FREEZE PRESERVATION OF SUGARCANE MIXED JUICE SAMPLES

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Abstract

Tests have shown that mixed juice samples can be freeze preserved over a period of seven days without detriment to either sucrose, pol or brix content. This permits the preparation of a secure weekly composite sample which facilitates the determination of sucrose in mixed juice by gas liquid chromatography at a far lower frequency of analysis and hence lower cost, than would obtain if individual hourly samples were analysed.

Introduction

Schäffler[1] has reported on a rapid and accurate method for the analysis of sucrose using a gas liquid chromatographic (gc) procedure and the practicability of this method for the determination of sucrose in mixed juice under routine laboratory conditions has been proven and reported by Brokensha *et al*[2]. However gc analysis of individual hourly mixed juice samples for all mills, even on a reduced frequency basis of two hourly samples per eight hour shift per mill, is relatively expensive and provision of a satisfactory weekly composite sample requiring at most a triplicate analysis at a central laboratory facility would give rise to substantial cost savings. Investigation into freeze preservation of juices was initiated by Huletts Research and Development and this work has been summarised by Morel du Boil[3]. The work showed that with rapid freezing and subsequent storage at −25°C, fructose, glucose and sucrose in mixed juice as determined by gc were satisfactorily preserved over a period of one week. However subsequent trials in which pol and brix for the fresh and frozen samples were compared (sucrose analysis was in these latter tests only determined on the frozen samples) were disappointing with the frozen sample results for pol and brix being significantly lower than the fresh results at the 95 per cent confidence level. A similar trend in apparent purity was also observed, thus indicating both deterioration and dilution at some stage of the procedure. However the actual reasons for these trends were not pinpointed.

Following on this work and in the light of a proposal that sucrose in mixed juice be determined by gas chromatography at all mills in the industry, the South African Sugar Technologists' Association's Factory Control Advisory Committee requested the Sugar Industry Central Board to investigate and where necessary adapt the freeze preservation procedure initiated by Huletts Research and Development with the view to its application on an industrial basis as part of the gas chromatographic analysis programme. The experimental details and results of this investigation are given below.

Experimental

In all the trials reported here the mixed juice used was the normal hourly collective preserved with mercuric chloride-potassium iodide solution at a concentration of 0,2 cm^3 preservative per litre of juice. For the fresh sample stream the juice was analysed upon receipt in the laboratory for pol and brix (routine analysis) as well as for sucrose (by gas chromatography).

In the case of the freeze preservation stream, five 20 cm^3 samples were placed in individual polythene sachets (100 micron thickness) which were then sealed with a heat sealer. The sachets were cut from a roll of flattened polythene tubing which when sealed with the 20 cm^3 sample had dimensions of approximately 75 mm^2 by 4 mm thickness. Earlier work had shown the necessity for the juice sample to be contained in a relatively thin layer to facilitate rapid freezing and thereby avoid deterioration.

At the outset it was decided the samples should be stored at −40°C, primarily to have a margin of safety in the event of a temporary power failure at the mill. However as a freezer with the capability to operate at −40°C was not immediately available trials were conducted at −25°C until the lower temperature freezer was obtained.

Freezing, storage and thawing conditions for each trial were as indicated below.

Trial 1

Individual sachets were placed in the deep freezer (−25°C) between two ice packs (also at −25°C) for 60 minutes. Under these conditions the juice sample temperature dropped from ambient to 0°C within ten minutes and had frozen completely after a further ten minutes. After a total time elapse of 30 minutes the sample was down to −10°C.

Upon completion of the ice pack treatment the frozen sachet was stored in the deep freezer at −25°C for 7 days. At the end of this period the sachets were removed from the freezer and placed in the cane drying oven room (temperature ±40°C) to thaw. After 45 minutes the thawing process was complete and the juice temperature had attained ±20°C. The sachets were then transferred to the pol room and wiped down to remove all adhering condensate before opening and transferring the contents to a beaker.

Trial 2

In this trial the sachet of mixed juice was shock frozen by placing it for 1 minute in an alcohol bath cooled to below −60°C with solid carbon dioxide.

After this treatment the frozen sachet was placed in a deep freezer operating at −40°C and stored under this condition for 7 days.

Thawing and analysis (for sucrose and brix) was as for Trial 1.

Trial 3

Test conditions were as for Trial 2 except that the samples were analysed for pol and brix.

Trial 4

In this trial the freezing and thawing procedure was as for Trial 2 except that after 6½ days the sachet was removed from the freezer at −40°C and stored for 12 hours in a freezer at −11°C before thawing. The reason for the latter step was to simulate conditions during transportation of the samples from the sugar mills to a central gc laboratory. During transportation the samples will be held in suitably insulated

containers and low temperatures maintained with frozen ethylene glycol (freezing point −11°C).

Analysis for pol and brix was carried out on the fresh and frozen samples.

Note: In the trials described above sucrose and pol analyses were not carried out simultaneously on the same frozen juice samples solely because of staff workload considerations relevant to the laboratory concerned.

Results and Discussion

Trial 1 (freezing and storage at −25°C).

The overall comparison between the fresh and frozen streams is as follows :

TABLE 1

	Suc. % M.J.			Brix % M.J.		
	Fresh 1	Frozen 2	Diff. 1 − 2	Fresh 1	Frozen 2	Diff. 1 − 2
Mean	11,479	11,464	+ 0,015	13,055	13,064	− 0,009
Std. Dev.			± 0,072			± 0,060
Std. Error			± 0,013			± 0,011
n			29			28
t (calc.)			1,154			0,818
T (95%) from tables			2,048			2,052

It is seen that there is a small decline in sucrose from the fresh to the frozen stream but that this is not significant at the 95 per cent confidence level.

The brix comparisons also show a very favourable comparison with no significant difference between the streams. This would indicate that there has been no moisture uptake or loss of any significance during the preservation and thawing.

Trial 2 (shock freezing at approximately −60°C and storage at −40°C).

The comparison between the fresh and frozen streams is as follows :

TABLE 2

	Suc. % M.J.			Brix % M.J.		
	Fresh 1	Frozen 2	Diff. 1 − 2	Fresh 1	Frozen 2	Diff. 1 − 2
Mean	10,485	10,465	+ 0,020	12,064	12,060	+ 0,004
Std. Dev.			± 0,108			± 0,051
Std. Error			± 0,019			± 0,009
n			31			32
t (calc.)			1,053			0,444
T (95%) from tables			2,042			2,040

The sucrose difference is similar to that found in Trial 1 but again is not significant at the 95 per cent confidene level. However the fluctuations in surose differences are wider in this trial (standard deviation ±0,108) compared with that found in Trial 1 (standard deviation ±0,072). Brix comparisons are very favourable.

Trial 3 (shock freezing at approximately −60°C and storage at −40°C).

Results are as follows :

TABLE 3

	Pol % M.J.			Brix % M.J.		
	Fresh 1	Frozen 2	Diff. 1 − 2	Fresh 1	Frozen 2	Diff. 1 − 2
Mean	10,431	10,442	− 0,011	12,064	12,058	+ 0,006
Std. Dev.			± 0,058			± 0,043
Std. Error			± 0,009			± 0,007
n			39			39
t (calc.)			1,222			0,857
T (95%) from tables			2,024			2,024

It is seen that there is a difference of −0,01 pol units between the fresh and frozen streams but that this is not significant at the 95 per cent confidence level. These pol results are most encouraging and indicate that under routine operations the pol comparison between the fresh and frozen streams can be used as a control to check that there has been no deterioration and/or moisture change. Brix comparisons carried out in conjunction with pol will assist if there is a change in pol to determine whether it is due to deterioration or moisture change.

Turning to the brix comparisons in this trial it is seen that as in the previous trials the comparisons between fresh and frozen samples are most satisfactory.

Trial 4 (shock freezing at appproximately −60°C; storage for 6½ days at −40°C followed by 12 hours at −11°C).

The results are given in Table 4 below.

TABLE 4

	Pol % M.J.			Brix % M.J.		
	Fresh 1	Frozen 2	Diff. 1 − 2	Fresh 1	Frozen 2	Diff. 1 − 2
Mean	9,834	9,849	− 0,015	11,536	11,506	+ 0,030
Std. Dev.			± 0,088			± 0,078
Std. Error			± 0,018			± 0,016
n			24			24
t (calc.)			0,833			1,875
T (95%) from tables			2,069			2,069

The differences found between fresh and frozen samples are not statistically significant and show that in terms of pol and brix there is no change. However the situation for sucrose has still to be determined. In the unlikely event that sucrose is affected, more stringent (lower temperature) conditions will have to be applied during the transportation stage.

Conclusion

The trials have shown that in terms of sucrose, pol and brix, mixed juice samples are satisfactorily preserved over a period of seven days at either −25°C or −40°C, thereby permitting without detriment the analysis of a weekly composite sample instead of indivdual hourly samples.

As regards the choice of temperature (−25°C or −40°C) the latter has been adopted as the lower temperature will provide a greater margin of safety in the event of a power failure at the mill. However it is clear from the first trial that there is no need for the initial shock freezing at approximately −60°C employed in the second trial and that initial

freezing in an alcohol bath stored in the deep freeze will be adequate.

The freeze preservation procedure is relatively simple with minimal time requirement. By permitting a secure weekly composite sample it has enabled gas liquid chromatographic analysis of mixed juice to be introduced at a substantially lower cost than would obtain if individual hourly samples are analysed.

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