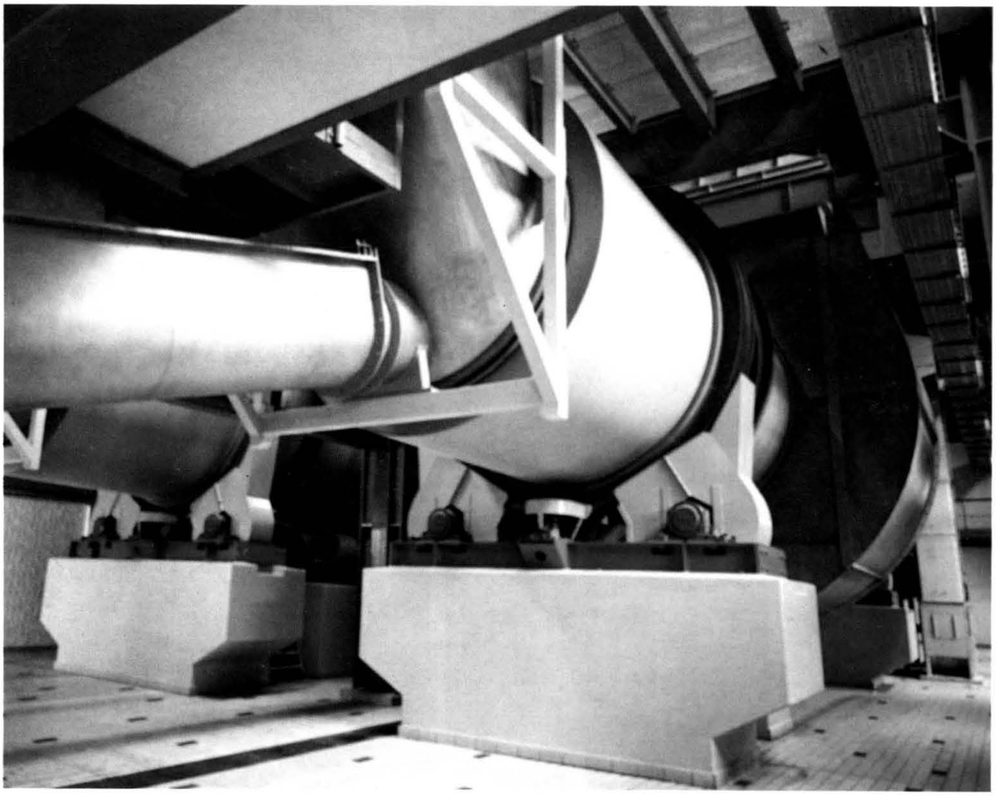

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CONTENTS October 1987

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Technical articles

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Abstracts section

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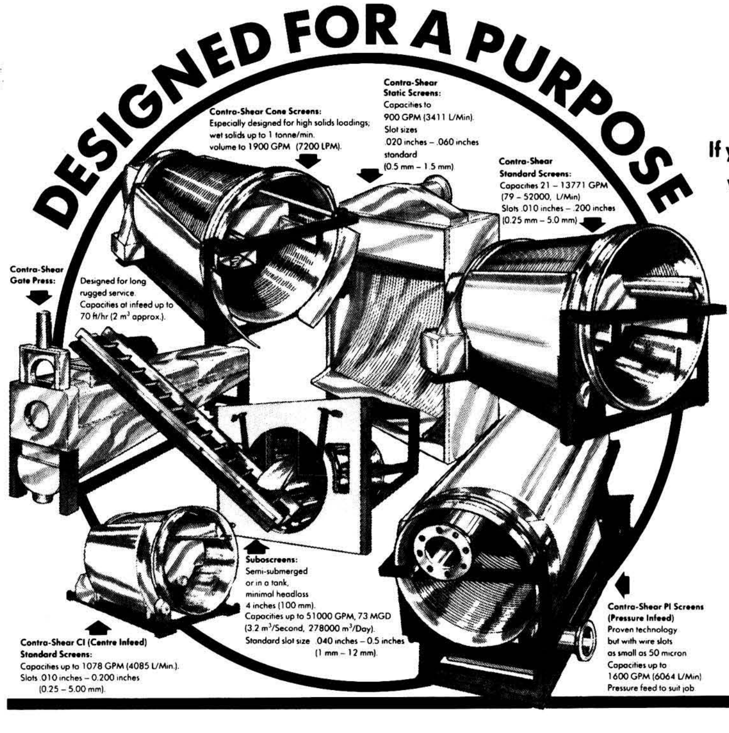

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News and views

UN sugar conference, 1987

A UN sugar conference was convened during September 10 and 11 at the London headquarters of the International Sugar Organization. A meeting of the Preparatory Committee to finalize the complete draft text of the Agreement was held on September 9. C. Czarnikow Ltd.[1] commented:

"This is in no way a step forward towards a new ISA with economic terms; rather is it a salvaging of the existing status quo with only an administrative agreement in operation. For some time past the work of the Council has been more taken up with matters concerning the financing of the ISO itself than with sugar matters and it would seem that a real change of heart will be needed before any progress towards an effective ISA can be made."

World sugar prices

A combination of poor weather in Europe, drought in India and possible typhoon damage in the Far East aided the sugar market during the first part of August and the London Daily Price for raw sugar, which had started the month at $151.60, rose to $157.60. The white sugar market was also buoyed by the fact that, with domestic supplies and Cuban imports limited by bad weather, there is a likelihood that purchases will be needed by the Soviet Union from the world market. Thus the LDP(W) rose from $172.20 to reach $185 on August 24. An unexpected selling tender by Mexico weakened the raw sugar market and, in the absence of positive factors, the LDP continued lower, ending the month at $147.60, while the white sugar price, only slightly affected by the raw sugar market, ended the month at $182.40.

US bill to increase Caribbean sugar imports

A feature of a new Trade Bill, under consideration by a joint Congressional committee, is a proposal for extension of the Caribbean Basin Initiative (CBI) for a further 12 years to September 2007. It also provides that any CBI beneficiary country with a sugar import quota should have that quota restored to its level for the period from September 1983 to September 1984. This would bring the group's current total quota of 340,000 short tons, raw value, to 1,123,782 tons. Since the current total quota is only just over 1 million tons, such a move would mean eliminating quotas for all non-CBI suppliers if the overall quota were not increased. Further, the US Department of Agriculture estimates that domestic output of sugar will rise to 6.8 million tons this season – other sources put it at up to 7 million tons – and traders expect the import quota for 1988 to be cut to well below a million tons.

Consequently, whether the overall import quota is raised by the increase for the CBI countries or limited to their 1,123,782 tons, there would be a surplus of sugar on the US market which would have to be re-exported at heavily subsidized prices. On balance, therefore, it seems unlikely that the proposals will meet with success.

World sugar balance, 1986/87[2]

The fourth estimate of the world sugar balance for the 12 months to end-August 1987 issued by F. O. Licht GmbH indicates a small increase in final stocks against a draw-down of 3.5 million tonnes expected in the first estimate in September 1986. Since the third estimate was published in June, although slightly lower crops are shown for the USSR, South Africa, Cuba and Fiji, higher figures are given for the US, Mexico, Brazil, China and India, raising total production to 104,125,000 tonnes, raw value, or some 200,000 tonnes more than the June estimate and 916,000 tonnes more than estimated consumption for the period.

Effects of changes in the world sugar market on Latin America and the Caribbean

During a seminar on international sugar marketing sponsored by GEPLACEA and a number of sugar traders, Dr. Eduardo Latorre, Executive Secretary of the group, discussed the changes affecting members[3]. The first structural change, he said, has been from a basically export-oriented industry to one in which the main production is for domestic consumption. In the mid-1950's, of the 12 million tonnes of sugar produced in the region, about 66% was for export, while in the mid-1980's the proportion had been reduced to 45%. Given the enormous differences in a country-by-country basis, some with an industry producing more than 7 million tonnes of sugar and others with less than 100,000 tonnes per year, the 22 GEPLACEA countries are grouped in three categories.

First, there are those 16 countries in which the principal use of sugar production is for domestic consumption — Argentina, Bolivia, Brazil, Colombia, Costa Rica, Ecuador, Guatemala, Haiti, Honduras, Mexico, Nicaragua, Panama, Peru, El Salvador, Uruguay and Venezuela. Then there are the 5 countries with specially agreed markets for most of their production: Barbados, Cuba, Guyana, Jamaica and Trinidad. Finally, there is one country where the main destination for production is the world free market: the Dominican Republic.

The second structural change has been where the exports go; in the mid-1950's about 50% of the region's 7.7 million tonnes of exports went to the US, about 30% to Western Europe and practically none to the Soviet Union, East Europe, the Arab countries or China. Thirty years later, the main buyers of the 13 million tonnes of sugar exported were the USSR (35%), the US (14%), Arab countries (13%), East Europe (9%), and China (5.5%).

The third and last change has been the move from exclusively a sugar industry to a sugar cane agro-industry. Three decades ago, the Latin American and Caribbean industry, for all practical purposes, produced only sugar and molasses, but today there is greater emphasis on by-products and particularly

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[1] *Czarnikow Sugar Review*, 1987, (1764), 121.
[2] F. O. Licht, *Int. Sugar Rpt.*, 1987, **119**, 369 - 377.
[3] *GEPLACEA Bull.*, 1987, **4**, (7).

in the manufacture of alcohol which, if made from cane juice, competes with sugar as an alternative product. Fifteen years ago there were over 600 sugar factories and no autonomous alcohol distilleries in the region; today there are 300 autonomous distilleries and the same number of sugar factories. It is true that most of the distilleries are in Brazil, where nowadays two-thirds of the 237 million tonnes of cane harvested go for alcohol production and the remainder for sugar, but many countries such as Argentina, Costa Rica, Ecuador, Guatemala, Jamaica and El Salvador, are making alcohol a significant part of their industry. Practically all of this alcohol is for national domestic consumption, except for some exports to the US under the Caribbean Basin Initiative which allows for duty-free entry.

Between 1960 and 1970, sugar production in Latin America and the Caribbean grew annually by close to 4%, slowing to 2.25% between 1970 and 1985, of which the last five years showed growth of only 1%. While this was partially due to opening of the US market in the 1960's, special arrangements for Cuba with the USSR and high sugar prices in the 1970's, the main factor was the growth of local consumption, particularly in Brazil and Mexico which together account for about 60% of the region's offtake. Today's rates of growth in per caput consumption and of population would, if continued to the year 2000, require 21 million tonnes of sugar, against 10 million tonnes in 1970 and 16 million tonnes in 1985.

Were conditions to permit a world sugar price of say 15 cents/lb in 1986 dollar terms, GEPLACEA calculates that production would rise to some 37 million tonnes, whereas if it were to remain around 6 cents/lb production would not rise much above 27 million tonnes. Thus export availability could vary between 6 and 16 million tonnes, a difference that underlines the need to achieve a more sensible economic order.

US call for farm subsidies elimination

In early July the US Administration presented a plan to GATT members which called for the elimination over a period of ten years of all agricultural subsidies, the dismantling to commence in September 1987. The immediate response from the EEC was that the proposals were unrealistic, and the failure of the Administration to obtain support in the Congress for its proposal to reduce the US loan figure from 18 to 12 cents/lb over a period casts doubt on its ability to secure subsidy elimination in its own country, especially as removal of financial support could seriously damage some agricultural sectors including the domestic sugar industry.

C. Czarnikow Ltd. note[4], however: "In some areas US government policy in the field of agriculture has already turned against protectionism; it is unfortunate that sugar still remain a commodity receiving the benefits of fiscal policies which subsidise the domestic industries at the expense of the mostly western hemisphere developing countries which used to rely on the US market to provide a guaranteed and high-priced outlet for a large proportion of their exports. However, it is suggested that, if the US can rely upon the cooperation of other members of GATT, hardly a single element of the current US farm program would survive in its present state.

"Though the first reaction of EEC officials may have been to reject the US proposals, the present financial straits to which the CAP has been reduced will probably make it necessary for a more measured response to be given eventually. The European Community Farm Commissioner has since stated that the EEC will never agree to the total elimination of production related subsidies and he added that the US proposals, extending beyond the program which had been agreed at GATT, went too far, too fast. Nevertheless, he stated that the community was prepared to negotiate on agricultural reform. Agricultural subsidies world-wide are leading to increasing financial problems while the drop in the rate of growth in sugar consumption has come partly as a direct result of high internal prices which frequently prevail even in countries which import sugar at world market prices."

White sugar market prospects

During a presentation by Mr. Jean-Pierre Mabilon, President of Amerop Sugar Corporation, before the Advanced Seminar on International Sugar Marketing, organized by GEPLACEA in New York in June, he discussed medium-term prospects for the white sugar market as follows[5]:

"There is no doubt that the potential for development on the world market belongs to white sugar. The domestic markets of industrialized countries such as the USA and Japan (buyers of raw sugar) are now saturated. New sweeteners (HFS, Aspartame) have, since 1980, accelerated the decline of sugar consumption in these countries. Further decline might be limited as the penetration of HFS – especially in the USA – seems to have reached its maximum but, overall, the demand for raw sugar will depend more and more on the policies of the two socialist 'giants': the USSR and China.

"Fortunately, the situation looks quite different for the white sugar market, where growth potential still looks strong. One main reason to justify this optimism is that developing countries (including oil producing countries) which, because of import restrictions, have reached a low level of stocks, will have to gradually increase their imports to meet the real needs of their domestic markets. Another favourable factor is that India is likely to remain a large buyer for years to come. Indian authorities are regularly increasing the sugar cane price, but not enough to encourage a massive production increase. Meanwhile, the satisfaction of consumers' needs seems to remain a priority concern of the government. Therefore the Indian deficit should persist, although maybe at a lower level than during the years 1985 to 1987.

"The USSR and China could also reappear as buyers of white sugar at any time. The fact that they recently

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[4] *Czarnikow Sugar Review*, 1987, (1763), 100.

[5] *Amerop-Westway Newsletter*, 1987, (165), 14 - 15.

withdrew from the whites market cannot be considered as definitive. One must remember that the USSR has to refine not only its own production but also the raw sugar it receives from Cuba. Consequently, for logistical reasons, this country could have to re-enter the whites market fairly soon.

"Finally, other persistent buyers of white sugar have appeared in the Western Hemisphere. Peru, Jamaica, Haiti and Ecuador are countries where the sugar industry is declining. In order to fulfill their obligations towards preferential agreements such as their US quota, they have had to import white sugar. In the case of Peru, the situation is even worse, as the gap between production and consumption keeps increasing. Overall, the import potential of these countries is far from negligible. It already represents about 350,000 tonnes, and could continue growing.

"On the supply side, very few changes can be expected. The rollover policy adopted by the EEC to 'neutralize' some of the C-quota sugars (those without price guarantee) has produced a stock increase above the minimum level. In 1986/87 the carryover stocks have reached 12.3 million tonnes. Will this surplus be absorbed next year by the domestic market, as was the case in 1981/82 and 1982/83? This remains to be seen, as it would mean the voluntary reduction of domestic production, which looks uncertain.

"In Brazil, production and consumption problems during the last season do not seem to have seriously affected exports of white sugar to various preferential destinations such as Iraq, Iran, India and Nigeria. As for Cuba, now the No. 3 exporter of whites, it appears doubtful that it will be able to increase its exports in the near future; they represent 500,000 tonnes which are going mainly to Egypt, Libya, Algeria, Iraq and Syria, in the frame of long-term contracts.

"The only possible change could come from the USA if the 'drawbacks' legislation is finally adopted. This could mean, from 1988 to 1991, an increase of 200 - 300,000 tonnes per year of white sugar exports from the USA. However, the impact on the world market should be limited.

"To summarize: one can assume that, in the near future, the supply and demand for white sugar will remain balanced but, in the longer term, there is certainly hope for, and reason to believe in, a further development of this market linked to world consumption growth and a possible revival in oil prices."

Land reform in the Philippines

A factor which could seriously undermine sugar production prospects in the Philippines is the issue of land reform. In a comprehensive program covering all agricultural land the President of the Philippines announced in July details of reforms which will limit individual land holdings. Although landowners have been assured they will receive adequate compensation for their land there have been angry protests from sugar, coconut and fruit plantation owners and there have been reports of likely serious unrest should the measure be proceeded with. The original plan, which had received the backing of the World Bank, was for the large estates to be broken up and redistributed over a period of five years. More recently it has been suggested that a ten-year period is more probable.

Reforms to land management will naturally meet with opposition and could well be disruptive, at least in the initial stages. However, there has already been a substantial contraction in the scale of the Philippine industry with the government passing measures to encourage diversification away from cane cultivation into other crops. Earlier this year Executive Order No. 114 required planters to relinquish 40% of cane lands with 30% forming part of the diversification program to other crops while 10% was to be sold to sugar workers. This drew strong protests from the National Federation of Sugar Cane Planters (NFSP) but the government had linked such policies to the repayment of outstanding old debts in respect of the 1984/85 crop by Nasutra, the new defunct national trading organization.

C. Czarnikow Ltd. comment[6]: "We feel that problems could arise if large agricultural holdings were to be broken up into a great number of individual small ones. Apart from the obvious immediate lack of expertise, there would not be the financial resources available to produce the industrial and commercial infrastructure which is needed in the sugar industry. It might of course, be possible for new owners to form themselves into cooperatives for the use of machinery and resources, if this were permitted under the regulations, but this might undermine the drive which is needed if the Philippine industry is to recover from the downward spiral of the past few years."

"It may be that the land reform program will follow a different pattern from the one which has been outlined, or special provisions might be made for those crops which appear most suited for large-scale production, but certainly, in their initial stages at least, land reform programs tend not to lead to enhanced production."

New York white sugar futures contract

A new futures contract in white sugar is to be introduced on the New York Coffee, Sugar and Cocoa Exchange, with trading scheduled to start on October 5. Dealers expect it to be successful, their opinion being based generally on the trend in sugar trading towards white sugar instead of raw sugar, greater liquidity than in London or Paris, increased arbitrage opportunities, and because there are no punitive freight charges in the contract[7].

Most tenders reported lately have been for white sugar sales or purchases, and many developing countries cannot afford to refine raw sugar. One source estimated the amount of white sugar traded in the overall world market at 70%.

Dutch finance for Kenya sugar factory rehabilitation[8]

Holland is to provide a $7 million soft loan for the renovation of the East African Sugar Industries factory at Muhuroni in Western Kenya.

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[6] *Czarnikow Sugar Review*, 1987, (1763), 102.
[7] F. O. Licht, *Int. Sugar Rpt.*, 1987, **119**, 383.
[8] *Amerop-Westway Newsletter*, 1987, (163), 16.

Removal of dextran from cane juice

By S. C. Jolly and Chandra Prakash
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S. C. Jolly

C. Prakash

Introduction

The term dextran has reached prominence in the sugar industry's lexicon as the formation of this material in sugar solutions presents many problems, from slime formation in raw sugar factories to the blockage of centrifugals in the refineries. The refinery faces another problem since the presence of dextran in refined sugar renders it unsatisfactory to industrial users. Further, the deterioration of sugar cane juice and raw sugar by dextran producing bacteria can lead to serious losses. For this reason, most of the work carried out on this aspect of the sugar industry has been bacteriological.

Dextran was first considered a troublemaker to sugar manufacturers in the late 1950's in Australia where it was found to affect the rate of filtration in the refinery, to spoil mill clarification and to increase the viscosity of sugar solutions. With the introduction of the chopper-harvester in Queensland in the 1960's a significantly high percentage of dextran in cane resulted and it became a subject of research for sugar technologists.

Since the formation of dextran creates a number of processing difficulties, it is desirable to eliminate dextran as much as possible and to crush the cane with minimum possible dextran content. Methods of removal of dextran are particularly based on its enzymatic hydrolysis[1,2]. During a recent visit to Australia, we observed that one factory having a viscosity problem due to dextran had been able to reduce the viscosity by the introduction of dextranase enzyme. An enzymic method for the removal of dextran for deteriorated cane juice has been outlined which involves incubation of mixed juice with dextranase at 60°C for 40 minutes[3] whereas Tilbury & French[4] proposed incubation at 50°C for 20 minutes, which unfortunately achieved only partial removal of dextran. Fulcher & Inkerman[5] preferred mixed juice for treatment with dextranase enzyme whereas Hidi & Staker[2] preferred the addition of dextranase to syrup or molasses. According to Inkerman & James[6], the treatment of mixed juice with dextranase results in major improvements in the factory performance and sugar quality during the crushing of badly deteriorated cane. Inkerman & Riddle[7] stated that the decision to add the dextranase should be made prior to an influx of deteriorated cane, otherwise the pan stage becomes rapidly "poisoned" with dextran. The work of Inkerman also indicates that pH measurements of first expressed juice can be used as a rough guideline for deciding when to use enzyme for the removal of dextran.

Among all the processes of clarification, only carbonatation has some limited effect on the removal of dextran from the process flow in a cane sugar refinery[8]. Sharma & Johri[9] stated in their article that dextran, being soluble in water, is not eliminated during the process of clarification. Day[10] stated that the dextran produced in juice includes soluble and insoluble fractions but that conditions present in the cane juice favour the formation of soluble dextran. There are no values available; however it is estimated that as much as 50% of the dextran produced in the juice may be of the soluble kind.

It was decided to make an investigation at our factory to see if the addition of the dextranase enzyme is economically justified. The effect of heating of the juice was also studied to see whether dextran formation occurs under normal processing conditions in a modern sugar factory where the juice is not cooled during processing. The effect of the de Haan process of clarification, which is followed in our factory, on dextran content of the juice was also included in the study to observe if it is capable of removing dextran from the juice.

Materials and methods

Fortnightly analyses of the dextran content of juice using the haze method[11] were carried out in the seasons 1983/84 and 1984/85 to ascertain the range of dextran content present; the average monthly values are presented in Table I.

The optimum dose of the enzyme (Novo 25L dextranase) to be added, the optimum temperature and the optimum reaction time were determined by varying

Table I. Dextran content, mg/litre/100°Brix

	1983/84		1984/85	
	Primary juice	Secondary juice	Primary juice	Secondary juice
December	19,665	23,103	18,534	23,993
January	19,453	25,355	13,245	15,873
February	18,079	20,000	15,377	19,667
March	20,117	26,703	14,352	18,154

Table II

Expt. No.	Brix	Purity	Dextran content, mg/litre/100°Bx Before treatment A	Dextran content, mg/litre/100°Bx After treatment B	Removal (A – B)/A
1	13.84	71.97	34,682	16,474	52.50%
2	14.22	72.93	28,692	13,783	51.96%
3	14.00	72.14	24,000	12,571	47.62%
Ave.	14.02	72.32	29,101	14,265	50.98%

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[1] Tilbury: *Proc. 14th Congr. ISSCT*, 1971, 1444 - 1458.

[2] Hidi & Staker: *Proc. Queensland Soc. Sugar Cane Tech.*, 1975, **42**, 331 - 344.

[3] Fulcher & Inkerman: *ibid.*, 1974, **41**, 179 - 186.

[4] *Proc. 15th Congr. ISSCT*, 1974, 1277 - 1287.

[5] *Proc. Queensland Soc. Sugar Cane Tech.*, 1976, **43**, 295 - 305.

[6] *ibid.*, 307 - 315.

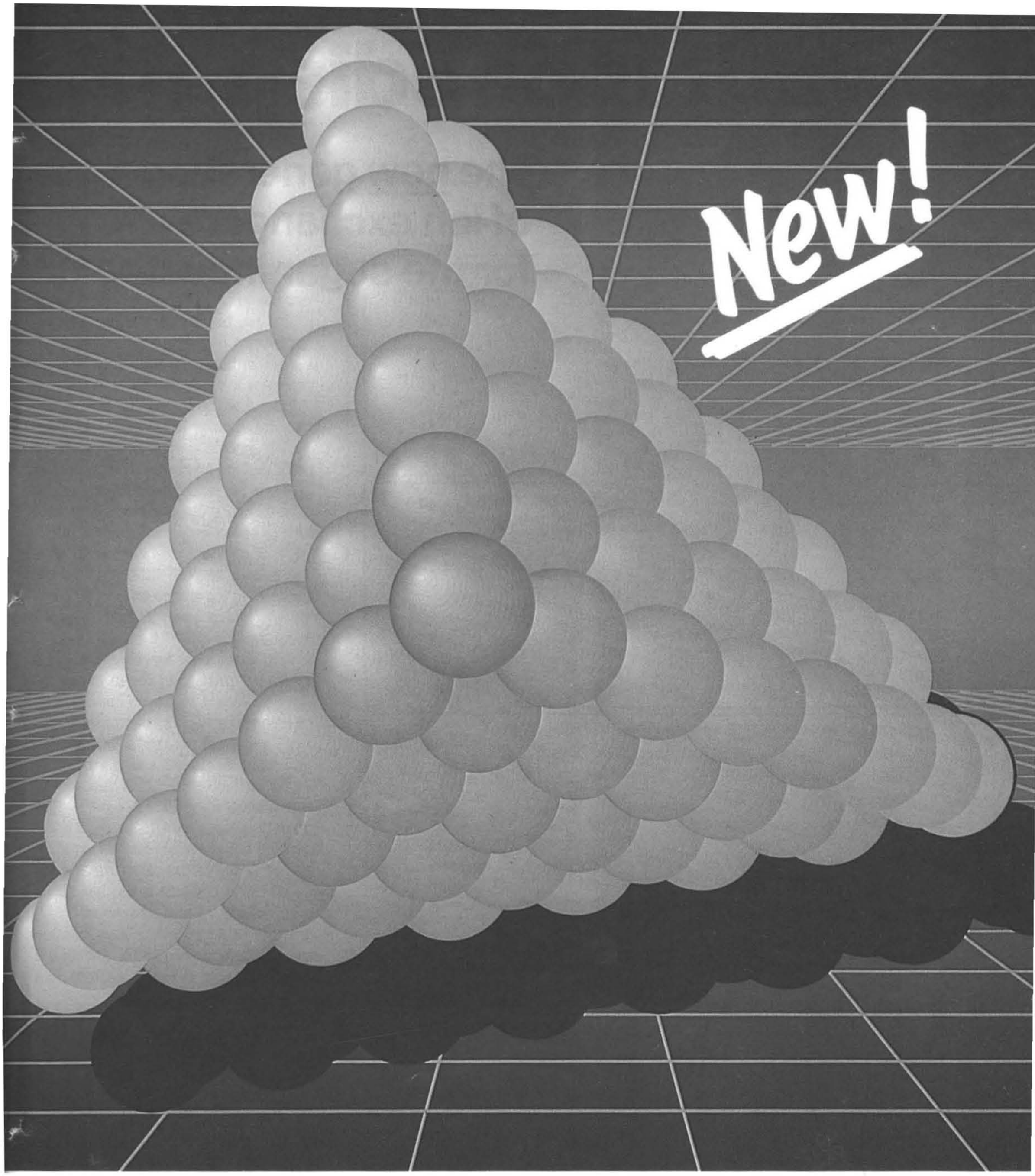
[7] *ibid.*, 1977, **44**, 215 - 223.

[8] Bose & Singh: *Indian Sugar*, 1981, **31**, 603 - 608.

[9] *Proc. 45th Ann. Conv. Sugar Tech. Assoc. India*, 1981, 123 - 143.

[10] *Sugar J.*, 1984, **46**, (10), 16 - 17.

[11] Keniry *et al.*: *I.S.J.*, 1969, **71**, 230 - 233.



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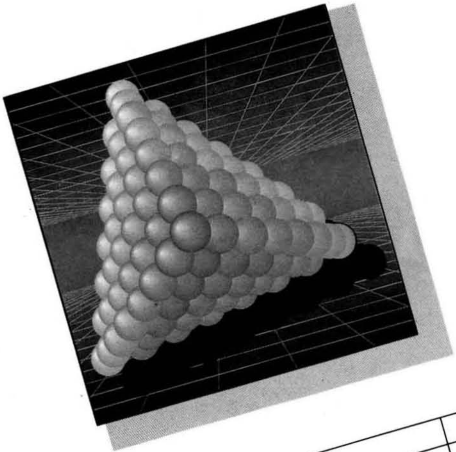

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Matrix			Polystyrene	Polystyrene	
Functional group			Quaternary amine	Sulphonic acid	
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Density		approx. g/ml	1.05	1.22	1.28
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these parameters. Subsequent experiments on the removal of dextran by the enzyme were carried out maintaining these optimum conditions and the results are presented in Table II.

To observe the effect of temperature on the dextran content of the juice, composite samples of primary juice and secondary juice were heated to 100°C for five minutes, filtered and cooled. The dextran content of the sample was determined before heating and after cooling. The juice was allowed to stand for eight hours under laboratory conditions and the dextran content again determined. The results are presented in Tables III and IV respectively.

During the 1984/85 season, fortnightly analyses were made of dextran in mixed juice samples; the juice was clarified by the de Haan carbonatation process on a laboratory scale during which the following conditions were maintained: juice temperature 55°C; lime dose added 10% milk of lime at 18°Bé; pre-liming time 3 minutes; pH of 1st carbonatation juice 10.2; pH of 2nd carbonatation juice 8.8; temperature of 2nd carbonatation juice 70°C; and pH of clarified juice 7.0.

The dextran in the clear juice thus obtained was also determined to compare it with that in the mixed juice. The average of the analyses is given in Table V.

Results and discussion

Dextran in raw juice: The dextran contents of raw juice (Table I) ranged from 13,245 to 20,117 mg/litre/100°Brix for primary juice and from 15,873 to 26,703 mg/litre/100°Brix for the secondary juice during the 1983/84 and 1984/85 seasons.

Effect of the enzyme on dextran in juice: Results of three sets of experiments showed that the following conditions were optimum for hydrolysis of dextran using the enzyme (Novo 25L dextranase):

Optimum dose to be added to the juice	100 ppm
Optimum temperature for the addition	60°C
Time required to complete the reaction	15 minutes

Table III. Effect of temperature on dextran in primary juice

Expt. No.	Dextran content, mg/litre/100°Brix A	B	C	Removal (A – B)/A
1	7,368	1,509	1,510	79.52%
2	6,120	2,363	2,363	61.39%
3	10,706	2,078	2,060	80.59%
4	7,336	1,062	1,058	85.52%
5	8,630	2,351	2,330	72.76%
6	7,784	1,743	1,743	77.61%
Ave.	8,136	1,842	1,837	77.36%

Table IV. Effect of temperature on dextran in secondary juice

Expt. No.	Dextran content mg/litre/100°Brix A	B	C	Removal (A – B)/A
1	11,609	2,250	2,248	80.62%
2	10,607	3,934	3,931	62.91%
3	11,864	2,108	2,047	82.23%
4	10,000	1,605	1,616	83.95%
5	10,543	2,345	2,368	77.76%
6	9,178	1,551	1,558	83.10%
Ave.	10,630	2,289	2,286	78.47%

Table V. Effect of clarification on dextran content in juice

	Dextran content, mg/litre/100°Bx Mixed juice A	Clarified juice B	Removal during clarification (A – B)/A
December	20,159	3037	84.93%
January	13,546	2914	78.49%
February	16,928	4182	75.30%
March	15,719	3325	78.85%
Average	16,588	3364	79.72%

Maintaining these conditions, experiments were conducted to observe the effect of addition of dextranase enzyme on the removal of dextran; the results are presented in Table II. They show that the dextran removal by the enzyme was about 48 to 52%. The cost of enzyme required to treat juice from 6000 tcd was calculated as around Rs. 200,000 (£11,000) per day which was not considered economical.

In a second phase of the investigation the effect was studied of heating of the juice to a high temperature followed by filtration in order to ascertain whether formation of dextran occurred during the processing of the juice.

Juice samples were first heated to 100°C for five minutes, filtered and cooled. The dextran content in the juice was determined before (A) and after (B) treatment and after a further eight hours (C in Tables III and IV). The analytical results of primary and secondary juices have been given in Tables III and IV, respectively. The results obtained with primary juice show that the dextran content was reduced by about three-quarters by heat treatment and that no substantial increase in dextran occurred in the next eight hours, indicating that the heating had killed the dextran-producing bacteria.

The results recorded with the secondary juice show that the dextran content in the heated juice was reduced by about the same extent as with primary juice and that it also did not increase appreciably during the subsequent eight hours. Further, the removal of dextran on heating the juice to 100°C followed by filtration suggests that a portion of dextran undergoes some unidentified chemical change, perhaps to an insoluble form or coagulated with proteins, so that it may be filtered off.

Effect of the de Haan clarification process on dextran in juice

The third phase of this study was to determine the effect of clarification on the dextran content of the juice. The average monthly analyses of dextran in mixed juice during the 1984/85 season are presented in Table V. They show that the average dextran content in 1984/85 was 16,588 mg/litre/100°Brix in the raw juice and 3,364 mg/litre/100°Brix in the clarified juice which indicates 79.72% removal of dextran during the clarification of the juice, confirming the observations recorded on heating the juice followed by filtration. It is clear that any insoluble dextran in the juice will be filtered off during clarification as is part of the soluble dextran rendered insoluble by some unidentified chemical change or coagulation with protein present in the juice. Thus, it may be concluded that the process of manufacturing sugar inhibits the formation of dextran, provided that the process of clarification is strictly under control. However, Honig[12] has reported that there may be formation of a "dextran capsule" during the process of sugar manufacture; this capsule is a thick slime layer around the cell wall of the organism which serves as a protective layer against adverse effects of heat and chemicals.

The next step in this work will be the addition of dextranase enzyme to the clarified juice or syrup to remove the remaining amount of dextran; this should reduce the processing difficulties caused by dextran while being more economical than raw juice treatment, since less enzyme will be required.

Summary

The dextran content ranges from 13,245 to 20,117 mg/litre/100°Brix in the primary juices at Saraswati sugar factory in India and from 15,873 to 26,703 mg/litre/100°Brix in secondary juices. Laboratory experiments indicate that 100 ppm of Novo 25L dextranase enzyme can hydrolyse about 51% of dextran present originally. On the other hand, it is possible to remove some 61 to 85% of dextran from the raw juice by heating it to 100°C followed by filtration which indicates that a major portion of dextran will be eliminated during the process of clarification. During tests of the effect of the de Haan process of carbonatation on the dextran content of the juice under laboratory conditions, it was observed that about 75 to 85% of the dextran content was eliminated.

Acknowledgement

Thanks are due to Mr. Ranjit Puri, Joint Managing Director, The Saraswati Industrial Syndicate Ltd., Yamunanagar, for his keen interest, encouragement and guidance during the investigation and his kind permission to present this paper. The authors are also grateful to Mr. R. P. Mittal, Deputy General Manager, Mr. O. P. Chhabra, Chief Engineer (Development), and Mr. A. C. Kudsia, Chief Chemist, for their valuable suggestions during the study.

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[12] "Principles of sugar technology", Vol. III. (Elsevier, Amsterdam) 1963, 631.

ANALYSIS

HPLC analysis of sugars in sugar cane stalks

By Danielle A. Celestine-Myrtil and A. Parfait

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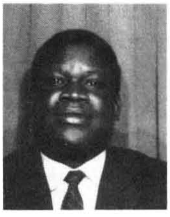
Introduction

On the islands of Guadeloupe and Martinique in the French West Indies some alcoholic beverages, mainly rum, obtained by fermentation and distillation, are manufactured directly from sugar cane juice[1]. Sugars and the non-sugar components of the juice influence the yeast and bacterial fermentation, and affect the aromatic profile of the rum[2-5].

D. A. Celestine-Myrtil



A. Parfait

The composition of sugar cane juice depends on cane varieties, cultivation conditions, climatic environments, cane maturity, harvesting methods, juice extraction technique, etc., and, in order to improve the quality of fermented products from sugar cane, a careful understanding of its composition was necessary. Accordingly, our researches were first made on the distribution of sugars in the cane stalk.

After a literature review it appears that, although sugar cane has been widely studied, little work has been done on sugars distribution in joints of sugar

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[1] Parfait: *Kemia-Kemi*, 1983, **10/11**, 982 - 983.

[2] Fahrasmane *et al.*: *Ind. Alim. Agric.*, 1983, **100**, 297 - 301.

[3] Idem: *J. Food Sci.* 1985, **50**, 1427 - 1430.

[4] Lencrerot *et al.*: *Ind. Alim. Agric.*, 1984, **101**, 763 - 766.

[5] Parfait & Jouret: *Ann. Technol. Agric.*, 1975, **24**, (3/4), 421 - 436.

cane stalks (nodes and internodes). The most recent on this topic came from van Dillewijn[6] and Alexander[7] after Ramaiah & Varahalu[8]. They gave Brix distribution in a sugar cane stalk.

In physiological research the relationships between enzymatic activities and sugar accumulation in sugar cane, sucrose and total reducing sugars distribution in the internodes of sugar cane stalks have only been investigated using colorimetric or enzymatic methods[9-12]. Classical optical measurements (polarimetry, Brix, colorimetry, etc.) incur interferences with many compounds other than sugar. Enzymatic methods are time-consuming, because the same sample has to be analysed three times consecutively to get results for sucrose, glucose, and fructose separately.

Recent developments in high pressure liquid chromatography (HPLC) have enabled the rapid and reliable analysis of mixtures of sugars with simple sample preparation. Many compositions of sugars have been determined by HPLC in fruits[13], in beets[14], in commercial drinks[15], in fermentation media[16-18] and in sugar cane juice[19-21].

The main purposes of this work were: the development of a rapid and reliable HPLC separation method by determining retention time reproducibility, linearity, detection and measurement limits for the sugars analysed (sucrose, glucose, fructose); the optim-ization of sugars extraction from ground sugar cane samples (duration and volume); and the quantitative investigation of sugars accumulation in the nodes and internode section of sugar cane stalks.

Material and method

Sampling

One stalk each of two commercial varieties of twelve months-old B 47258 and B 69566 sugar cane were harvested from fields of the Centre Technique de la Canne et du Sucre (C.T.C.S.) in March 1986 in Guadeloupe. The stalks were cut as near as possible to the soil and their green tops were retained although the leaves were taken off. The nodes were numbered from the base of the stalk considering the lowest as number one. Internodes were numbered accordingly. The stalks were first washed to remove foreign matter, then sawn to separate nodes and internodes. These were then weighed, ground and frozen separately before analysis.

Dry matter determination

5g each of ground duplicate samples were taken, dried in an oven at 80°C during 48 hours and weighed again.

Sugar extraction and purification

Extraction of sugars from the weighed sample was carried out with distilled water at 80°C in stoppered Erlenmeyer flasks. Different volumes and durations of extractions were studied. The extracts obtained were purified by passing through a Waters Sep-Pak C-18 column. Retention of sugars on the column was investigated. The extracts were then filtered through 0.22 µm Millipore filters. The samples ready for HPLC analysis were reprocessed filtrates.

HPLC method

A Waters Sugar Analyser I liquid chromatograph equipped with a Waters R 401 refractive index detector on calibre 8X coupled to a recorder-integrator (Waters Data Module) was used for this work. The injection valve was a Waters Rheodyne model with a 20 µl loop, and the column used was a 4 mm i.d. × 300 mm Waters Sugar Pak I cation exchange column in Ca^{++} form. Column temperature was 85°C, and the eluent was 0.0001 M calcium acetate in distilled water filtered through 0.45 µm filters under vacuum. The mobile phase flow rate was 0.5 ml/min, and quantitative analysis was made by comparison of test results with those given by external standards; the standard solution was prepared with: 60 g/litre sucrose, 12 g/litre of glucose, and 12 g/litre fructose.

This solution was diluted with water to determine measurement and detection limits, linearity and repeatability for the different sugars considered.

Results and discussion

HPLC method characteristics

Sucrose, glucose and fructose were base-line separated in less than 15 minutes (Figure 1).

Detection and measurement limits

Good base line was reached at a setting of 8.10^{-5} RI units.

Detection limits of the system given in concentration or in quantity for an injection volume of 20 µl are:

Sucrose 10 mg/litre i.e. 0.2 µg
Glucose
or fructose 30 mg/litre i.e. 0.6 µg

Measurement limits were:

Sucrose 100 mg/litre i.e. 2 µg
Glucose
or fructose 200 mg/litre i.e. 4 µg

Response detector linearity

The detector response to different concentrations of each sugar was linear throughout the range of concentrations found in the injected samples of sugar cane.

Linear regression equations between area and sugars concentrations were:

Sucrose: $y = 67.297x + 1.01$
(correlation coefficient 0.999)
Glucose: $y = 63.802x + 1.74$
(" " ")
Fructose: $y = 63.820x + 1.61$
(" " ")

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[6] "Botany of sugar cane", (Chronica Botanica Co, Waltham, Mass), 1952.

[7] "Sugar cane physiology", (Elsevier, Amsterdam), 1973.

[8] *Madras Agric. J.*, 1938, **26**, 458 - 464.

[9] Hatch & Glasziou: *Plant Physiol.*, 1963, **38**, 344 - 348.

[10] Devi *et al:* in "Advances in photosynthesis research" Vol. IV, Ed. Sybesma (Martinus Nijhoff/Dr. W. Junk), 1984, 7.881 - 7.884.

[11] Matsui: *Tech. Bull. Fac. Agr. Kagawa Univ.*, 1985, **36**, (2), 117 - 125.

[12] Idem: *J. Japanese Soc. Food Sci. Technol.*, 1985, **32**, 655 - 660.

[13] Shaw & Wilson: *J. Sci. Food Agric.*, 1983, **34**, 109 - 112.

[14] Mulcock *et al.: I.S.J.*, 1985, **87**, 172 - 175, 188 - 194, 203 - 207.

[15] Vidal-Valverde *et al.*: *J. Sci. Food Agric.*, 1985, **36**, 43 48.

[16] Duarte-Coelho *et al.: J. Liquid Chromatogr.*, 1985, **8**, 59 - 73.

[17] Morawski *et al.: Sugar y Azúcar*, 1983, **78**, (2), 170 - 177.

[18] Tannock: *Sugar J.*, 1985, **47**, (8), 21 - 25.

[19] Abeydeera: *I.S. J.*, 1983, **85**, 300 - 306.

[20] Charles: *ibid.*, 1981, **83**, 169 - 172, 195 - 199.

[21] Wong-Chong & Martin: *Louisiana Agric.*, 1982, **25**, (3), 21 - 24.

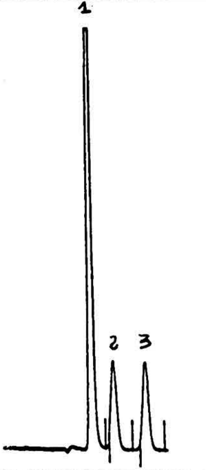

Fig. 1a. Chromatogram of sugars standard solution

Fig. 1b. Sample chromatogram: 1. Sucrose, 2. Glucose, 3. Fructose (Column: Sugar Pak I; Eluent: 20 mg/litre calcium acetate; Flow rate: 0.5 ml/min; 85°C; 800 p.s.i.)

with sucrose varying from 0.1 to 20 g/litre, glucose from 0.2 to 4 g/litre, and fructose from 0.2 to 4 g/litre.

Method repeatability and precision

In order to examine the precision of the method, ten repeated injections of ten dilutions of the standard sugars solution were made. The diluted standard solution contained 6.0 g/litre sucrose 1.2 g/litre glucose and 1.2 g/litre fructose. The results of these determinations are presented in Table I.

Table I. Precision of sugar analysis by HPLC

Sugars	Mean area x	Standard Deviation σ_{n-1}	Precision (RSD) $\sigma_{n-1} . t / \sqrt{n} . x$
Sucrose	271.994×10^6	1.94	± 0.6%
Glucose	66.324×10^6	1.67	± 2.2%
Fructose	64.649×10^6	1.14	± 1.6%

n = 10; t = 2.262 with 95% confidence limits[22]

The characteristics of the HPLC method are in good agreement with those found by Vidal-Valverde *et al.*[15] with the same column. Detection and measurement limits which define method sensitivity are better than those determined by Abeydeera[19] on a Shodex column but poorer than those obtained by Tannock[18] using a Sugar-Pak I column. Generally speaking, however, the HPLC method developed here was considered satisfactory.

Sugar extraction and purification

Extraction volume: Extraction was carried out on dried ground samples, but the variations due to extraction volume were established using a range of 6 to 13 for the ratio R, where R = water volume added (ml)/fresh sample weight (g). Duration of extraction was 30 minutes and results obtained are shown in Table II.

R values of 6.66 and 8.33 do not permit total sucrose extraction while, for values of 10, 11.66 and 13.33, extraction seems to be complete. No important variation was noticed for sucrose concentration for values of R between 10 and 13.3. For this reason, during sugar cane analysis, R was fixed at 10.

Table II. Effect of volume variation on sugars extraction (mg/g fresh weight)

R	Sucrose	Glucose	Fructose
6.66	60.5	9.7	8.7
"	58.7	9.6	8.6
8.33	61.8	10.0	8.2
"	61.2	9.2	7.8
10.00	64.7	10.2	9.0
"	64.0	9.9	8.5
11.66	64.5	9.4	8.1
"	63.0	8.2	7.9
13.33	63.9	9.4	7.9
"	64.0	10.1	8.5

Extraction duration:

Different durations of extraction from 0 to 30 minutes were tested with the same sample, using a ratio R = 10 and at 80°C. Values obtained are given in Table III.

Table III. Effect of duration variation on extraction, mg/g fresh weight

T, min.	Sucrose	Glucose	Fructose
0	67.4	10.5	9.0
"	67.1	10.6	9.0
5	67.1	10.6	9.3
"	67.2	9.9	8.7
10	68.8	10.3	8.0
"	68.2	10.4	8.7
15	69.7	10.5	8.6
"	72.0	10.6	9.4
"	71.0	10.6	9.2
"	69.9	11.0	9.2
20	67.3	10.0	8.8
"	66.9	9.7	7.5
30	68.8	10.4	8.8
"	67.9	10.0	8.5

The best sucrose extraction results were obtained for a duration of 15 minutes; consequently this was chosen for stalk analysis.

Extraction efficiency: Three successive extractions were made on the same sample with R = 10 and T = 15 minutes at 80°C. The results of this experiment are shown in Table IV.

The first extraction gave about 84 - 93% of total sugar depending on the sugar considered, as was found by Mulcock *et al.*[14] with sugar beet.

It appears that sugars present in small quantity (glucose, fructose) are better extracted (>90%) than sucrose in the first run. Only one extraction will be done in sample analysis, because the

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[22] Lacroix: *Mem. Soc. Chim. France*, 1963, **5**, 892 - 898.

ISJ Abstracts

Cane sugar manufacture

Separating out the soil: rotary filter operation a matter of decision

P. Atherton. *BSES Bull.*, 1986, (16), 22 - 23.

In a discussion of the main factors influencing filter performance, it is emphasized that increase in the amount of soil entering a factory, e.g. when cyclone-damaged cane is harvested as in north Queensland in 1986, places an increased load on the clarifiers and makes good filtration difficult to achieve. The available filter area and the mud solids loading are two major factors; 0.55 m^2 per tonne of cane/hr is considered adequate for all but abnormally high loadings. Optimum operation lies probably somewhere between the two extremes of a low speed of 7 - 8 rpm with a thick cake (e.g. 12 mm) and a speed of 15 - 16 rpm with a thin cake of 6 mm, but only experience at the factory in question will indicate the most suitable conditions; a thin cake is preferable for pol loss minimization, whereas thick cake tends to increase losses but does reduce the quantity of solids recirculated through the clarifiers by increasing the retention efficiency. Because of this conflict, selection of ideal conditions is only possible on the spot and is often difficult. An adequate bagacillo supply is essential for good filtration, generally enough being needed to give a fibre:mud solids ratio of 0.35 - 0.40; a higher ratio (e.g. 0.50 - 0.60) gives a thicker filter-cake of poor washing properties and resultant higher losses. Increase in bagacillo fineness improves retention, mud solids output and pol loss at a constant wash water consumption % cane. For low pol losses of e.g. 0.3 - 0.4% pol on cane, about 200% water on cake is required at normal mud solids loadings; a flowmeter should be installed in the wash water line to ensure that the water is being applied at a required rate. Uniform application along the drum of each filter is essential, and the best means of achieving this is by using a series of dribblers or sprays. Following periods of wet weather it is usually far more important to maintain a high mud output from the filters than it is to aim for a low pol loss; under these circumstances, wash water consumption is often reduced to below the desired level because of the decreased permeability of the heavier cake. Maintenance of filtration equipment in a satisfactory state of repair is vital for an acceptable performance level, and the main items requiring attention are listed.

The direct cost of corrosion in sugar factories and the savings that can be made by adoption of best corrosion control measures

S. S. Kumar and K. S. Rajagopalan. *Indian Sugar,* 1986, **36,** 343 - 356.

The costs of corrosion of each individual piece of equipment in a sugar factory of 1250 tcd capacity are calculated and the cost benefits of corrosion prevention measures indicated.

Microcrystalline sugar drying with a spouted bed

H. C. Tso, C. H. Chen and R. Y. Chang. *Rpt. Taiwan Sugar Research Inst.*, 1986, (113), 9 - 18.

A spouted bed, consisting of a 1.5 m high plastic column 18.3 cm in diameter and with a conical base, was used in an investigation on continuous drying of microcrystals of sugar. The sugar was fed at the top of the column by a vibratory unit and hot air was injected into the bottom of the column; the sugar was entrained by the air and fell after travelling a certain height. A moisture removal rate of 0.06 kg/kg dry solids/ min was achieved at a sugar moisture content of 2 - 3%, a relative humidity of 40% and a bed temperature of 55°C. The gas-to-particle heat transfer coefficient was 3000 - 5000 kcal/m^3/hr/°C, and a dead space of 24% was found in the bed.

Treatment of the waste stream from a sugar mill by *Azotobacter*

Y. T. Chuang. *Rpt. Taiwan Sugar Research Inst.*, 1986, (113), 29 - 39.

In laboratory studies on sugar factory effluent treatment with *Azotobacter* AP8a, 97 - 98% BOD removal was achieved after 24 hours at an initial level up to 3000 ppm, after which there was a sharp fall in performance with increased initial BOD to 6000 ppm. Continuous treatment of waste water containing 1500 ppm BOD with a mixture of 70% *Azotobacter* cells and 30% ordinary activated sludge gave an average BOD removal of 85% and 95% after 16 and 24 hours, respectively. No nitrogen source was added during treatment.

Clarification of Egyptian cane raw juices. I. Evaluation of continuous liming-sulphitation and cold liming processes

M. M. B. El Sabbah and A. Z. Abd El-Latif. *Zuckerind.*, 1986, **111,** 1135 - 1138.

Determination of CaO, P_2O_5, (Fe_2O_3 + Al_2O_3), SO_3, SiO_2, N, MgO, K, Cl, reducing matter, waxes and gums in raw juice treated by laboratory cold liming followed by heating to b.p. showed that the impurities contents increased during the campaign. Continuous liming + sulphitation used at Abu Kourkas was found to be less effective than liming + heating in regard to the removal of waxes, gums and nitrogenous compounds but was better in removal of Fe, Al, Mg, and P compounds.

Measuring vacuum pump performance

R. A. H. Chilvers and D. J. Love. *S. African Sugar J.*, 1986, **70,** 417 - 420.

See *I.S.J.*, 1987, **89,** 89A - 90A.

Strategies for developing the sugar industry in Indonesia: resources utilization and technology transfer

B. Hutabarat *et al. Gula Indonesia,* 1986, **12,** (3), 21 - 26, 35 - 54.

A survey is presented of the performance of the Indonesian sugar industry and the practicalities of establishing new sugar-producing areas are considered.

Comparison is made between the older established industry in Java and the newer ones set up in other parts of Indonesia. Agricultural, socio-economic and technological aspects are covered.

Development of micro-processor controls for bagasse-fired furnaces

W. Keenliside and K. McGrew. *J. Amer. Soc. Sugar Cane Tech.*, 1986, **6,** 82 - 90.

A system for automatic control of combustion in a bagasse-fired furnace is described which was tested on a 50 short tons/hr unit equipped with a spreader stoker and in which the undergrate air (the only controllable source and providing 75 - 85% of the total combustion air) was preheated and the system driven by a 100-hp steam turbine. The bagasse was fed by four chutes, each with its own variable-speed chain conveyor. Details are given of the principal sensors in the control system (oxygen analyser, an orifice plate and differential pressure cell for steam flow, a pressure cell for steam, and thermocouples for temperature measurement) and of their operation. Maximum steam generation and peak combustion efficiency were ensured by a fuel control loop and excess air (forced draught) loop; because of the slowness of response of the boiler to changes in fuel flow, steam flow and pressure were combined to give a measure of the required total heat output. Trial results demonstrated the ability of the control system to respond to load changes over a limited range of conditions.

1984 ASI milling studies

H. S. Birkett, S. J. Clarke, W. Keenliside, J. A. Polack and J. Stein. *J. Amer. Soc. Sugar Cane Tech.*, 1986, **6,** 91 - 101.

Results of milling tests conducted at eight factories in Louisiana were analysed on the basis of cane and bagasse pol and moisture contents and juice pol and refractometric Brix; samples were collected as frequently as possible over 1-hr periods. The data are tabulated and predictions made (based on average parameter values) of the effects of some of the more important variables on mill performance. Two recommendations are made whereby extraction could be greatly improved: application of more imbibition water than is the normal practice in Louisiana or Florida, and greater attention to the moisture in bagasse leaving *all* of the mills in the tandem, not just that from the last mill.

Time-temperature studies – crystallization from final molasses

M. Saska and J. P. Garandet. *J. Amer. Soc. Sugar Cane Tech.*, 1986, **6,** 121 - 125.

A computer model based on an empirical equation for crystal growth rate in impure solutions[1] is aimed at optimum low-grade massecuite cooling to provide the best possible exhaustion within a given time (according to the size of crystallizer and process rate), to minimize losses in the centrifugals and to improve crystal size uniformity. The effect on molasses exhaustion of a considerable spread in massecuite retention time in the crystallizers at four Louisiana factories appeared to be negligible, while the influence on measurement of the sucrose solubility coeffficient for molasses from Florida, Louisiana and Texas of the reducing sugars:ash ratio at relatively low values (of the order of 2.5) of the ratio between impurities and water contents was also found to be small, as found in other countries. The aim was to derive a target purity formula suited to local conditions that would replace the South African formula currently used in the US. A study of the effect of crystal content on final molasses exhaustion showed that massecuite viscosity could be effectively reduced by dilution with molasses or by pre-spinning to remove some of the crystals and recycling the mother liquor.

Dextran control – management is the key

J. A. Polack. *Sugar Bull.*, 1986, **65,** (6), 7.

Four areas in which good management will minimize the dextran problem are: cane harvesting, cane reception (with proper scheduling to ensure that the earliest cane is processed first), factory sanitation and sugar storage (where high-dextran may be blended with low-dextran sugar to avoid penalties). Each of these areas is briefly discussed and the effect of weather conditions on dextran levels also mentioned.

A review of experience with continuous vacuum pans in Tongaat-Hulett Sugar

P. W. Rein. *S. African Sugar J.*, 1987, **71,** 23 - 29.

See *I.S.J.*, 1987, **89,** 28 - 34.

Impact of sugar cane area and production on the duration of cane crushing in Bihar

V. P. Singh and B. V. S. Sisodia. *Indian Sugar Crops J.*, 1986, **12,** (1), 1 - 3.

A study is made of relationships between some important economic variables of cane growing and processing, and of the degree of influence of cane area and cane production on the crushing period in a major sugar-producing state of India.

Clarification of vacuum filter filtrate

R. Lokan. *Bharatiya Sugar*, 1986, **11,** (14), 13 - 14.

Problems caused by clarifying filtrate from the rotary mud filters together with raw juice and by the high volume of clarifier mud resulting from very dirty cane led to the decision to clarify the filtrate on its own at a factory in Guadeloupe. A short-retention clarifier was modified for the purpose, and the result was an improvement in clarifier and filter performance. Mud remaining in the bottom of the clarifier was also sent to the rotary filters at liquidation, whereas previously the absence of a filter-press meant that it could not be treated.

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[1] Wright & White: *Proc. 15th Congr. ISSCT*, 1974, 1546 - 1560.

Beet sugar manufacture

Experiences in sugar industry waste water treatment in lagoons

I. Toókos. *Cukoripar*, 1986, **39**, 153 - 157 *(Hungarian).*

Results obtained in lagooning of sugar factory effluent in Hungary are discussed and compared with those achieved in other countries, including Poland, West Germany and the USA. Factors determining the level of degradation of organic matter and the amount of water treated are examined.

On sugar boiling and energy

J. G. Ziegler. *Sugar J.*, 1986, **48**, (12), 5 - 8.

The author shows, by reference to case histories, that many of the measures proposed for reducing heat requirements in the pan station ignore basic requirements of the boiling process and of pan operation; consistently good boiling aimed at obtaining a high yield of good quality crystals per strike will save more energy than most of the schemes suggested. The disadvantages of various practices are explained.

Sugar extraction from beet cossettes in vessels with open electrodes

V. A. Zaets, L. G. Vorona, M. P. Kupchik, A. B. Matvienko and T. A. Mikhailik. *Elektron. Obrab. Mater.*, 1986, (3), 57 - 59; through *Ref. Zhurn. AN SSSR (Khim.)*, 1986, (23), Abs. 23 R585.

Single-pass trials were conducted under factory conditions in an experimental diffuser to determine the effect of an electric field on sugar extraction from cossettes. Extraction with open electrodes was found to be possible, which considerably simplifies the design aspects of the process equipment.

Electrical heating of low-grade massecuite

Yu. M. Zhurbitskii, A. V. Karpenko and V. T. Garyazha. *Sakhar. Prom.*, 1986, (11), 10 - 12 *(Russian).*

A resistance heater is described that in tests reheated massecuite of 91°Bx from approx. 41° to 50°C at a throughput of 14 tonnes/hr, a voltage of 380 V and a power consumption of 3.1 kWh/tonne. Reheating reduced molasses purity and raised white sugar yield by 0.04% on beet by comparison with the absence of reheating. Massecuite fed under gravity above the sloping wall acting as deflector at one end of the heater flowed along between vertical partitions running from top to bottom of the heater (these partitions and the external side walls acting as electrodes) and was discharged through ports on each side at the two bottom corners furthest from the feed end.

Removal of saponin from waste water by electrocoagulation using aluminium electrodes

V. V. Sakhnenko and V. V. Stetsenko. *Sakhar. Prom.*, 1986, (11), 20 - 23 *(Russian).*

An experimental unit consisting of a glass column was used in investigations (after laboratory studies) in which waste water was fed at the bottom and passed up between four aluminium electrodes; the metal ions from these formed flocs of hydroxide with the saponin. In the upper section of the column other impurities in the water were adsorbed on the surface of other newly formed flocs and were carried to a discharge port towards the top under the action of upward flow of the water and of gas bubbles formed at the cathode. At a flow in the range 0.125 - 1.25 litres/min (corresponding to a residence time of 2 - 20 min), a current density of 37 - 111 A/m^2 and an aluminium consumption of 62% on weight of saponin, the COD was reduced from 2400 mg/litre by an average of 83% and the saponin content from approx. 160 mg/litre by 84%.

Analysis of causes of sugar factory stoppages

A. I. Slyn'ko and V. D. Naumenko. *Sakhar. Prom.*, 1986, (11), 23 - 25 *(Russian).*

Reasons for unscheduled stoppages in Ukrainian sugar factories in 1983 and 1984 are analysed. The average length of the stoppages was about 5 hours, and by far the greatest number involved diffusers.

Filter-cake sweetening-off on a rotary vacuum filter using jet-cavitation sprayers

P. M. Barabanov, A. F. Nemchin and V. D. Novoseletskii. *Sakhar. Prom.*, 1986, (11), 25 - 28 *(Russian).*

A patented sprayer tested on filter-cake discharges a jet of water made up of uniform finely dispersed micro-streams formed from fine bubbles created by cavitation of pressure-fed water. In the trials, the unit gave lower cake losses than a film sprayer and at reduced water consumption.

Prospects of developing condensers for sugar factories

V. N. Gorokh, A. I. Sorokin, B. F. Us and K. O. Shtangeev. *Sakhar. Prom.*, 1986, (11), 31 - 33 *(Russian).*

The principal factors affecting condenser performance are discussed, including the specific contact surface area in mixing condensers. Possible paths to follow in improving these are suggested, and the potential of air-cooled surface condensers is indicated.

The effect of lime burning and slaking conditions on raw juice purification

L. D. Shevtsov, L. M. Verchenko, B. N. Valovoi and R. Ts. Mishchuk. *Sakhar. Prom.*, 1986, (11), 34 - 36 *(Russian).*

Investigations showed that thick juice lime salts content and colour were highest and 1st carbonatation juice filtration the most difficult when lime of high dispersion and high activity was obtained by burning at 750°C and slaked with water at 80°C. Improvement in juice treatment was achieved by slaking coarse lime of low activity (obtained at 1250°C) with water at 80°C or by slaking the other lime at 20°C. The temperature effect was most noticeable on the filtration coefficient.

The effect of slimy bacteriosis on the technological properties of sugar beet and its processing

Yu. D. Golovnyak *et al. Sakhar. Prom.*, 1986, (11), 37 - 42 *(Russian).*

Examination of the chemical composition and processing properties of beets at various stages of infection by slime-forming bacteria showed that they would be suitable for sugar extraction only if processed in a 1:9 ratio along with healthy beets. Laboratory and factory trials on the treatment of diffusion juice with dextranase indicated that it is possible to process a larger proportion of affected beet. Optimum conditions for use of the enzyme were 55°C, pH 5 - 6, dosage of 5 mg/litre at a dextran content of 200 mg/litre (and pro rata) and a treatment time of 10 min. Treatment increased 1st and 2nd carbonatation juice filtration and settling properties.

One mathematical approach to appraisal of the use of mechanization in the beet yard of a sugar factory

I. S. Tkachenko and V. A. Tkachenko. *Sakhar. Prom.*, 1986, (11), 42 - 45 *(Russian).*

A micro-computer program is described for evaluation of beet yard mechanization.

Forced circulation in vacuum pans

M. Tahiri. *Sucr. Maghrebine*, 1986, (28), 37 - 44 *(French).*

After discussing aspects of massecuite stirrer operation, the author describes the Air Gut system (marketed by Extraction De Smet S.A.) under which preheated compressed air is injected in the bottom of the pan and causes accelerated upward movement of the massecuite as well as improved heat transfer. Details are given of trials in Spanish beet sugar factories in which the system increased massecuite crystal yields and reduced colour formation. Some 40 of the systems have been installed in eight Spanish factories, and others have been introduced in Cuban factories, in Denmark and at Donstiennes factory in Belgium.

A novel sugar silo, the Colleville underground silo

R. Sambussy. *Sucr. Franç.*, 1986, **127**, 329 - 335 *(French).*

Details are given of the design and construction of a 62 m long, 32 m wide bulk sugar silo built at Colleville sugar factory on a site carved out of a chalk hillside in the form of a terrace 15 m above the road. The pitched roof and low side walls stand above ground on a level platform, while the underground part of the silo has an inverted conical cross-section that is a mirror image of the roof section, so that the pile of sugar (up to 22,000 tonnes) has a diamond-shaped cross-section with a maximum height between apices of 32 m. Sugar is fed by horizontal conveyor housed in an aerial tunnel straddling the road and enters via a tower at the end of the silo at rooftop level; it is reclaimed via a horizontal conveyor at the base of the silo which transfers it to an elevator in the tower from which the sugar is carried by the feed conveyor in reverse.

Trial on a vibrating-plate sulphitation vessel

M. T. Ibragimov *et al. Sakhar. Prom.*, 1986, (12), 27 - 29 *(Russian).*

A vertical sulphitation vessel with horizontal perforated plates that vibrate gave a 2nd carbonatation juice SO_2 utilization efficiency of 99.1% as against 80.2% in a conventional vessel.

Different sugar house concepts and their effect on energy consumption

R. F. Madsen. *Zuckerind.*, 1986, **111**, 1121 - 1126 *(German).*

The consumption of energy in the sugar house for evaporation of water and in the form of electricity is discussed and the typical sugar house scheme used in Denmark is described; this involves mixing together of *A*- and *B*-sugar (both of 99.8°Bx and 100 purity) for use as direct-consumption white sugar, whereas only *A*-sugar is used in the conventional West European scheme. The advantage of the Danish scheme lies in the saving of water that is used for *B*-sugar melting and for washing of the resultant respun sugar for production of white sugar. This is demonstrated by two examples of each scheme in which the thick juice purities are 92 and 94. The basic requirements of the Danish scheme are listed and the effect of *B*-massecuite Brix (90, 91 and 92°) at constant purity of 89.5 on the sugar house scheme is indicated, whereby run-off purity rises by about 2.5 units per unit fall in Brix. Centrifugal washing schemes are discussed and four systems compared: (1) washing of *A*- and *B*-sugar with water only at 3.5% and 5% on massecuite, respectively, (2) washing with *C*-sugar melt plus water at 4% for *A*-sugar and 8% for *B*-sugar, (3) washing with run-off from the same product after dilution to 70°Bx and then with water, at 5% run-off for *A*-sugar and 10% for *B*-sugar, and (4), washing with run-off as in (3) but followed by the use of *B*-sugar melt in the case of *A*-sugar and of *A*-sugar melt for *B*-sugar treatment. Method (3) is the standard system in Denmark and uses only 65 - 67% of the water consumed in (1); although (4) gives a sugar quality and colour that are the same as in the other methods, it makes it difficult to keep the ash content, particularly in *B*-sugar, as low as in the other systems. However, it does have a number of advantages (in addition to the lowest water consumption) and is used by one factory.

Pulp pressing additives

P. Mottard and A. Carrière. *Zuckerind.*, 1986, **111**, 1128 - 1135 *(German).*

See *I.S.J.*, 1987, **89**, 39A.

A steam contact heater

P. Hoffman. *Listy Cukr.*, 1986, **102**, 240 *(Czech).*

A heat exchanger utilizing low-pressure

vapour from the pan station or from the last evaporator effect is used to raise the temperature of water for use in e.g. diffusion from e.g. 60 - 70°C to 78 - 90°C as at Modrany sugar factory or from approx. 9°C to 65 - 70°C as at Kostelec. The water is top fed and flows down through a series of plate and ring sections, while the vapour enters at the side of the vessel beneath the lowest section.

The risk of frost damage

M. Martens. *Le Betteravier*, 1986, **20**, (213), 13 *(French)*.

The risk of frost damage to beets piled in field clamps, the nature of the damage and the adverse effect of the resultant degradation products on limed juice filtration are briefly discussed.

Effect of carbocalcium saccharates on the removal of colouring matter

I. F. Bugaenko and E. P. Sklyar. *Sakhar. Prom.*, 1987, (1), 19 - 20 *(Russian)*.

In laboratory studies of the possible effect on colorant removal of saccharates formed during carbonatation, 50% molasses solution was added to a limed sucrose solution at five different stages in the process, all other parameters being constant. Results showed that the saccharates formed in the initial stages of gassing with CO_2 subsequently broke up with the formation of crystalline $CaCO_3$, so that they contributed nothing further to the purification process.

The use of microwave heating to obtain a seed suspension

L. I. Trebin, L. A. Kupchik and A. P. Lapin. *Sakhar. Prom.*, 1987, (1), 22 - 23 *(Russian)*.

Comparison was made between microwave heating and convection heating using an electric plate as a means of evaporation in the production of seed crystals. Results showed that, under identical controlled conditions, the nucleation rate with the use of the microwave system was more than double that using the other system, while the mean crystal size was 27 - 37% lower, with greater uniformity and a smaller quantity of conglomerates.

Application of high-performance liquid chromatography to evaluation of the efficiency of new techniques for sucrose extraction and purification

N. U. Fishchuk, V. V. Mank, M. P. Kupchik, V. Ya. Davydov and G. N. Filatova. *Sakhar. Prom.*, 1987, (1), 25 - 27 *(Russian)*.

HPLC using a Cheminert chromatograph with a 250 mm x 4.8 mm column filled with LiChrosorb Si-60-10 was employed in separation and quantitative analysis of sugars in raw and 2nd carbonatation juice; elution was carried out with 4:1 acetone:water containing 0.4 mg/ml piperazine, and a LDC refractometric detector was used. The results obtained showed that diffusion in an electric field did not appreciably increase the reducing sugars content in the juice, which was therefore of higher purity than that obtained by conventional diffusion and needed no purification. However, such a process would consume too much energy, and it would be more desirable to use the technique only in the head section; although the results were not as good as with the method applied for the entire diffuser, they were still better than those given by conventional diffusion, and the very low reducing sugars content would allow a decrease in the amount of lime used in traditional carbonatation.

Reducing the sugar content in molasses – a reserve for raising the performance level

A. L. Shoikhet, L. I. Chernyavskaya and A. P. Pustokhod. *Sakhar. Prom.*, 1987, (1), 28 - 29 *(Russian)*.

Information is provided on the varieties of beet supplied to Babino-Tomakhovskii sugar factory and of the average composition of samples in 1984 and 1985, showing excessive amounts of ash and other non-sugars and low natural alkalinity. As a consequence of the low beet quality (blamed partly on incorrect fertilization) and of under-utilization of low-grade crystallizers (with too short a cooling period and hence an above-optimum final temperature) the molasses sugar contents were excessive. Advice is given on ways of remedying the situation.

Operation of a A1-PDS-20 diffuser with cossettes prescalding

I. A. Oleinik *et al. Sakhar. Prom.*, 1987, (1), 30 - 32 *(Russian)*.

Trials are reported on the use of evaporator bleed to prescald cossettes to 68°C by a fully automatic system, as a result of which pulp losses were reduced and raw juice purity raised while the K, Na and Ca contents in the juice were lower than without prescalding.

Evaluation of the level of process automation in raw juice purification

A. P. Ladanyuk and A. A. Sushchenko. *Sakhar. Prom.*, 1987, (1), 32 - 34 *(Russian)*.

Statistical evaluation of the level of automation in a typical Soviet carbonatation system showed that it falls far short of requirements. While the situation could be remedied by increasing the number of automatically measurable parameters and by improving and extending the control methods, the use of computers is considered vital for a satisfactorily high level of automation.

A combined automatic level control system in the prescalder of a tower diffuser

A. F. Makhlai. *Sakhar. Prom.*, 1987, (1), 34 - 35 *(Russian)*.

A system is described for automatic control of the juice and cossettes levels in a prescalder which is combined with automatic maintenance of the balance between juice entering the tower diffuser from the prescalder and juice being withdrawn.

Modification of a rotary pulp trap for raw juice

A. G. Babak. *Sakhar. Prom.*, 1987, (1), 35 - 36 *(Russian).*

The poor performance of rotary screens used to remove fine pulp particles from raw juice led to modifications of the system whereby mixing of untreated with treated juice was prevented. The alterations are described.

Modification of Be1KZ voluted burners

Yu. A. Cherednichenko, V. F. Naumenko, V. Kh. Liberman, A. A. Poloiko and N. P. Cherpak. *Sakhar. Prom.*, 1987, (1), 36 - 37 *(Russian).*

A modified gas/oil burner with voluted blade-type swirler is described and the performance of a sugar factory boiler before and after fitting of the new design discussed.

Experiment on sugar beet storage using urea-formaldehyde foamed plastic covers

A. I. Solov'ev, A. I. Pivovarov, V. A. Knyazev and A. Kh. Starushenko. *Sakhar. Prom.*, 1987, (1), 40 - 41 *(Russian).*

Trials were conducted at a number of factories on storage of beets for up to 120 days (after spraying with milk-of-lime) in piles covered with foamed plastic which protected the beets against frost and was as effective as but much cheaper than rush mats.

Device for removal of heavy impurities from a beet sample

V. N. Mardal', A. I. Levitskaya, A. Ya. Zagorul'ko and A. L. Shoikhet. *Sakhar. Prom.*, 1987, (1), 41 - 43 *(Russian).*

Heavy mechanical impurities such as stones and metal are removed from beet samples by a system incorporating a vertical tank with conical bottom. Dilute filter-cake in the tank is maintained within a given density range by adjustment of water feed, and an electrically driven vertical bladed disc on one side and towards the bottom of the tank prevents settlement of the cake; the impurities fall to the bottom while the beets, being lighter than the cake, float on the surface. After 15 - 20 seconds a pivoted horizontal screen, of virtually the same cross-section dimensions as the tank and located at the side away from the feed port, swings slowly up through the tank, scooping up beets; cake drains off through the screen which retraces its path once it has reached a given angle and deposited the beets at a point from which they are carried by conveyor and rinsed with water. When the conical section of the tank is full, the impurities drop through a gated opening onto a vibratory screen which separates the cake for recirculation to the top feed tank while the impurities are discharged via a chute. The system has worked entirely satisfactorily, increasing the accuracy of dirt tare determination and reducing the costs of manual labour.

A new twin-trough progressive defecation vessel with reverse liming

F. Kozaczka and S. Wacnik. *Gaz. Cukr.*, 1986, **94**, 97 - 99 *(Polish).*

A prelimer comprising twin troughs side-by-side with juice flow along one and back through the other is based on the Brieghel-Müller prelimer with U-shaped troughs divided into compartments and shafts running the length of the troughs with stirrer paddles attached to them. The new design is lighter in weight than previous Polish models, the main shaft bearings are more durable and operation and maintenance are easier.

Possibilities of saving heat by modifying the carbonatation process

W. Lekawski and K. Urbaniec. *Gaz. Cukr.*, 1986, **94**, 99 - 101 *(Polish).*

Heat is lost during carbonatation as a result of mass and heat exchange between the juice and CO_2; the temperature of the gas rises almost to that of the juice, evaporation of which causes a greater amount of saturated steam to mix with the gas. By preheating and wetting the CO_2 and conducting carbonatation under pressure, the heat losses can be considerably reduced, e.g. by two-thirds (equivalent to 1.6 - 2.6% steam on beet) when 1st carbonatation is carried out at 0.13 MPa and 78°C. Condensate from the juice heater at approx. 75°C may be used to pretreat the CO_2 used in 1st carbonatation, and evaporator condensate at 95 - 100°C to treat the gas used in 2nd carbonatation. Even further savings are possible if boiler flue gas containing 12 - 14% CO_2 is used instead of CO_2 from the lime kiln.

A low-grade pan stirrer

V. Bulena. *Gaz. Cukr.*, 1986, **94**, 102 - 104 *(Polish).*

See *I.S.J.*, 1986, **88**, 115A.

A belt trash separator

J. Simek. *Listy Cukr.*, 1986, **102**, (12), 1 p. *(Czech).*

An inclined endless rubber belt carrying rows of rakes is installed over a section of beet flume of reduced dimensions relative to the rest of the flume; the rakes pivot freely from the belt so as to assume a virtually horizontal position as they pass at 0.10 - 0.15 m/sec through the upper level of the water in the flume and hang loosely on the downward run from the upper pulley. The light impurities caught by the rakes fall into a chute from the belt as it passes over the upper pulley. The unit is available in two sizes corresponding to daily slices of 2000 and 3500 tonnes of beet.

Anaerobic treatment of beet factory waste waters

J. P. Lescure, P. Bourlet, D. Verrier and G. Albagnac. *Sugar Tech. Rev.*, 1986, **13**, 179 - 231.

A survey with 119 references is presented of the literature on anaerobic treatment of beet sugar factory effluent with generation of methane. The work covers the possibilities and limitations of anaerobic treatment, biological and kinetic aspects of the fermentation process, the composition of sugar factory waste waters and the role of a methanizer in effluent circuits, anaerobic processes, digester operation, and the energy and economics involved.

Experiments on the use of enzymes as (pulp) pressing aids

K. Buchholz and K. Matalla. *Zuckerind.*, 1987, **112**, 26 - 29 *(German).*

Studies on enzymatic hydrolysis of pressed pulp were followed by experiments in which fresh pulp was incubated with enzyme solutions under controlled conditions and then pressed. Comparison with untreated controls showed a sharp rise in the dry solids content but with passage of a considerable portion of the dry solids into solution with prolonged treatment. Moreover, the initial positive effect was achieved only with uneconomically high enzyme concentrations and/or prolonged reaction times. The results were similar for the various mixtures of pectinases, pectin esterases, hemicellulases and cellulases. The effects of temperature and pH were also investigated.

The path to continuous crystal footing production

K. E. Austmeyer and T. Frankenfeld. *Zuckerind.*, 1987, **112**, 36 - 45 *(German).*

The possibility of raising product quality and reducing energy consumption by continuous boiling on a continuously produced crystal footing is discussed. Past efforts to produce sugar of suitable crystal size distribution in continuous pans such as the MSMPR (Mixed Suspension Mixed Product Removal) type widely used in other industries have proved unsuccessful. It is thought that the MSCPR (Mixed Suspension Classified Product Removal) system of Szarejko[1], in which a solid conical bowl centrifuge is installed in the vapour space for classification of the crystals, would not work successfully (although no tests appear to have been conducted on the system) because of incrustation in the centrifuge. The concept of crystal footing production and the requirements of a low crystal growth rate and a high shear gradient are discussed; reference is made to batch crystal footing production[2] and to continuous production using crystallization by cooling in a tower system comprising stirred compartments one above the other. Tests on the use of a multi-tray settler provided with rakes and of a hydrocyclone for crystal classification demonstrated the unsuitability of both systems, whereas a solid conical bowl centrifuge for wet screening used in conjunction with the above-mentioned tower crystallizer has proved satisfactory by separating the fine from the coarse fraction, increasing the crystal content of the coarse fraction by 50% and allowing the fine crystals to be dissolved in the preheated cooling water and re-utilized as feed.

Preliminary information on operation of a sectional tubular (heat) exchanger for condensate-raw juice

P. Hoffman. *Listy Cukr.*, 1987, **103**, 9 - 12 *(Czech).*

Results are given of trials with two sections of a horizontal tubular heat exchanger for raw juice using condensate as heating medium. At a juice flow rate of 2.0 - 4.5 m/sec corresponding to an hourly throughput of 33 - 73 tonnes, the temperature was raised by 8 - 12°C from 46 - 50°C; the heat transfer coefficient ranged from 2000 to 3800 W/m^2/°C and pressure loss on the juice side was 28 - 62 kPa and on the condensate side 20 -26 kPa. The unit was less costly but of higher productivity than a plate heat exchanger that underwent parallel tests.

The monitoring and design of juice heaters

P. Hoffman. *Listy Cukr.*, 1987, **103**, 13 - 16 *(Czech).*

So as to monitor the operation of an evaporator or modify an evaporator or heating scheme it is necessary to know the amount of vapour that is needed for juice heating under given conditions or the maximum heat rise obtainable with a given quantity of vapour; in Czechoslovakia, 0.2% steam on beet per °C temperature rise is normally assumed where the juice volume is 110% on beet, but a precise calculation must be made where a different volume of juice is involved. Equations are presented for calculation of the parameters required for monitoring of an existing juice heating system; for the heat transfer coefficient, an equation of Sazavsky is found to be better than that of Baloh or Speyer[3]. A similar calculation procedure is also demonstrated for new heaters and for calculation of pressure losses, and a worked example is given concerning thin juice heating.

The effect of the surface properties of muds on non-sugars adsorption in 2nd carbonatation and sulphitation

L. D. Bobrovnik, V. Z. Semenenko and N. S. Fedorova. *Sakhar. Prom.*, 1987, (2), 18 - 20 *(Russian).*

In a study of the physico-chemical properties of calcium carbonate and calcium sulphite mud formed during 2nd carbonatation and sulphitation it was found that PO_4^{---} in added trisodium phosphate enhanced the adsorptive capacity of the mud by raising the value of the optimum alkalinity at which the zeta-potential and calcium salts removal were maximum. This finding confirmed the validity of an earlier recommendation to use phosphate instead of sodium carbonate.

Functioning of an automatic process control scheme at Il'ich sugar factory, Yagotin

K. F. Gerbut, B. A. Eremenko, V. D. Novoseletskii and V. I. Radalovskii. *Sakhar. Prom.*, 1987, (3), 27 - 29 *(Russian).*

Operation of a computerized automatic process control system is discussed and details are given of specific process controls and the benefits obtained.

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[1] Polish Patent 72776.

[2] Austmeyer: *I.S.J.*, 1986, **88**, 3 - 7.

[3] "Wärmeatlas für die Zuckerindustrie" (Verlag M. & H. Schaper, Hannover), 1975.

Sugar refining

Anti-explosion dust separators

L. Bouilliez. *Sucr. Franç.*, 1987, **111**, 37 - 41 *(French).*

Illustrated descriptions are given of NEU dust separation equipment which is designed and constructed so as to minimize the risk of an explosion or limit the effects if an explosion does occur.

Protection of silo galleries against the effects of a sugar dust explosion

J. Winter and J. P. Pineau. *Sucr. Franç.*, 1987, **111**, 42 - 46 *(French).*

The spread of fire and of shock waves from a sugar dust explosion along conveyor galleries and up an elevator shaft in a bulk silo can be prevented by a barrier of inert dust or water that is automatically ejected by the action of a detector. How the system works and its effectiveness are discussed.

Monitoring installations. Measurement of dust in the air and on the ground

J. P. Ducatillon. *Sucr. Franç.*, 1987, **111**, 47 - 49 *(French).*

Methods and equipment used to monitor the quantity of dust in the air and on the ground in the vicinity of equipment handling sugar (including icing sugar) are described and some results obtained are discussed.

Study of the influence of temperature during the process of affining raw sugar using industrial syrup

J. Jover P., I. Machado L., X. Vila M. and M. Pérez P. *Centro Azúcar*, 1984, **11**, (2), 123 - 132 *(Spanish).*

In a series of experiments, affination was carried out using syrup at 32, 40, 50 and 60°C and the pol, reducing sugar content, moisture, grain size and colour index of the affined sugar were measured. Colour removal increased from 57.06 to 69.57% over the range of temperatures, approximately 4% per 10°C, while reducing sugars elimination increased by about 0.1% per 10°C. Removal of non-sugars rose by some 0.15% per 10°C, but sucrose dissolved rose by 0.73% per 10°C. It was not possible to determine the optimum temperature for affination by the Fibonacci method because none of the curves of variation of the parameters measured showed maxima or minima.

Determination of the consumption of powdered activated carbon in a continuous system of decolorization

A. Curbelo S. *Centro Azúcar*, 1984, **11**, (3), 69 - 76 *(Spanish).*

A system is described for automatic control of the feed of active carbon slurry to refinery liquor, based on measurement of liquor flow rate and colour, whereby the ratio of slurry to liquor flow is adjusted to a particular value which depends on the colour of the treated liquor.

Economic comparison of stage systems in the decolorization of refinery liquors by powdered activated carbon

A. Curbelo S. *Centro Azúcar*, 1984, **11**, (3), 77 - 82 *(Spanish).*

Calculations have been made whereby it is shown that use of a two-stage countercurrent system of carbon treatment gives a 40 - 45% saving in carbon consumption, compared with a single-stage batch treatment, whereas a 2-stage co-current system gives a 30 - 35% saving.

Study of the method of seeding in the production of refined sugar

O. Tintorer D., J. F. Mazorra A. and R. Mut B. *Centro Azúcar*, 1984, **11**, (3), 105 - 112 *(Spanish).*

A study has been made of the crystallization process in the refinery and it is concluded that improvements are required on the current practice of "shock" seeding. The presence of a high level of reducing sugars in the syrup hinders crystal growth in the pan, and the importance of the pressure in the pan after seeding is emphasized in respect of establishing the grain and reducing energy consumption. Studies should continue on determining the numbers of crystals formed on shock seedling so as to be able to effect control, and it is noted that syrup feed in the later stage of seeding should be strictly controlled to match the evaporation level.

Design and operation of the Fives-Cail Babcock continuous vacuum pans at Elsdorf

H. Herold. *Zuckerind.*, 1987, **112**, 118 - 122 *(German).*

An illustrated description is given of the FCB continuous pan and its operation, and details are given of the two pans used for *A*-raw sugar and low-grade massecuite at Elsdorf, with information on automatic control and on pan cleaning by boiling thin juice at low vacuum for about 60 min.

Recycling of non-sugars in sugar refineries

A. M. James, M. A. Clarke and R. S. Blanco. *Sugar J.*, 1987, **49**, (8), 12 - 15.

See *I.S.J.*, 1987, **89**, 54 - 59.

Assessment of the technological properties of AV-17-2P porous decolorizing anion exchange resin

G. A. Chikin *et al. Teoriya i Prakt. Sorbts. Protsessov*, 1986, (18), 84 - 89; through *Ref. Zhurn. AN SSSR (Khim.)*, 1987, (8), Abs. 8 R483.

A refinery syrup decolorizing technique using AV-17-2P polymer anion exchange resin was investigated and the dependence of the resin performance on its original parameters (by comparison with standard parameters) indicated. Reasons are given for changes in the basic physico-chemical properties of the resin with length of service and methods of retarding the "ageing" process are suggested which would increase decolorizing efficiency.

Starch based sweeteners

Continuous isomerization of glucose in starch hydrolysates with immobilized glucose isomerase

M. Boruch and E. Nebesny. *Acta Aliment. Pol.*, 1985, **11**, (4), 413 -425; through *Ref. Zhurn. AN SSSR (Khim.)*, 1986, (23), Abs. 23 R612.

Continuous glucose isomerization was studied using Sweetzyme-S and Sweetzyme-Q immobilized isomerases of 213 and 215 units/g activity, respectively. The following were used as substrates: a glucose solution of 40% concentration and DE 99.9, a solution containing 76% glucose and of DE 84 obtained from *B*-crystallization, hydrolysate of 53% glucose content and DE 68 and a corn syrup of 21% glucose content and DE 40. Before isomerization, the solutions were treated by ion exchange and $MgSO_4$ added as activator. The degree of isomerization in the first few days of column operation was the same regardless of substrate and DE. Oligosaccharides present in the substrate had a negative effect on the action of the isomerase by reducing its stability and the fructose yield; their effect was greater at lower DE. The colour of the substrates increased as the isomerization progressed, the increase being greater with more colouring matter in the original material. The colour of the solutions was lower where the isomerase retained its activity over a long period.

High fructose corn syrup

S. J. Jadhav, R. B. Natu and D. B. Ghule. *Bharatiya Sugar*, 1986, **11**, (11), 23, 25 - 26.

The manufacture, composition, properties, sweetness, handling, storage and food application of HFS are described.

Continuous granulated glucose production by crystallization in a fluidized bed

A. Sroczynski, M. Boruch, J. Skalski and E. Nebesny. *Biotechnol. Chem. Zywn., Ses. Nauk.*, 1985, 154 - 156; through *Ref. Zhurn. AN SSSR (Khim.)*, 1986, (24), Abs. 24 R616.

A technique is proposed for production of granulated glucose using crystallization in a fluidized bed. A glucose syrup of 80% dry solids and a DE of 96 - 97 is sprayed at 80°C onto a bed of glucose in a fludized state brought about by the use of air at 65 - 75°C. The end-product (glucose monohydrate) contains 9% water.

Pinch technology applications in corn processing

J. V. Shah and H. D. Spriggs. *Sugar y Azúcar*, 1986, **81**, (12), 26 - 27, 30 -31, 33.

Pinch technology is the term used for the series of principles and design rules developed around the concept of a process pinch (the most constrained part of the process) within the general framework of process integration. By means of composite heating and cooling curves it is possible to determine the minimum energy requirements. The fundamentals of the technique are described and its use demonstrated in the case of two studies relating to corn wet milling for the production of starch and HFS; it was found that the energy consumption could be reduced by 20%. In one case, although use of mechanical vapour recompression in conjunction with syrup evaporation would have saved energy, the fact that it would also have prevented use of bleed vapour for process heating purposes elsewhere in the factory reduced its benefits.

Saccharification of starch in an immobilized glucoamylase monolithic reactor

F. Shiraishi, K. Kawakami and K. Kusunoki. *Kagaku Kogaku Ronbunshu*, 1986, **12**, (4), 492 - 495; through *Ref. Zhurn. AN SSSR (Khim.)*, 1987, (1), Abs. 1 R469.

Tests were conducted on continuous starch saccharification using immobilized glucoamylase fixed to a support in a monolithic reaction vessel. Cylindrical sections of the reactor were filled with the supported enzyme in a honeycomb form but with circulation channels. Each of the 10 sections placed one above the other was at an angle of $\pi/4$ radians relative to its neighbour. Laminar flow was maintained in the reactor. It was found that in this type of vessel the saccharification rate was more than double at a starch concentration of 460 g/litre; 47% hydrolysis was achieved within 12 hr. The active half-life of the enzyme was 79 days.

Maltulose formation during saccharification of starch

F. F. Dias and D. C. Panchal. *Starch/Stärke*, 1987, **39**, 64 - 66.

Maltulose precursors are formed as a result of chemical isomerization of reducing end units of glucose during enzymatic liquefaction and autoclaving of starch in the enzyme-enzyme process of glucose manufacture. The precursors are hydrolysed during subsequent saccharification with amyloglucosidase to form maltulose as one of the products. Temperature, pH and DE of the liquefied starch during autoclaving and α-amylase treatment are important parameters in control of precursor formation – the lower the values of the first three the smaller is the amount of maltulose formed. No maltulose is formed if acid liquefaction precedes saccharification, while maltitol is produced instead of maltulose when the reducing end unit is blocked by hydrogenation to sorbitol.

Glucose crystallization with preliminary preparation of the initial massecuite

M. S. Zhigalov, V. S. Shterman, A. R. Sapronov, K. Liding and I. R. Khanukhov. *Sakhar. Prom.*, 1987, (4), 41 - 43 *(Russian).*

Retention of 30% of the massecuite in the crystallizers for use as seed footing in the production of crystalline glucose hydrate has a number of disadvantages, which can be eliminated by preliminary preparation of massecuite based on a syrup of lower Brix than normally. Experimental results have confirmed the benefits of the new system, including provision of a consumer product 10 days earlier than with conventional processing.

Laboratory studies

An analytical approach for the determination of colour and other impurities in cane

G. R. E. Lionnet. *Proc. 60th Ann. Congr. S. African Sugar Tech Assoc.*, 1986, 62 - 65.

An equilibrium method for the determination of a number of constituents in cane involves immersion of a sample of cane pulp in water at constant temperature, whereby the cane absorbs the water and various soluble constituents are extracted. Experiments at pH 6.2 - 6.4 and 80°C with five samples of shredded cane (obtained using a Jeffco cutter-grinder) of varying weights immersed in 2 litres of water were used to determine pol and Brix % cane, total phenols by the Folin-Ciocalteu method and amino-nitrogen by the ninhydrin method and to measure absorbance at 280 nm (pH 4) and 420 nm (pH 4 and 9). Equilibrium was reached after about 40 min. The linearity of a plot of $1/C_\infty$ *vs.* $1/W$, where C_∞ = concentration (g/cm^3 at infinite time) and W = weight of cane (g), was excellent in all cases, with correlation coefficients ranging from 0.942 to 0.999. The method was applied to investigation of changes in impurity content caused by the presence of tops and trash, by burning as against trashing, by moisture stress and in rind, nodes and internodes of a clean cane stalk. Results showed that tops introduce greater concentrations of impurities than trash, rind contributes significant quantities of colour impurities, burnt cane contains higher levels of the selected impurities and gives higher absorbances, and that drought causes considerable increase in the concentration of colour impurities and precursors, particularly amino-N. Average values were found for a partition coefficient K (concentration of constituent in extract/concentration of constituent in pulp); for pol and Brix it was 1, indicating the validity of direct cane analysis (DAC) for these two parameters but not for the others. While the equilibrium method requires more equipment than conventional cane analysis methods and is tedious, it has a number of advantages; however, it is not seen as a replacement for DAC or techniques involving pressing but rather as complementary to them.

Application of multi-dimensional data analysis to raw sugar quality evaluation

J. A. Farinas. *Cukoripar*, 1986, **39**, 115 - 118, 149 - 153 *(Hungarian).*

The fundamentals of multi-dimensional data analysis (MDDA) and of principal component analysis (PCA) are explained and their application in computerized evaluation of the quality of raw sugar from 13 Cuban sugar factories demonstrated using sub-routines of cluster and discriminant analysis developed at the Computer Centre of the Hungarian Institute of Planning.

Polyphenol contents of plantation white sugar

D. B. Ghule, R. B. Natu and S. J. Jadhav. *Bharatiya Sugar*, 1986, **11**, (8), 57 - 61.

The total polyphenol (TPP) contents in M-30 and S-30 plantation white sugars produced by cooperative sugar factories in Maharashtra in 1984/85 were found to be in the range 59 - 155 (average 101) and 57 - 119 (average 88) ppm, respectively. A modification of the Swain & Hills method[1] was used for the determination.

Comparative evaluation of the complexometric and oxalate methods of determining lime salts in products of sugar manufacture

V. A. Loseva, I. S. Naumchenko and V. M. Perelygin. *Rpt. Voronezh Tech. Inst.*, 1986, 9 pp; through *Ref. Zhurn. AN SSSR (Khim.)*, 1986, (23), Abs. 23 R600.

Comparative evaluation of the oxalate and complexometric methods was conducted on model $CaCl_2$ solutions, after which products of beet sugar manufacture were analysed. It was found that the complexometric method gave a lower Ca^{++} ion content as a result of transfer to $CaOH^+$ ions, and that the oxalate method was more accurate. The difference between the results of the two methods was 12 - 29%. The oxalate method is recommended for analysis of sugar manufacture products and particularly for determination of $Ca(OH)_2$ solubility.

Thermophysical properties of frozen pure sugar solutions

M. A. Gromov. *Sakhar. Prom.*, 1986, (11), 17 - 20 *(Russian).*

Tabulated data are given for density, specific heat, thermal conductivity and the coefficient of thermal diffusivity of pure sugar solutions of up to 35% concentration for the first two parameters and up to 30% for the last two, at temperatures in the range between –4°C and –40°C. The content of supercooled water was also determined for up to 50% sugar concentration at given temperatures between –34°C and –7°C. The results of the various measurements are discussed.

Approaches to the validation of derivatized multi-component analysis by GLC

M. Tarka. *Anal. Lett.*, 1986, **19**, (5/6), 537 - 541; through *Anal. Abs.*, 1986, **48**, Abs. 12A24.

A technique is presented that should aid in validating an analytical method. The GC separation of peracetylated nitriles of aldoses was examined as example, with rhamnose as internal standard (IS), the detector response being measured as the relative response factor (RRF) for each of the four components (glucose, sorbitol, fructose and sucrose) in amounts of 1 mg. The RRF is calculated as [mass (i)/mass (IS)] × [area (IS)/area (i)], where i is any sample component other than IS. Analytes were examined as individual derivatives mixed with the IS, and as groups of derivatives (prepared by using hydroxylammonium chloride and acetic anhydride). The RRF variation that occurs when the amount of one component or the reagent is varied is also examined.

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[1] *J. Sci. Food Agric.*, 1959, **10**, 63 - 68.

Determination of sucrose in the presence of glucose in a flow-injection system with immobilized multi-enzyme reactors

B. Olsson, B. Stalbom and G. Johansson. *Anal. Chim. Acta*, 1986, **179,** 203 - 208; through *Anal. Abs.*, 1986, **48,** Abs. 12D93.

Sucrose is determined in a packed-bed reactor by its reactions with co-immobilized β-fructofuranosidase, aldose-1-epimerase (I) and glucose oxidase (II). Glucose in the sample is decomposed in a pre-reactor containing co-immobilized I, II and catalase. The H_2O_2 produced is measured at 514 nm after reaction with a reagent comprising 20 mM 3,5-dichloro-2-hydroxybenzenesulphonate, 1mM 4-aminoantipyrine and 1 mM EDTA in 0.1M citrate buffer at pH 6. The response is rectilinear from the detection limit, 0.1 μM, up to 0.5 mM. The coefficient of variation at the 0.2 mM level is 0.3% (n = 20).

Automatic refractometers

Anon. *Ann. Rpt. Mauritius Sugar Ind. Research Inst.*, 1985, 49 - 50.

Although Bausch & Lomb refractometers had been used in all 19 Mauritius sugar factory laboratories for 25 years and had proved to be sturdy and reliable, age and unavailability of spares led to their replacement by Schmidt & Haensch Refractomat-L instruments. However, a number of problems arose, including the adverse effect of suspended solids on Brix readings. Filtration of samples before Brix measurement proved effective as a means of eliminating suspended solids, and the readings given by the two types of refractometer were then very close, any difference having negligible effect on cane payment calculations.

The chemical composition and properties of colouring matter in cane raw sugar and in products from its processing

L. D. Bobrovnik, S. I. Shul'ga, G. I. Sheiko and N. V. Dubinina. *Sakhar. Prom.*, 1986, (12), 11 - 14 *(Russian).*

Colouring matter was separated from remelt liquor and *A*-raw syrup and fractionated on DEAE-cellulose; elution was carried out, successively, with water and solutions of Na_3PO_4, Na_2HPO_4, NH_4Cl and NaOH. The composition of each fraction was established and an empirical formula allotted; infra-red absorption spectra were also obtained and are described in some detail. Differences were noted between the remelt and raw syrup fractions in terms of their spectra and band characterisitics, but nitrogen was found in all cases. The studies confirmed that colouring matter containing carboxyl groups is adsorbed to a greater extent by active carbon than substances not containing carboxyl groups but having imino groups which were removed from the column with Al_2O_3, a polar adsorbent. During refining there is marked amino-sugar condensation plus complexing, particularly esterification, of colorants. The need for polar adsorbents to remove some of the colouring matter confirms the suitability of remelt treatment with a combination of active carbon and $CaCO_3$.

Determination of the juice coefficient of beet

A. K. Buryma. *Sakhar. Prom.*, 1986, (12), 14 - 18 *(Russian).*

An empirical formula suggested by Zagorul'ko and incorporated in official Soviet methods of chemical control and loss calculations bases the juice coefficient β_0 on the volume of juice in a 26-g brei sample and the juice density. However, calculations and statistical analysis show that the values of β_0 obtained differ sufficiently from values calculated by other equations as to make the formula invalid. In its place is proposed the ratio between the sugar content in beet cossettes as determined by aqueous digestion and the sucrose content in the beet juice.

The occurrence of model colouring matter in molasses from sugar manufacture

N. G. Gulyuk. *Sakhar. Prom.*, 1986, (12), 18 - 21 *(Russian).*

Examination of the absorption spectra of model colorant fractions obtained by alkaline degradation of sugar and by caramelization, and chromatographic analysis of melanoidins showed that factory molasses contained no colouring matter analogous to the model fractions. It is therefore recommended to use instead colorant fractions that have been obtained directly from beet sugar products by liquid chromatography, taking care to ensure that no changes occur in their structure during the operation.

Determination of bisulphite compounds of fructose and glucose in products of sugar manufacture

I. P. Chepurnoi, A. M. Belikova and V. M. Volodina. *Sakhar. Prom.*, 1986, (12), 25 - 27 *(Russian).*

Bisulphite derivatives of fructose and glucose resulting from sulphitation were determined by feeding SO_2 from a Kipp's apparatus at 2 - 3 ml/min into 6 -7% aqueous solutions of the sugars separately and together during heating to 90 - 95°C; 1 ml samples of the solutions were taken before and after the gas injection and concentrated in a rotary evaporator at 60°C, after which the sugars were converted to their trimethylsilyl derivatives by the Brobst & Lott method[1]. Chromatography was conducted in a 50 cm × 0.5 mm column at a programmed temperature rise of 4°C/min from 125° to 280°C and then at constant temperature. The areas of the peaks were read by digital integrator. Sulphitation without heating over a period of days resulted in accumulation of only 2% bisulphite derivatives, whereas sulphitation with heating for 1 hour gave more than 6%. There was only one peak representing a fructose bisulphite, while six bisulphite derivatives of glucose occurred; this was attributed to anomerization of the glucose derivatives at high temperature and in an acid medium. Molasses contained 0.2 - 0.4% glucose bisulphite derivatives; the fructose derivatives were

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[1] *Cereal Chem.*, 1966, **43,** (1), 35 - 43.

masked by the sucrose peak. For determination of the derivatives in white sugar, in which their content would be far lower, pre-concentration is needed as well as removal of the masking effect of sucrose. A procedure is described.

Experience with new analytical methods for the sugar industry

G. Pollach, W. Hein, G. Rösner and H. Berninger. *Zuckerind.*, 1986, **111,** 1101 - 1107 *(German).*

New analytical approaches are described that have been developed at the Sugar Research Institute at Fuchsenbigl in Austria. Details are given of the equipment and procedures used in drying mashed beet cossettes, pressed pulp and carbonatation mud and in their analysis to obtain a beet end balance, including losses. Pre-drying allows samples to be easily transported and enables homogeneous samples to be obtained by crushing as well as repeat analyses. Requisite formulae are given for calculation of the various parameters. Details are given of an automatic accelerated technique for sugar determination in beet brei based on the use of a polarimeter, electronic weigher and automatic refractometer but without the time-consuming filling of a measuring flask to the mark. Density of the sample is calculated from the RDS and the volume calculated from the total weight and density; the sugar content is then calculated from these and the pol reading. For determination of bacterial infection in beet diffusion with which L-lactic acid formation is associated, the total lactic acid in molasses is analysed enzymatically and the value for D-lactic acid subtracted from the total.

Determination of sugars by thermochemical separation during reactions using immobilized enzymes

R. K. Owusu and A. Finch. *J. Wuhan Univ., Natur. Sci. Ed.*, 1986, (1), 57 -64 through *Ref. Zhurn. AN SSSR (Khim.)*, 1986, (24), Abs. 24 R621.

A method is proposed for determination of glucose, fructose, mannose and sucrose by thermochemical monitoring in fermentation reactions. Glucose oxidase, catalase or hexokinase immobilized on porous glass is used as catalyst. The limit of detection is 7 – 25 × 10^{-3} moles, and the error is ±4%.

The influence of products of aluminium sulphate hydrolysis on the removal of raw juice non-sugar

V. N. Shalatanov and A. A. Lipets. *Sakhar. Prom.*, 1987, (1), 20 - 22 *(Russian).*

In investigations of the behaviour of SO_4^{--} anions in aluminium sulphate used for juice purification, hydrolysis by NaOH was followed by estimation of the sulphate anion content in the filtered, washed precipitate obtained; with increase in pH there was an irreversible reduction in the content and the anions became weak ligands, while the strength of the OH^- ions overtook that of the Al^{+++} ions. Since the coordination number of Al in the experiments was 6, reduction in the content of one ligand in the internal coordination sphere of the complex increased the content of the other ligand; this change affected the intermolecular forces acting on the surface of the Al hydroxide and hence the degree of hydration. Aluminium gels precipitated in a weak alkaline medium had the highest degree of hydration, and subsequent tests showed that the maximum juice purification efficiency was obtained at pH 8.5 - 9.0, i.e. at which Al hydroxide precipitation was maximum.

The effect of aluminium sulphate on the electrokinetic potential of calcium carbonate mud

V. A. Tsekhmistrenko, S. P. Olyanskaya, L. M. Khomichak, O. P. Tkachenko and N. A. Arkhipovich. *Sakhar. Prom.*, 1987, (1), 24 - 25 *(Russian).*

Increase in the quantity of aluminium sulphate added to wet $CaCO_3$ mud was found to reduce the zeta-potential of the carbonate by 11.7 - 19.5 mV in the concentration range up to 0.1% (up to 0.02 ppm $CaCO_3$); the reduction was particularly marked with a rise in sulphate concentration from 0 to 0.04 - 0.06% (to 0.008 - 0.012 ppm $CaCO_3$), after which it was negligible with further increase in the concentration of the reagent. In the case of 1st carbonatation mud, there was no change in the nature of the charge as a result of aluminium sulphate addition and the greatest reduction in the negative charge occurred with an increase in the sulphate concentration from 0 to 0.08% (0 - 0.016 ppm mud).

Alkaline degradation of monosaccharides. VII. A mechanistic picture

J. M. de Bruijn, A. P. G. Kieboom and H. van Bekkum. *Starch/Stärke*, 1987, **39,** (1), 23 - 28.

While it has been known for a long time that monosaccharides are unstable in alkaline medium and undergo, besides isomerization, degradation reactions that irreversibly lead to carboxylic acid products, the mechanism of these reactions has been only partly explained for lack of a suitable method of quantitative analysis of the reaction mixture. However, a HPLC method has been developed[1] which has proved to be a convenient and reproducible means of analysing carboxylic acids. From data on alkaline degradation of monosaccharides such as D-fructose and D-glucose a mechanistic picture has been developed of the reaction in which the extent of degradation, particularly of ketoses, is governed by the reaction conditions; hence, the extent of retro-aldolization of ketoses largely determines the amount of lactic acid formed, while the ratio between benzilic acid formation, alpha-dicarbonyl cleavage and aldolization of alpha-dicarbonyls as key intermediates in the reaction further affects the composition of the C_1 - C_6 acid component of the product as well as the total amount of oligomers.

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[1] de Bruijn *et al.*: *I.S.J.*, 1984, **86**, 195 - 199.

By-products

Glycerol production by fermentation. A review

–. Vijaiki, P. Shore and N. G. Karanth. *Process Biochem.*, 1986, **21**, (2), 54 - 57; through *Ref. Zhurn. AN SSSR (Khim.)*, 1986, (24), Abs. 24 R511.

Biochemical synthesis of glycerol is economically viable in those countries having adequate supplies of molasses. Other forms of raw material such as potato, grain, straw, bran and wood require chemical or fermentative prehydrolysis, which considerably complicates the technology. Moreover, molasses contains all the mineral salts needed for normal yeast growth. Among microorganisms that produce glycerol, sulphite and osmostable forms of yeasts are of industrial value. The features of batch and continuous glycerol fermentation systems are outlined, as well as the use of immobilized yeasts and membrane technology.

Process for obtaining chemical pulp from bagasse by prehydrolysis with a mixture of acids

R. Sotolongo, R. López, A. Ruiz, J. Cantillo and G. Iglesias. *Tecnol. Quim.*, 1986, **7**, (1), 55 - 58, 86; through *Ref. Zhurn. AN SSSR (Khim.)*, 1986, (24), Abs. 24 R613.

Results are reported of studies on bagasse pretreatment with a 2:1 H_2SO_4:HNO_3 mixture. An experiment design method was used in which the prehydrolysis time (2 - 4 hours) and concentration of acid mixture (0.1 - 0.3N) were chosen as independent variables; optimum values were 2 - 3 hr and 0.2 - 0.3N, respectively. Prehydrolysis was conducted in glass vessels at 100°C. Cellulose yield was approx. 37%. The properties of the cellulose obtained were similar to those of cellulose produced by other known methods. The α-cellulose content was ≥90% and the degree of polymerization ≥900, i.e. above the lower recommended limit; the pentosan content was below 5%.

By-products of the sugar cane industry — a synopsis

Anon. *Bharatiya Sugar*, 1986, **11**, (13), 7 - 11.

A survey is presented of cane by-products utilization.

Future of molasses-based alcohol distillation industries in developing countries

B. B. Paul. *Bharatiya Sugar*, 1986, **11**, (13), 13 - 16.

Aspects of ethanol production from cane molasses are discussed, including the equipment and technology used.

Production of ethanol by direct fermentation of sugar cane juice

R. B. Natus, S. J. Jadhav and N. A. Ramaiah. *Bharatiya Sugar*, 1986, **11**, (13), 19 - 20.

A laboratory batch fermentation experiment is reported in which 5 litres of 15 - 18°Bx cane juice were inoculated with *Saccharomyces cerevisiae* to yield 8.11 - 8.42% alcohol within 20 - 24 hr at 100% efficiency.

Sucrose esters: production and applications

T. G. Kolekar, S. J. Jadhav and N. A. Ramaiah. *Bharatiya Sugar*, 1986, **11**, (13), 23, 25 - 26.

A non-solvent process is described in which sucrose was reacted with methyl stearate in the presence of potassium carbonate and soap to yield a whitish powder having a m.p. of 58°C, a saponification number of 107 and an acid value of 9 which TLC showed to be a mixture of mono-, di- and higher esters with a preponderance of mono-ester. Possible applications are briefly discussed.

Conversion of distillery effluent to a bioassimilable form — a promise for effluent disposal

J. M. Pavgi, R. Paode and R. Bhutani. *Bharatiya Sugar*, 1986, **11**, (13), 29 - 31, 33 - 34.

A brief evaluation is made of four processes for vinasse treatment: incineration, anaerobic digestion to yield methane, anaerobic lagooning and conversion to humus. The last is considered to have greatest benefit and is discussed further in some detail.

Alcohols for petrochemicals

S. L. Venkiteswaran. *Bharatiya Sugar*, 1986, **11**, (13), 35 - 42.

Ethanol production by fermentation and its use as chemical feedstock in place of petrochemicals are discussed in relation to the situation in India.

Molasses and other by-products

R. S. Dubey. *Bharatiya Sugar*, 1986, **11**, (13), 43, 45, 47, 49 - 52, 57 - 58.

By far the greater part of the article concerns molasses utilization, including its use as raw material for the production of various chemicals, and the use of ethanol obtained from it as chemical feedstock; only brief mention is made of cane waste as animal fodder, while cane wax recovery from filter-cake is described more fully.

Use of condensate from spent wash evaporation plant

P. L. Kulkarni and C. L. N. Raju. *Bharatiya Sugar*, 1986, **11**, (13), 59, 61 - 62.

While condensate from the 1st effect in a multiple-effect evaporator used to concentrate vinasse can be used as boiler feedwater, the condensate from the succeeding effects has too low a pH for this application. However, experiments showed that it is of potential value for dilution of molasses used in alcoholic fermentation.

Use of press-mud for crop production

S. P. Kale and B. N. Shinde. *Bharatiya Sugar*, 1986, **11**, (13), 63, 65, 67, 69 - 70.

The value of filter-cake as a fertilizer for sugar cane and other crops is discussed.

Production of quality compost from sugar cane trash and its effect with superphosphate on nutrient uptake and wheat yield

M. S. Bhosale and N. D. Patil. *Bharatiya Sugar*, 1986, **11**, (13), 81, 83, 85 - 86.

An experiment is reported on cane trash decomposition using a mixed culture of *Trichorus spiralis, Aspergillus* sp., *Paecilomyces fusisporus* and *Trichoderma viride* to yield a compost within 4½ months. The original C:N ratio was adjusted by addition of urea. Application with single superphosphate, N and K of compost having a C:N ratio of 40:1 gave a yield which was slightly higher than that given by farmyard manure + NPK.

A report on a technological breakthrough in manufacturing paper from bagasse

Anon. *Bharatiya Sugar*, 1986, **11**, (13), 87, 89, 91.

Reference is made to the success achieved by an Indian paper mill in mechanical pulping of bagasse and producing newsprint based on not less than 50% of this pulp.

Alcohol policy

S. L. Venkiteswaran. *Bharatiya Sugar*, 1986, **11**, (13), 101 - 102.

The Indian policy on alcohol production from cane molasses for use as chemical feedstock is discussed in relation to the competition for available molasses of industrial and potable alcohol.

A tentative paper project attached to a sugar mill

Anon. *Bharatiya Sugar*, 1986, **11**, (13), 111, 113, 115 - 117.

A proposed bagasse paper plant capable of producing as little as 10 tonnes of paper per day that could be erected adjacent to a sugar factory is described. The costs are indicated and the question of effluent treatment and disposal is discussed.

Biostil — technical breakthrough in alcohol manufacture

J. M. Pavgi, A. P. Gore and R. Bhutani. *Bharatiya Sugar*, 1986, **11**, (13), 105 - 109.

The Biostil process of alcohol manufacture from molasses is described and its advantages indicated.

Bagasse — a promising raw material and its technological features

T. K. R. Setty, S. B. Subramanya and B. K. Rajaram. *Bharatiya Sugar*, 1986, **11**, (13), 131, 133, 135, 137, 139, 141, 143, 145, 147, 149.

The physical and chemical properties of bagasse are described and its drying, transportation, storage, depithing, pulp and paper manufacture discussed.

Recent progress in ethanol fermentation from sugar cane and its by-products

J. P. Stupiello. *GEPLACEA Bull.*, 1986, **3**, (11), 9 pp.

A survey is presented of experience with alcoholic fermentation in Brazil. Problems in direct fermentation of untreated cane juice led to trials on various systems. The procedure found to be the most suitable involves 2-stage screening of 13 - 14°Bx mixed juice to eliminate impurities causing blockage and wear of equipment; bagasse removed between the two stages is recycled to milling. The corrosive properties of the juice are reduced by liming to pH 6.0 - 6.2 after which the juice is heated to 102 - 105°C and clarified; this helps to reduce the bacterial count and yields juice of pH 5.6 -5.8 which is pre-evaporated to 13 - 20°Bx and fermented. Some of the pre-evaporated juice is evaporated to 60°Bx and stored to allow fermentation to be carried out during the off-season. The must contains approx. 11% ethanol. About half of the distilleries in Brazil use *Saccharomyces uvarum* IZ 1904, a yeast of higher fermentation efficiency than *S. cerevisiae* and of high thermal tolerance. At a high cell concentration of approx. 100 g/litre and an ethanol content of 7.5 - 11%, fermentation is completed within 5 hr at 92% efficiency. The roles played by factors such as pH, yeast nutrition, cell concentration, etc. are discussed and comparison made between aspects of cane juice and molasses fermentation. Almost all the Brazilian distilleries use the Melle-Boinot batch process, although continuous multi-stage fermentation was introduced in the early 1980's but is still little used since its efficiency is below that of the batch process. Tower fermentation, using flocculation instead of centrifuging for yeast separation, is also being tested but so far has proved less efficient than the other systems mentioned. Reference is also made to the Biostil syrup fermentation process which has the advantage of reduced vinasse yield (0.8 kg/kg ethanol compared with 2.6 kg/kg with molasses fermentation).

Removal from beet molasses by extraction with organic solvents of a fraction toxic to *Aspergillus niger*

M. Druri and Z. Zakowska. *Przem. Ferm. i Owoc.-Warzyw.*, 1986, **30**, (3), 14 - 16; through *Ref. Zhurn. AN SSSR (Khim.)*, 1987, (1), Abs. 1 R390.

A method is proposed of three-stage extraction with organic solvents (methanol, propanol and ether, respectively) for the removal from beet molasses of a fraction containing components that are toxic to *A. niger*. The method is recommended for analytical control of beet molasses.

Carbohydrates from biomass in the perspective of tomorrow's chemical industry

P. L. Soni and A. Agarwal. *Indian Sugar*, 1986, **36**, 405 - 413.

A survey (32 references) is presented of the literature on sucrose, cellulose and starch derivatives and their applications.

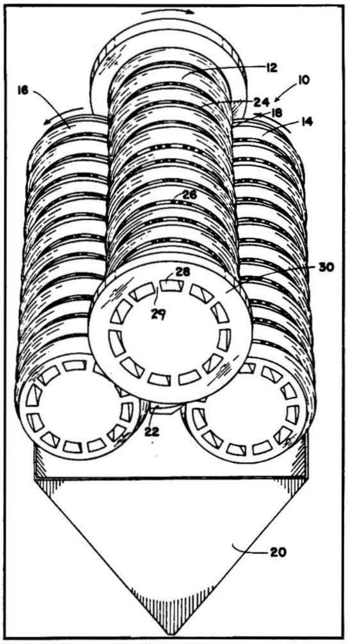
Patents

UNITED STATES

Cane mill roller

J. Bouvet, of San Anselmo, CA, USA. **4,546,698.** September 9, 1983; October 15, 1985.

One or more rollers of a cane mill are provided with circumferential V-grooves 24 at the bottom of which holes 26 open into axial passageways 28 that extend the length of the roller; additional juice squeezed from the cane blanket flows into the holes and along the passageways to be discharged at the ends of of the rollers and thus greatly increase extraction. Generally, one of the rollers (usually the top roller 12) in a 3-roller mill has flanges 30 at the ends which extend over the ends of the bottom rollers 14 and 16 in the areas of maximum pulp compression to prevent extrusion of the bagasse from the sides of the mill. Since these flanges also tend to block the ends of the longitudinal passageways, the passageways are given a spiral configuration; thus, when the bagasse is squeezed between rollers 14 and 12, the juice is discharged at a point B that is well clear of the flange of roller 30. The spiral configuration is also of advantage in that, as the roller passes through the line of maximum compression A, it is supported along its length by the columns of metal 29 between the passageways, which minimizes the risk of fracture and allows higher compression pressures to be applied between the rollers as well as providing for the use of wider channels accommodating a larger number of perforations 26. If a roller becomes worn after extended use, it can be machined down to the tops of the spiral columns 29 and a sleeve, provided with the V-grooves 24 and perforations 26, placed over it.



Butanol production

R. Datta and J. G. Zeikus, *assrs.* CPC International Inc. **4,560,658.** December 5, 1983; December 24, 1985.

Butanol is obtained by fermentation of carbon-containing material such as solubilized starch, sugar syrup, glucose or sucrose with *Clostridium acetobutylicum* in the presence of dissolved carbon monoxide (which gives increased yields of butanol while suppressing formation of hydrogen and acetone). The process is carried out at 34 - 40°C and pH 4.5 - 6.5 for up to approx. 72 (76) hours.

Alcoholic fermentation of sugar cane

C. E. R. Asturias, of Guatemala City, Guatemala. **4,560,659.** September 1, 1983; December 24, 1985.

Ethanol is obtained by combining comminuted dried cane pieces of 0.25 - 4.0 (0.25 - 2.2) (0.5 - 2.2) cm average diameter with *Saccharomyces cerevisiae* and water, and simultaneously extracting sucrose and fermenting it; those cane pieces from which sucrose has been extracted are withdrawn and replaced with a fresh supply of cane. The ratio of cane to water is 1:1.4 - 1:1.7 by weight, and the fermentation is conducted at 95°C, pH 4 - 7 and a pressure of 50 - 200 lb/in^2.

Continuous alcohol production

K. Watanabe, T. Yamada, T. Sazanami and E. Asahara, *assrs.* JGC Corporation and Kansai Paint Co. Ltd. **4,562,154.** August 11, 1983; December 31, 1985.

Ethanol is produced by continuous fermentation of e.g. molasses or refined sugar having a sugar content of approx. 20% w/v (23 - 27%) (>27%) with three immobilized yeasts; A is a yeast from the group *Saccharomyces rouxii*, *Zygosaccharomyces japonicus*, *Z. majar* and *Z. soya* which has an alcohol-producing activity and is superior in sugar resistance to B, which is a conventional yeast from the group *Saccharomyces cerevisiae*, *S. formosensis*, *S. robustus* and *S. carlsbergensis*, while C is an alcohol-producing yeast from the group *Schizosaccharomyces pombo*, *S. octosporus* and *S. mellacei* and has a greater alcohol resistance than yeast B. The fermentation takes place at 30 - 32°C (30°C) in three successive zones, one per yeast, and an ethanol-containing broth is recovered from the final zone with an alcohol content of at least 18% (approx. 20%) by volume.

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Table IV. Quantification of sugars (mg/g fresh weight) after repeated extractions

Sugars	First extraction	Second extraction	Third extraction	Total	% of total sugar recovered in the first extraction
Sucrose	69.00	7.5	5.0	81.5	84.7
Glucose	12.4	1.0	NM	13.4	92.5
Fructose	10.6	1.0	NM	11.6	91.4

NM = non-measureable

purpose of this work was to compare behaviours of sugars along the stalks of different cane varieties. These extraction errors were acceptable because comparative, rather than absolute, values were required and because only one cane stalk was analysed as representative for each variety.

Test of sucrose hydrolysis during extraction

An experiment was carried out to study physical hydrolysis of sucrose at the elevated temperature (80°C) during extraction. Two sucrose solutions were treated for 30 minutes at 80°C, and values are given in Table V.

Table V. Effect of conditions of extraction on sucrose

	pH	Sucrose before extraction procedure, g/litre	Sucrose after extraction procedure, g/litre	% Sucrose recovery after extraction
Solution 1	5.7	7.0	7.2	102
Solution 2	5.8	14.0	13.9	99

Glucose and fructose were not detected and sucrose recovery was about 100%. The pH of the solutions were around the average of the range measured on extraction solution of ground sugar cane (5.4 - 6.0). Inversion of sucrose is highly improbable under these conditions.

Repeatability of extraction method

The results obtained from extraction volume, time and efficiency experiments led us to study repeatability in the following conditions: a single extraction at R = 10, i.e. 50 ml water added to a fresh sample weighing 5 g; and 15 minutes duration at 80°C.

A group of subsamples of ground and dried cane (n = 14) were extracted and the experimental data are shown in Table VI.

Precision calculated in Table V includes HPLC method precision and extraction method precision. Considering results obtained for HPLC method (Table I), precision of the full method is satisfactory.

Comparing with the results of Mulcock *et al.*[14], on sugar beet, water extraction of sugars from ground sugar cane is better than from ground sugar beet. Numerous methods of extraction of soluble carbohydrates from plant material have been described. These include the use of cold water[13,23], hot water[24,25], hot ethanol[9,14,26], and a methanol-chloroform-water mixture[10].

However, several of the methods are time, solvent and energy-consuming and many require the use of expensive equipment. The extraction method proposed here is simple and rapid and can be applied satisfactorily to sugar cane. It permits reliable analysis of different parts of sugar cane stalks without extracting juice which is particularly difficult with nodes.

Table VI. Method accuracy (repeatability)

	Mean concentration x, mg/g fresh weight	Standard deviation	R.S.D., $\sigma_{n-1} . t / \sqrt{n} . x$
Sucrose	68.3	1.9	± 1.6%
Glucose	10.4	0.4	± 2.0%
Fructose	8.6	0.8	± 5.6%

n = 14; t = 2.16, with 95% confidence limits[14]

In this procedure, the efficiency of the physical method used to express juice does not interfere with the sugar cane composition results. Different water-soluble components (sugars and non-sugars) present in sugar cane can be extracted in this way.

This extraction technique permits more complete analysis of water-soluble components of stalks compared with the methods of Abeydeera[19], Charles[20] and Wong-Chong & Martin[21].

Extract purification

The sample after extraction is purified by chromatography on a Waters Sep-Pak C18 micro-column. This procedure ensures complete removal of apolar compounds; in sugar cane samples, mainly coloured products were retained as found by Shaw[13] and Cheng & Wang[27].

Study made on duplicate samples of possible retention of sugars on the Sep-Pak column gives results shown in Table VII.

Table VII. Effect of Sep-Pak treatment on sugars extract composition, g/litre

Sugar	Before	After	% Recovery
Sucrose	1.995	2.00	100%
Glucose	0.505	0.495	98%
Fructose	0.490	0.49	100%

Because of the repeatability of the method used, the recovery of the three sugars after Sep-Pak procedures can be considered to be about 100%. Thus, Sep-Pak treatment does not modify method precision.

Method characteristics including extraction, purification and HPLC analysis, determined during this study, were considered to be satisfactory for use in our researches on sugars distribution in sugar cane stalks.

The following conditions were accepted for extraction:

. duplicate samples

. R = 10 as: 50 ml water volume per

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23 Shaw & Wilson: *J. Sci. Food Agric.*, 1981, **32,** 1242 - 1246.

24 De Vries *et al.*: *J. A.O.A.C.*, 1979, **62,** (6), 1292 - 1296.

25 McKee: *J. Sci. Food Agric.*, 1985, **36,** 55 - 58.

26 Bettenfeld & Voilley: *Sci. Aliments*, 1983, **33,** 1242 - 1246.

27 *Taiwan Sugar*, 1984, **31,** (1), 8 - 12.

5 g fresh sample weight
. duration: 15 minutes
. temperature: 80°c
. one run

Method precision is given in Table VI for each sugar considered (sucrose, glucose and fructose).

(To be continued)

CHEMISTRY

Alkaline degradation of monosaccharides

Part IV[§]: Characterization of oligomeric products formed during the alkaline degradation of monosaccharides

By J. M. de Bruijn[‡], A. P. G. Kieboom[*], H. van Bekkum[*], P. W. van der Poel[‡], N. H. M. de Visser[‡] and M. A. M. de Schutter[‡]

Introduction

Insight into the alkaline transformation of monosaccharides is of importance for industrial sucrose manufacture as the invert sugar present in beet diffusion juices (0.5 - 1.5 g/100 g sucrose) is decomposed during the juice purification by the addition of lime at high temperature (ca. 80°C). Complete degradation of invert sugar is desirable for good process control in sugar manufacture for practical reasons, e.g. formation of thermostable compounds, and preventing colour formation by the Maillard reaction of reducing sugars with amino acids.

As shown in a recent literature survey[1] on the reactions of monosaccharides in aqueous alkaline solution, the mechanisms of the reversible initial transformations of monosaccharides, i.e. ionization, mutarotation, enolization and isomerization, are well understood. However, the complex mechanisms of the subsequent irreversible reactions, by which reducing sugars are converted into both thermostable acid products and unfavourable coloured compounds, are only partly elucidated. For lack of a convenient analysis method for the determination of all degradation products, a systematic investigation concerning the influence of reaction parameters on the degradation reactions involved, e.g. retro-aldolization of monosaccharides, β-elimination of the enediol anion species, and the subsequent benzilic acid rearrangement of α-dicarbonyl intermediates, has not been achieved so far. A recently developed HPLC analysis technique[2] for routine analysis of degradation mixtures made it possible to reinvestigate the alkaline degradation of monosaccharides in more detail. A screening[3,4] of the influence of several reaction parameters on the final product composition revealed that the amount of carboxylic acid products containing more than six carbon atoms, denoted as $C_{>6}$ acids, may be substantial and depends on the conditions of the alkaline degradation reaction. For instance, 40-50% of the monosaccharides are converted into these oligomeric acidic products at an OH^- concentration between 10^{-3} and 10^{-1} M when the initial monosaccharide concentration exceeds 10^{-2} M. The formation of $C_{>6}$ acids has very often been overlooked by workers in this field. Only a few investigators[5-9] have been


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[§] For part III, see reference 3.

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[1] de Bruijn *et al.*: *Sugar Technol. Rev.*, 1986, **13**, 21.

[2] Idem: *I.S.J.*, 1984, **86**, 195.

[3] Idem: *Recl. Trav. Chim. Pays-Bas*, 1986, **105**, 176.

[4] Idem: *12th Int. Carbohydrate Symp.* (Utrecht, 1984), *Abstracts of Papers*, 189; *3rd European Symp. Carbohydrates* (Grenoble, 1985), *Abstracts of Papers*, 318.

[5] Lindström & Samuelson: *Acta. Chem. Scand.*, 1977, **B31**, 479.

[6] Idem: *ibid.*, 1976, **B30**, 691.

[7] Idem: *ibid.*, 1979, **B33**, 531.

[8] Idem: *Carbohydr. Res.*, 1978, **64**, 57.

[9] de Wit: "Gedrag van glucose, fructose en verwante suikers in alkalisch milieu"; *Ph.D. Thesis* (Delft University of Technology), 1979.

aware of the formation of products, up to 40 - 50%, other than the C_1 to C_6 acids. Characterization of products formed besides these C_{1-6} acids in the complex degradation mixtures has been attempted[10-20] most often in relation to colour formation during the industrial sucrose manufacturing process. However, complete elucidation of the nature and structure has not been achieved.

As part of our research on the alkaline degradation of monosaccharides we have studied the nature and structure of the $C_{>6}$ acids by the use of several separation techniques, e.g. gel filtration and precipitation, and by structural analysis techniques, e.g. UV, IR, NMR and GC-MS. The results of this study are presented in this paper.

Experimental

Materials

D-glucose, D-fructose, raffinose and ammonium carbaminate (Merck), maltose (BDH), lead (II) acetate (Lamers & Indemans), and 2,4-dinitrophenylhydrazine (Baker) were reagent-grade and were used without further purification. 1-^{13}C-D-glucose, containing 99% ^{13}C, was obtained from C.E.A., France. Metasaccharinic acid was prepared by selective alkaline degradation of 3-O-methylglucose as described elswhere[21]. The hydrogenation catalyst 5% ruthenium on carbon (Drijfhout, Amsterdam) was activated for 2 hr at 400°C prior to use. Sephadex G-10 (40-120 μm, mol. wt. fractionation range 0 - 700) was supplied by AB. Pharmacia. The cation exchange resin AG 50W-X8 (H^+-form, exchange capacity 1.7 mmol/ml, 100 - 200 mesh) was purchased from Bio-Rad.

Procedures

Alkaline degradation experiments: The reactions were carried out in a 500 ml thermostatted vessel under nitrogen as described elsewhere[3]. During the experiments both the temperature and OH^- concentration were kept constant. After complete conversion of the monosaccharides, as indicated by the termination of the OH^- consumption, the reaction mixtures were neutralized at approx. 0°C by the addition of a weak cation exchange resin (BioRex 70 H) which was subsequently filtered off.
Standard alkaline degradation mixture: Standard alkaline degradation product mixtures were prepared from 1.8 g D-fructose in 400 ml solution (0.025 M) under the following reaction conditions: 0.01 M KOH, H_2O, 78 °C, N_2, 7 hr, 100% conversion. This standard mixture, which was known from earlier experiments[3] to contain 42.5% oligomeric acidic products, was used throughout for the experiments described. In some cases, the reaction mixture was concentrated before use.
Pre-separation of oligomeric products: Two methods were used for the pre-separation of oligomeric acidic products from C_{1-6} carboxylic acids:

(i) Preparative liquid chromatography by size-exclusion using the cation exchange resin AG 50W-X8 (sulphonated styrene-divinylbenzene copolymer, crosslinkage 8%, $-SO_3H$ content 1.7 meq/ml, 100-200 mesh; glass column of 400 mm length and 18 mm i.d.; eluent 0.005 M aqueous H_2SO_4, flow 7 ml/hr). The effluent was collected by an automatic fraction collector.

(ii) Precipitation of oligomeric products by Pb (II) acetate[15d,e] (300 g/l) at pH ~ 12, followed by centrifugation. The residue was suspended in water and Pb (II) was precipitated with ammonium carbaminate (76 g/l) as its carbonate. The dissolved $C_{>6}$ acids were separated from the Pb (II) carbonate by centrifugation. The supernatant was subjected to analysis.

Analysis methods

Molecular weight distribution: The molecular weight distribution of acidic products in alkaline degradation mixtures, without pretreatment as well as after pre-separation of the oligomeric acids, was determined by gel filtration on Sephadex G-10 (glass column of 800 mm length and 26 mm i.d., water as eluent, flow 40 ml/hr, RI detection). D-glucose, maltose and raffinose were used to gauge the Sephadex column.
Determination of unsaturated groups: The carbonyl content of the oligomeric acidic products was determined quantitatively by the 2,4-dinitrophenyl hydrazine precipitation method[22]. The sum of both olefinic and carbonyl groups present in the $C_{>6}$ acids was determined by measuring the hydrogen uptake upon hydrogenation of 10% of the standard alkaline degradation mixture over 200 mg 5% Ru/C in 150 ml water (pH = 7) at 30°C and 1 atm H_2 in an automatic hydrogenation apparatus[23].
UV and IR spectroscopy: UV spectra of aqueous alkaline degradation mixtures were recorded at 20°C and pH 7 on a Pye Unicam SP 8-250 UV/VIS spectrophotometer. KBr tablets of dry samples, obtained by freeze-drying of degradation mixtures, were prepared in order to record IR spectra on a Beckman IR 4210 infrared spectrophotometer.
^{13}C NMR spectroscopy: 1-^{13}C-D-glucose (450 mg) in 100 ml solution (0.025 M) was degraded under standard reaction conditions (see above) and distribution of the ^{13}C label in the various products was determined quantitatively by ^{13}C NMR. The ^{13}C NMR spectra of concentrated reaction mixtures in D_2O were recorded on a Nicolet NT 200 WB spectrometer (50 MHz). Reliable quantitative data were obtained using gated ^{1}H-decoupling, a pulse width of 12.0 μs (45° flip angle),

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10 Kelly & Brown: *Sugar Technol. Rev.*, 1978/79, **6**, 1.
11 Liggett & Deitz: *Adv. Carbohydr. Chem.*, 1954, **9**, 247.
12 Fleming *et al.*: *Proc. 13th Congr. ISSCT*, 1968, 1781.
13 Ramaiah & Kumar: *ibid.*, 1768.
14 Brandes: "Untersuchungen zur Kenntnis von Bräunungsprodukten in technischen Zuckersäften", *Ph.D. Thesis* (Technischen Universität Carola-Wilhelmina, Braunschweig), 1982.
15 (a) Prey & Andres: *Zeitsch. Zuckerind.*, 1973, **98**, 373; (b) Prey & Holle: *ibid.*, 1974, **99**, 113; (c) Prey & Wesner: *ibid*, 1975, **100**, 341; (d) Prey & Andres: *ibid.*, 1976, **101**, 245; (e) Prey *et al.*: *ibid.*, 453, 592, 660; 1977, **102**, 84; (f) Andres *et al.*: *Zuckerind.*, 1978, **103**, 753; (g) Idem: *ibid.*, 1979, **104**, 278.
16 Parker: *Sucr. Belge*, 1970, **89**, 119.
17 Vukov: *Zucker*, 1965, **18**, 168.
18 Ziderman *et al.*: *Carbohydr. Res.*, 1975, **43**, 255.
19 Martinez & Kara-Murza: *Revista Cienc.*, 1976, **7**, 141.
20 Kara-Murza *et al.*: *I.S.J.*, 1970, **72**, 90.
21 de Bruijn: "Monosaccharides in alkaline medium: isomerization, degradation, and oligomerization", *Ph.D. Thesis* (Delft University of Technology), 1986.
22 "Quantitative organic analysis via functional groups", 4th Edn. Eds. Siggia & Hanna (Wiley-Interscience, New York), 1979.
23 Mansveld *et al.*: *Anal. Chem.*, 1970, **42**, 813.

and a pulse delay of 100.0 s. The identification of signals in the spectrum was performed as described elsewhere[21].

HPLC and GC-MS analysis: HPLC analysis of carboxylic acid products was performed as described before[2] (column HPX 87 in the H$^+$ form from Bio-Rad, column temperature 60°C, eluent 0.01 M aqueous trifluoroacetic acid, flow 0.6 ml/min, RI detection). For GC analysis[2] (capillary CP Sil 5 column of 48.5 m length and 0.52 mm i.d., flame ionization detector, temperature program: 5 min at 75°C, followed by increasing the temperature to 280°C at a rate of 8°C/min, 5 min at 280°C) the acidic products were converted into their trimethylsilyl derivatives. Identification of a number of trimethylsilylated carboxylic acids was performed with Varian GC-MS system Mat 44 in which the GC part was identical with that described above. Two types of ionization technique were used: (i) electron impact (80 eV) in which serious fragmentation of the molecules occurred and (ii) chemical ionization (electrons of 200 eV energy were used to generate ions from the methane reagent gas) in order to obtain the molecular weight of the acid compounds.

Results and discussion

Isolation and molecular weight distribution

Quantitative analyses of the C_{1-6} acids in the reaction mixtures from the alkaline degradation of monosaccharides revealed substantial deficits in the mass balance (up to 50%)[3]. GC analysis of the head-space of the reaction vessel during the alkaline degradation showed minor amounts of volatile compounds. This agrees with a carbon analysis of the freeze-dried standard alkaline degradation mixture containing 57.5% C_{1-6} acids, which emphasized that essentially all the carbon orginating from the monosaccharide is still present. The unknown products (42.5%) could not be detected by GC, whereas by HPLC a rise of the base line is observed immediately after the void peak which extends to the first peak of the C_{1-6} acids, i.e. metasaccharinic acid (Figure 1). In view of the properties of the cation exchange resin column applied for this HPLC analysis (see Experimental) the chromatogram points to size-exclusion of a complex mixture of products with molecular weights higher than those for the C_{1-6} acids. Determination of the molecular weight distribution of this alkaline degradation mixture was carried out by gel filtration on Sephadex G-10 (Figure 2). In addition, HPLC analysis of each fraction of the gel filtration established that the grey region in Figure 2 represents degradation products other than the C_{1-6} acids.

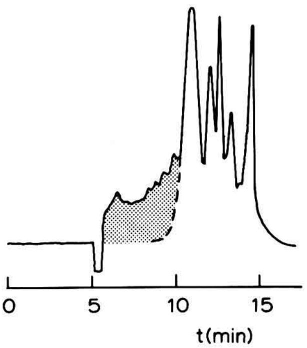

Fig. 1. HPLC separation of the carboxylic acid products in the standard degradation mixture of D-fructose (see Experimental). The grey region represents the oligomeric reaction products

The analytical HPLC results (size exclusion) induced us to perform a preparative separation of 11.2 g of a freeze-dried standard degradation mixture using the cation exchange resin AG 50W-X8 in the H$^+$ form. Three fractions (A, 240 mg; B, 405 mg; C, 645 mg of dry

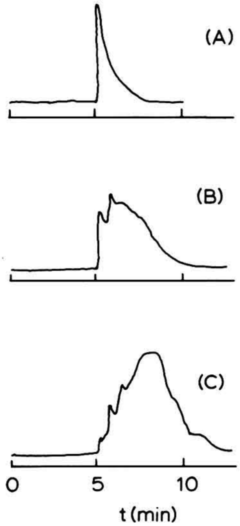

Fig. 3. HPLC analysis of fractions A, B, and C of oligomeric reaction products by preparative separation on AG 50W-X8 (H$^+$ form)

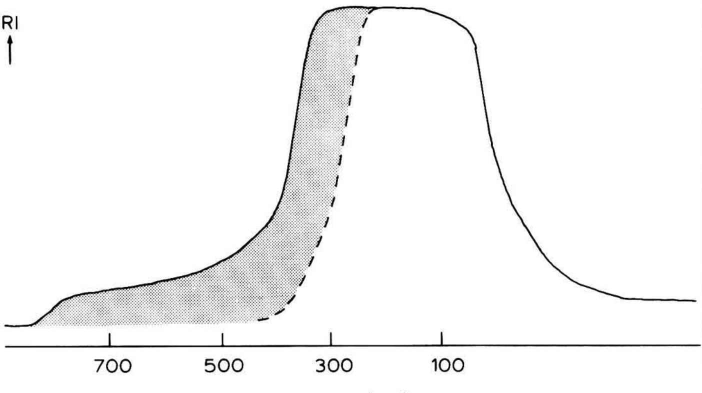

Fig. 2. Gel filtration of carboxylic acids in the standard degradation mixture in order to determine the molecular weight distribution (see Experimental). The grey region represents the oligomeric reaction products

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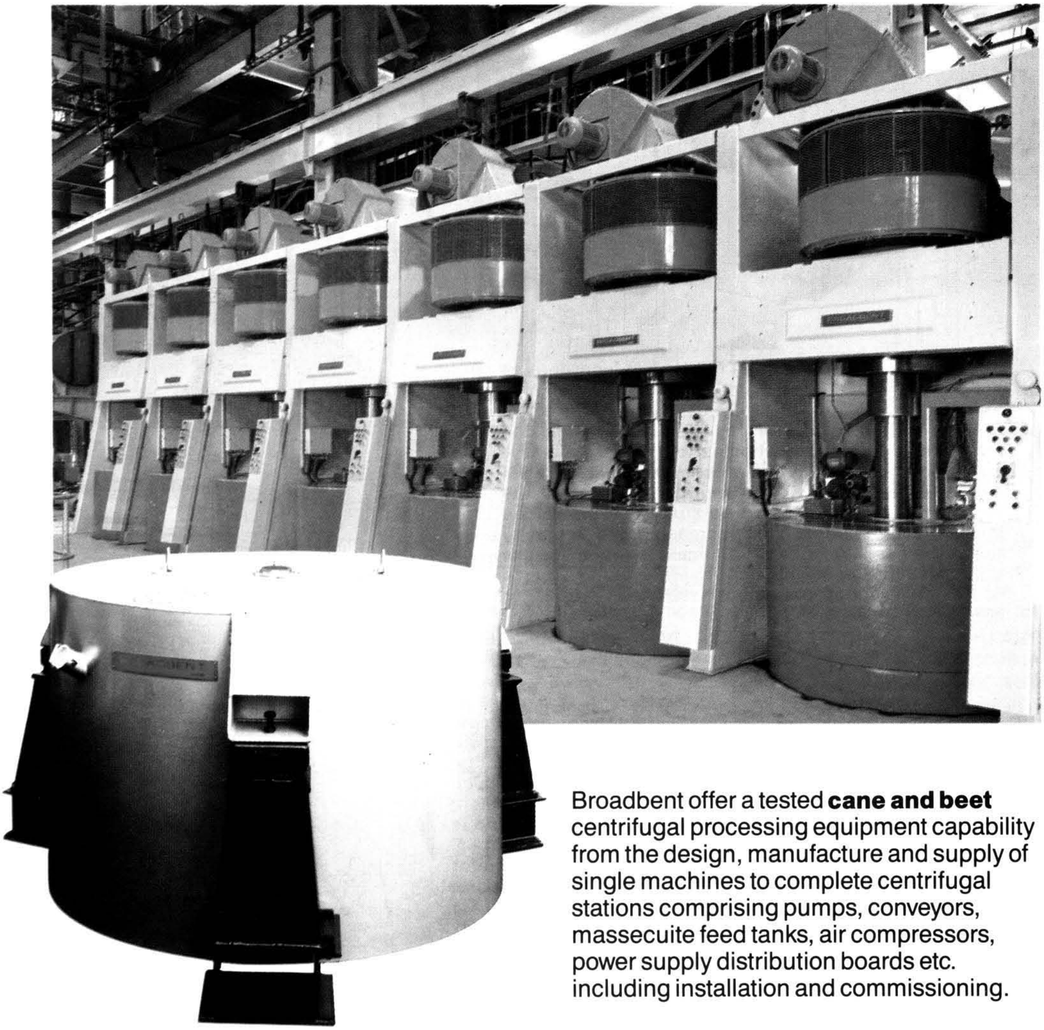

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matter) were collected containing only oligomeric products as demonstrated by HPLC analysis (Figure 3).

The molecular weight maxima regions present in these three fractions according to gel filtration appeared to be ~ 350, ~ 500 and ≥ 700 (Figure 4), which points to products of dimerization, trimerization, etc., i.e. oligomerization of monosaccharide moieties.

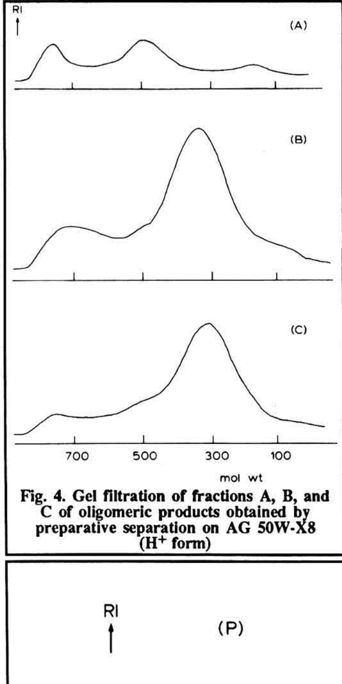

Fig. 4. Gel filtration of fractions A, B, and C of oligomeric products obtained by preparative separation on AG 50W-X8 (H⁺ form)

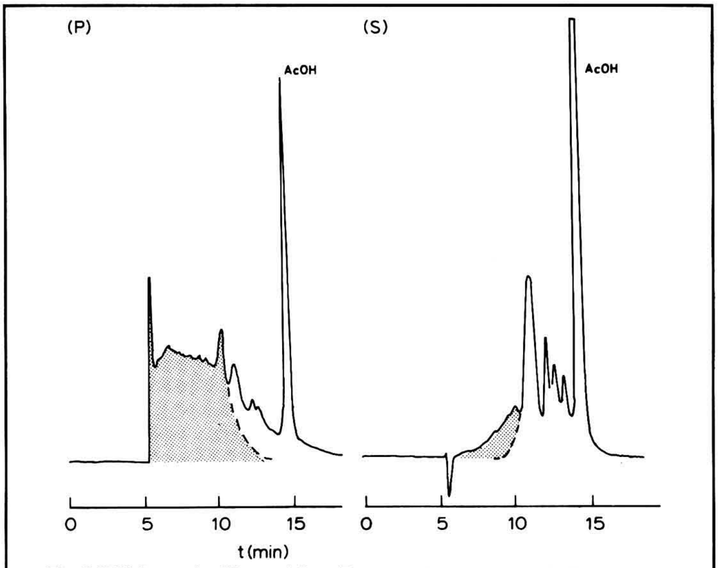

Fig. 5. HPLC analysis of Pb-precipitate (P) and the Pb-supernatant (S). The grey region represents the oligomeric products. AcOH results from the Pb(OAc)$_2$ used

The other method applied for the pre-separation of the oligomeric products was precipitation with Pb(II). In this way the precipitate of 5.6 g of a freeze-dried standard degradation mixture, after removal of Pb(II), yielded 1.0 g of dry product. This product contained mainly oligomeric constituents as shown by HPLC analysis (Figure 5P). The supernatant of the Pb(II) precipitation, on the other hand, contained mainly C_{1-6} acids (Figure 5S). The molecular weight distribution of the oligomeric products (gray region in Figure 6P) shows a close similarity with those obtained by preparative chromatographic separation

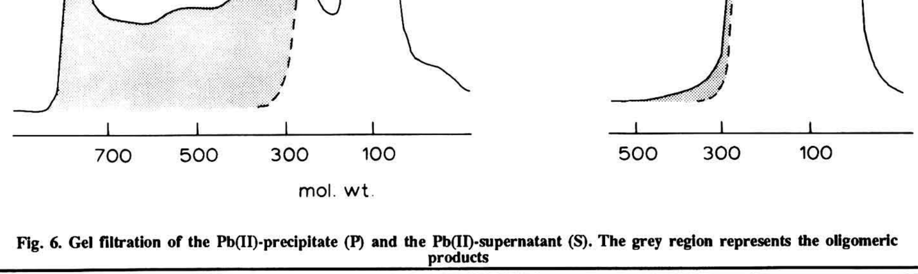

Fig. 6. Gel filtration of the Pb(II)-precipitate (P) and the Pb(II)-supernatant (S). The grey region represents the oligomeric products

on the cation exchange resin. The supernatant (Figure 6S) contains only a small amount of oligomeric products with lower molecular weight, showing Pb(II) precipitation to be an efficient separation technique. Fractions of the oligomeric products obtained by the two methods described have been used for the elucidation of their molecular structure.

Presence of unsaturated and acid groups

A degradation mixture (0.1 M D-glucose, 4.4×10^{-2} M KOH, H_2O, 78°C, N_2, 7 hr, 100% conversion) containing 53.0% oligomeric products[3] was treated with 2,4-dinitrophenylhydrazine in order to determine its carbonyl content. Assuming an average molecular weight of 350 it was found that 8 mol-% of the oligomeric compounds contains a carbonyl group. Hydrogenation of 10% of the standard degradation mixture, containing 0.45 mmol oligomeric products (on the basis of an average molecular weight of 350), over Ru/C gave a hydrogen uptake of 0.086 mmol. Assuming complete saturation, the total content of the unsaturated groups amounts to 19 mol-%, i.e. the oligomeric products contain comparable amounts of carbonyl (8 mol-%) and olefinic (11 mol-%) groups. From the hydroxyl ion consumption during the standard alkaline degradation of D-fructose and the known $C_{1\text{-}6}$ acid composition a value of 1.37 acid groups per C_6 moiety of the oligomeric product is obtained. Therefore, we will denote these products as $C_{>6}$ acids.

UV spectroscopy

During the alkaline degradation of monosaccharides a chromophore is formed as indicated by the appearance of a strong absorption maximum at 265 nm (Figure 7). The rate of formation of this chromophore parallels that of carboxylic acid products. After complete conversion of the monosaccharides the absorption at 265 nm remains constant, whereas the absorption at 420 nm, which is generally used in the sugar industry as a measure of colour formation, gradually increases (Figure 8).

The absorption maximum of the chromophore in aqueous solution at pH_{20} 7 shifted towards lower wavelength (hypsochromic shift) upon decreasing the pH and this was accompanied by a decrease of the absorbance (hypochromic effect). On the other hand, an increase of the pH above 7 only lowers the absorbance. A possible structure for the chromophore which explain these phenomena has been included in Figure 9. Such β-dicarbonyl structures are known to exhibit pH-dependent keto-enol tautomerization phenomena as described for malonaldehyde[24] and 2,5-dimethylcyclohexan-1,3-dione[12] in water. The hypochromic effect caused by an increase of the pH above 9 indicates an ionization of the enol into the enolate structure which agrees with the ionization constants[25] (water, 25°C) of pentan-2,4-dione (pK = 8.24), hexan-2,4-dione (pK = 8.49), and 3-methylpentan-2,4-dione (pK = 9.16).

In conclusion, the β-dicarbonyl chromophore might be considered as a structural moiety of the oligomeric acid products (cf. Table II). As the absorption at 265 nm is a measure of the total content of this chromophore, a linear relation between this absorption and the total amount of oligomeric products is expected to exist if the percentage of chromophore is constant. This, indeed, is the case as demonstrated by Figure 10. The intercept on the absorbance axis is caused by substantial absorption of carboxylate groups (at 190 - 200 nm) which partly overlaps with the absorption of the chromophore.

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[25] "Ionisation constants of organic acids in aqueous solution", IUPAC chemical data series - No. 23, 1st Edn. Eds. Serjeant & Dempsey (Pergamon Press Ltd., Oxford), 1979.

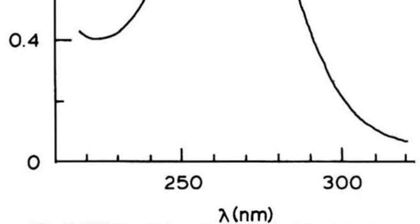

Fig. 7. UV absorption at pH_{20} = 7 of the standard alkaline degradation mixture of D-fructose (0.025 M) after 30 × dilution

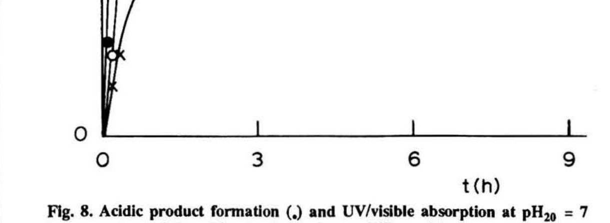

Fig. 8. Acidic product formation (●) and UV/visible absorption at pH_{20} = 7 [as percentage of the ultimate absorption at 265 nm (o) and at 420 nm (×)] as a function of time for the degradation of D-glucose (0.025 M) in 0.01 M aqueous KOH at 78°C under nitrogen

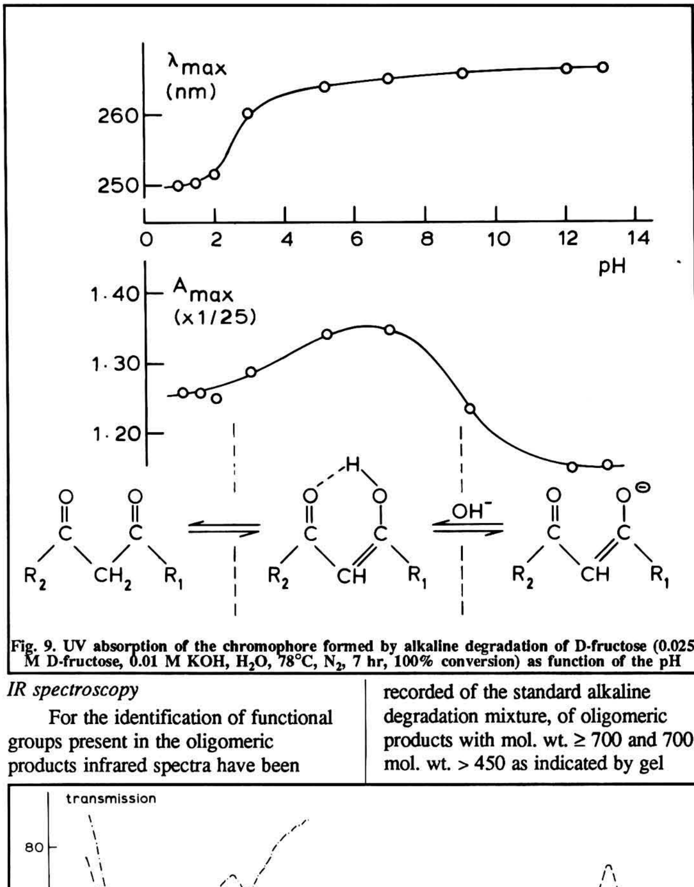

Fig. 9. UV absorption of the chromophore formed by alkaline degradation of D-fructose (0.025 M D-fructose, 0.01 M KOH, H_2O, 78°C, N_2, 7 hr, 100% conversion) as function of the pH

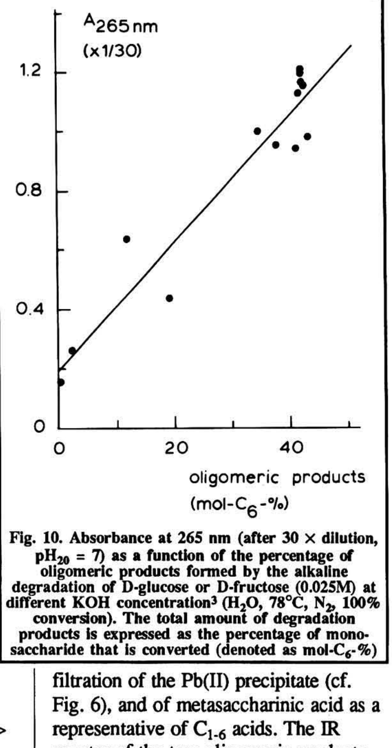

Fig. 10. Absorbance at 265 nm (after 30 × dilution, pH_{20} = 7) as a function of the percentage of oligomeric products formed by the alkaline degradation of D-glucose or D-fructose (0.025M) at different KOH concentration[3] (H_2O, 78°C, N_2, 100% conversion). The total amount of degradation products is expressed as the percentage of monosaccharide that is converted (denoted as mol-C_6-%)

IR spectroscopy

For the identification of functional groups present in the oligomeric products infrared spectra have been recorded of the standard alkaline degradation mixture, of oligomeric products with mol. wt. ≥ 700 and 700 > mol. wt. > 450 as indicated by gel filtration of the Pb(II) precipitate (cf. Fig. 6), and of metasaccharinic acid as a representative of C_{1-6} acids. The IR spectra of the two oligomeric products were identical. From the spectra given in Figure 11 the following characteristic absorption bands[26] appear for the oligomeric products:

(i) A carbonyl band at 1700 cm^{-1} which is in accordance with the β-dicarbonyl moiety indicated by UV spectroscopy.

(ii) Carboxylate bands at 1600 cm^{-1} and 1400 cm^{-1} which support the (poly)carboxylate nature of the oligomeric products, in accord with earlier results[14,16,19,20,27].

(iii) Hydroxyl bands at 3400 cm^{-1} and 1050 cm^{-1} which indicate the presence of alcohol groups along the carbon chain of the oligomeric products.

(To be continued)

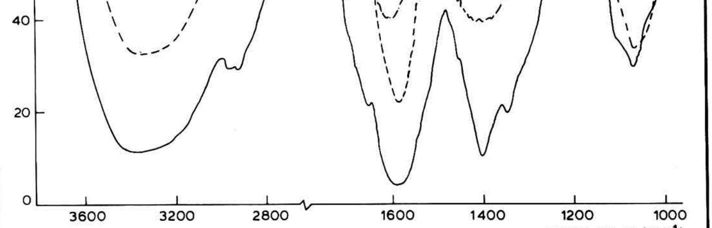

Fig. 11. IR spectra (KBr) of alkaline degradation products as their K^+ salts.
-------- standard alkaline degradation mixture,
— — — metasaccharinic acid,
- — - — oligomeric products with mol. wt. ≥ 700 or with 700 > mol. wt. > 450

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[26] "Practical infrared absorption spectroscopy" 5th Edn. Ed. Nakanishi (Holden-Day Inc., San Francisco, and Nankodo Company Ltd., Tokyo), 1969.

[27] Chou & Rizzuto: *Proc. Tech. Sess. Cane Sugar Ref. Res.*, 1972, 8.

Facts and figures

ISJ readership survey draw

With our April issue we distributed a questionnaire intended to provide information on our readership and, as an inducement to complete and return the questionnaire, entries from respondents were to be placed in a prize draw to the value of $300 in subscriptions or sugar books. Closing date for entries was August 31 and the draw took place on September 1. We are pleased to announce that the winner was Mr. Raymond Rivalland of C.E.R.F, La Bretagne, 97490 Ste.-Clotilde, Réunion.

Argentina sugar exports, 1986[1]

	1986	1985
	tonnes, raw value	
Bulgaria	0	66,729
Chile	3,532	12,934
Paraguay	0	12,443
Peru	0	2,174
Uruguay	0	2,350
USA	51,242	60,546
	54,774	157,176

Sri Lanka sugar factories for sale[2]

The Sri Lanka government plans to sell 49% of its equity in the loss-making state sugar corporation which owns factories at Kantalai and Hingurana. Both are in the east, where rebels have been fighting for a Tamil state. They have operated at only 60% of capacity since opening in 1961, owing to a lack of cane.

Malaysia sugar exports, 1986[3]

	1986	1985
	tonnes, raw value	
Bangladesh	0	8,804
Brunei	2,554	4,693
Fiji	0	12,228
India	21,742	13,044
Indonesia	32,770	1,848
Maldives	1,114	1,630
Pakistan	35,588	0
Singapore	30,715	20,837
Sri Lanka	6,869	30,109
Other countries	589	10
	131,941	93,203

Australia-China trade agreement[4]

Under a new long-term contract, Australia will export 450,000 tonnes of raw sugar to China over three years beginning in 1988. Price details are not known. Queensland officials expect China to import more than that stipulated in the contract, however; in 1986 Australia exported 443,000 tonnes of sugar, raw value, to China — about 16% of Australia's total sugar exports.

Alcohol from bagasse study in Japan[5]

An experimental unit for production of alcohol from bagasse is being set up by Kyowa Hakko Kogyo K.K., Tokyo Engineering Corporation and Kurita Water Industries, all three of which are members of the "Union for Developing New Fuel Oil Technology". The unit can treat 720 kg of bagasse per day for production of more than 200 litres of alcohol.

South Africa sugar exports, 1986[6]

	1986	1985
	tonnes, raw value	
Canada	128,465	171,068
Israel	0	74,155
Japan	363,548	399,781
Korea, South	193,994	227,594
Mozambique	0	10,748
Portugal	0	30,121
Sri Lanka	0	13,537
USA	22,525	67,021
Unknown	165,145	30,299
Other countries	0	902
	873,677	1,025,226

Australian bagasse paper pulp project[7]

Innisfail, Mackay, Bundaberg or the Burdekin may be chosen as the site for a $Aus 460 million mill to process bagasse into pulp for paper production. BHP Engineering and Cane and Pulp Pty. Ltd., of Western Australia, were inspecting bagasse sources and possible mill sites in May. The mill would need an assured supply of 150,000 tonnes, and paper pulp would be exported to South-East Asia for manufacture into paper and cardboard.

Dominican Republic sugar exports, 1986[8]

	1986	1985
	tonnes, raw value	
Dutch Antilles	0	321
Haiti	293	1,086
Morocco	26,780	0
Surinam	0	5,607
Trinidad	0	12,326
Tunisia	34,788	0
USA	357,789	465,085
USSR	51,243	224,689
Other C. America	9,683	12,493
	480,756	721,607

Japan cane crop reduction[9]

Sugar cane output in Japan in 1986/87 fell to 2,240,000 tonnes from 2,640,000 tonnes a year earlier owing to a reduced crop area and a hot, dry summer in 1986, according to the final report on the crop of the Ministry of Agriculture.

Jamaica sugar development finance[10]

The World Bank has approved loans to Jamaica including one of $34 million to help the country's sugar industry to become self-sufficient and meet both domestic demand and the export quotas of the USA and the European Community.

India sugar imports 1986[11]

	1986	1985
	tonnes, raw value	
Brazil	304,585	244,155
Bulgaria	14,020	0
China	28,056	194,028
Cuba	1,541	108,512
Czechoslovakia	65,455	14,586
EEC	316,394	697,891
Korea, South	110,519	170,710
Malaysia	21,539	12,965
Mauritius	13,396	0
Philippines	12,274	64,597
Poland	15,592	79,087
Thailand	68,883	101,344
USA	0	68,499
Vietnam	0	9,519
Yugoslavia	74,224	15,342
	1,046,478	1,781,235

Hawaiian sugar industry make a profit[12]

The Hawaiian sugar industry was profitable in 1986, aided by record yields of sugar per acre, higher operating efficiency and lower costs, and produced a 3.7% return on the $300 million currently invested in the industry. The average price for 1986 production is estimated at $332 per short ton against an average cost of $312.10, $20.67 below the 1985 cost, while raw sugar production increased to 1,042,450 tons, up 30,200 tons on the 1985 figure. Most of this gain came from a 2.5% higher yield which rose to 12.5 tons of sugar per acre. Molasses production rose from 278,000 tons to 291,000 tons, while electricity generated and sold to the public utility rose by 30% to 433 million kWh.

Venezuela sugar expansion[13]

Sugar cane production in Venezuela has risen significantly during the past two years. In 1984, 4,750,000 tonnes of cane were harvested from 85,175 hectares whereas 7,360,000 tonnes of cane were harvested from 138,000 hectares in 1986, an increase of 55%. Sugar production in 1986 reached nearly 600,000 tonnes, which covers about 75% of domestic requirements and it is hoped that selfsufficiency will be reached in 1990.

---

[1] *I.S.O. Stat. Bull.*, 1987, **46,** (7), 1.
[2] F. O. Licht, *Int. Sugar Rpt.*, 1987, **119,** 318.
[3] *I.S.O. Stat. Bull.*, 1987, **46,** (6), 33.
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[5] *Biomasse Actualités*, 1987, (59), 4.
[6] *I.S.O. Stat. Bull.*, 1987, **46,** (6), 42.
[7] *Australian Canegrower*, 1987, **9,** (6), 5.
[8] *I.S.O. Stat. Bull.*, 1987, **46,** (7), 13 - 14.
[9] *Reuter Sugar Newsletter*, June 19, 1987.
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[11] *I.S.O. Stat. Bull.*, 1987, **46,** (7), 28.
[12] *Sugar y Azúcar*, 1987, **82,** (7), 10.
[13] F. O. Licht, *Int. Sugar Rpt.*, 1987, **119,** 365.

PLANT LIQUIDATIONS . . .

GODCHAUX-HENDERSON SUGAR REFINERY, RESERVE, LA., U.S.A.

WASH HOUSE

(1) Parson scale 4,000#/drop
(1) Mingler 525 cu.ft.
(1) Mixer 1,320 cu.ft.
(1) Melter 1,100 cu.ft.

ION EXCHANGE AND CHAR

(32) Char filters, 10' dia. x 20' H, Bone Char, 2,000,000 #
(1) Ion Exchange; (4) 300 cu.ft. resin tanks

POLISHING FILTERS

(4) Industrial leaf filters, 500 sq.ft. SS

PANS AND EVAPORATORS

(1) Evap. 3-effect calandria, 14,400 sq.ft.
(7) Vacuum pans, w/circulator, cu.ft. sizes: (1) 2,080, (2) 2,000, (1) 1,380, (1) 950, (1) 915

CENTRIFUGES & CRYSTALLIZERS

(2) Western States 48 x 36 centrifuges
(9) Broadbent 38 x 30 centrifuges
(4) Continuous centrifuges, (2) BMA
(4) Remelt crystallizers, 1,500 cu.ft., 3,440 cu.ft.
(6) Seed, Mingled Sugar and Strike crystallizers, 816 cu.ft.

GRANULATORS

(1) Link Belt Roto Louvre, 9' x 35'
(4) Hersey 6' dia. x 24'

CONVEYING TO SILO

(1) Richardson 1,500 #/drop scale
(5) Tyler Hummer screens; (4) 4' x 8', (1) 4' x 7', recirculating elevator and conveyors
(3) Redler conveyors, 55 TPH each

POWDER AND SOFTS

(1) Schutz-O'Neil #28 (on 10X)
(1) Mikro Atomizer (on Sucrofine), 2,000 #/hr.

PACKING LINES

(1) Parsons "Burlap" scale & bagger, 50 kg 1980
(1) Burlap bag printer
(2) Fishbein sewing machines, 1980
(4) St. Regis valve packers, 25 to 100#
(1) Thayer paper bag filler, 25 to 100# w/sewer
(1) Union Camp 12/5# line
(1) Consolidated 6/10# line
(1) Consolidated 30/2# line
(1) DPM 24/1# w/box maker (on 10X)
(1) Mateer polyethylene fill & seal
(1) Union Camp 12/5# filler

LIQUID SUGAR

(2) Enzinger 320 sq.ft. SS filter
(1) Industrial 400 sq.ft. press filter
(1) Precoat, 800 gallons
(2) Inverters, 4,600 gallons
(9) Sucrose and Invert storage, 10,000 gal.
(1) DeLaval plate exchanger
(1) American heat reclaim, 774 sq.ft. exch.

UTILITIES

(1) Comb./ Eng. 130,000 #/hr boiler, 500 psi gas
(4) Generals (1) 2,500 kW, (1) 1,500 kW, (2) 625 kW

MISCELLANEOUS

(2) 20,000 gal. FRP tanks
New stores and spares, approx. $1,000,000 worth
Machine shop and maintenance equipment
Pumps - throughout the plant

AT LANTIC SUGAR, CANADA

(1) Silver "Super" beet pilers, (1) new 1983
(1) Marcel Mouyard beet piler, conveyor; with stoner, tare house, weigh system, 1982
(1) BMA-Harland beet pump, 450 HP
(1) BMA vertical diffuser, 5,000 tons per day
(4) Stord-Bartz dewatering presses
(2) CPM pellet mills, 150 HP
(1) Eberhardt vertical lime kiln, rated 200 tons per day
(1) BMA 3,600 TPD clarifier
(5) BMA pressure leaf filters
(1) BMA evaporation system, stainless steel tubes, 90,000 sq.ft.
(5) BMA horizontal crystallizers, 9' dia x 30' L
(1) BMA 52" Type T1000 automatic batch centrifuge
(8) BMA vacuum pans, 1,200 cu.ft., automatic controls
(5) Continuous centrifuges: (3) BMA Mdl. K1000, (2) Silver K1000
(1) Keystone-Volcano 160,000 #/hr boiler, 230 psi, #6 oil/gas, 1982

FROM GREAT WESTERN SUGAR, COLORADO, U.S.A.

(1) 35,800 sq.ft. triple effect evap., SS
(4) Ogden beet slicers, 75 HP, 1975
(1) Silver slope diffuser, 3,200 TPD
(3) Eimco 8' x 12' vacuum filters
(4) CPM pellet mills, Mdl. 75, 100 HP
(2) Raymond 5027 limestone pulverizers
(2) Tyler "Robal" 4' x 12' screens
(1) Trough belt conveyor, 24" ¥ 200' long
(4) 100,000 cu.ft. Butler bolted bins, galvanized
(4) FMC inclined screens, 6' x 12'
(1) Type 316 ELC SS fume scrubber
(4) Calandria vacuum pans, 2,400, 1,870 and 1,325 cu.ft.
(5) U.S. Autojet filters, 600 sq.ft.
(1) Eimco 70' dia. "Clarithickener"
(1) APV SS plate heat exchanger, 1,000 sq.ft.

(1) Bagasse rotary dryer, 10' dia x 36'
(1) Nadler triple effect evap., from 1,500,000 #/day refinery, 1979
(2) Link Belt 5' x 12' x 1 deck screens
(2) Silver continuous centrifugals, Mdl. 104, 30 HP
(4) Werkspoor crystallizers, 10' x 33'
(1) Cane mill 42" x 84" - 5 roll
(10) Nash vacuum pumps, CL203, CL402, CL2002, CL1002, CL3001, CL6001, CL9001
(1) Link Belt Roto Louvre dryer/granulator, SS, 7'6" x 30'(1) APV 40,000 # evap. SS
(2) G.E. 3,000 kW generators 395 psi/45 psi exhaust
((2) GM 900 kW 480V diesel generators
(4) Sweco screens 48" dia. and 72" dia., SS

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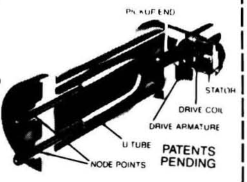

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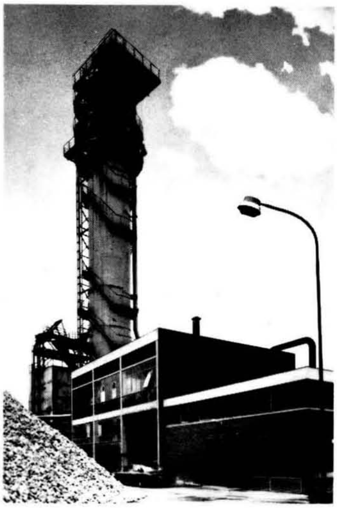

"Eberhardt" lime kiln

Rotating hopper lime kilns

The burning system of these new kilns, for outputs up to 350 tonnes CaO per 24 hr, includes coke mixture firing and forced air draught operation.

Construction features include:

Vertical skip hoist with maximum operating safety.

Rotating hopper for the limestone-coke mixture above the kiln top with special discharge conveyor trough for even distribution of material over large shaft sections without deviation (West German Patent DBP 1,758,155).

Platform installed in the upper part of the kiln with easy access as inspection and working platform for refractory lining. An officially approved winch can also by supplied for personal transport.

High working shaft corresponding to the requirements of the sugar and chemical industries.

Lime discharge through a double gate system, with 4-6 vibrating conveyors according to kiln size.

Level measurement by means of a gamma-ray device.

Fully automatic operation.

Heat consumption: <3750 kJ/kg burnt lime (<900 kcal/kg burnt lime).

The kiln produces lime with high reactivity, with less than 2% residual CO_2 in burnt lime and more than 40% by volume CO_2-content in waste gas.

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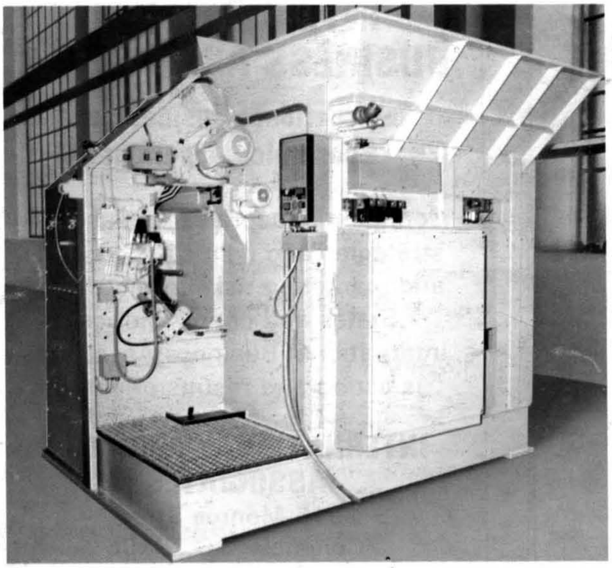

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