SUGAR ENTRAINMENT MONITORING

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Abstract

A sequentially operated auto procedure is now available to flocculate and remove suspended and coloured bodies from condenser and other factory waters. The addition of lead also inactivates bacteria and samples can safely be analysed automatically by a centrally based service laboratory.

Introduction

On-line monitoring of evaporator and vacuum pan entrainment levels is the only method that provides a continuous indication of accidental sugar losses. Dale and Lamusse[1] proposed the use of a flame photometer for entrainment monitoring. The technique has however certain disadvantages:

1) It is an indirect procedure and assumes a constant sucrose to potassium ratio.
2) It is extremely difficult to measure entrainment in injection and condenser waters due to blockages from suspended material and the effect of dilution on the method's sensitivity.
3) Due to the high purity of refinery syrups the potassium flame photometer cannot be used to monitor entrainment during sugar refining.
4) Vapour sampling is virtually essential and it is well-known that true isokinetic sampling is extremely difficult.
5) Evaporators and pan vapour lines must be fitted with sampling valves.
6) Ancillary equipment (sampling probe, condenser, high vacuum pump, barometric leg and water seal) is essential.

Experimental

See Appendix.

Results and Discussion

Sugar specific analyser –

Some of the disadvantages of the flame photometer can be eliminated by using a continuous sugar-sensitive autoanalyser. The automated procedure, developed by Huletts[2] for the determination of sucrose, is based on its reaction with hydrochloric acid and resorcinol in a 95°C heating bath. Sucrose is inverted to fructose and glucose. Only fructose reacts with resorcinol to form an orange coloured compound, which is measured at 520 nm. Ferric sulphate serves as a sensitizer and also cancels out iron interferences.

However, continuous monitoring produced several serious shortcomings:

1) Sediment from condenser waters gradually built-up in the flow cell of the colourimeter, continually off-setting the recorder's baseline.
2) Due to the high background (colour/sediment) of the sample, a blank was needed, forcing us to use a dual channel instrument.
3) Continuous analysers are normally situated on the factory floor. A lack of trained staff especially between 18h00 and 08h00 resulted in many technical difficulties (e.g. power cuts, recorder paper and pens drying-up, dirt and air-bubbles trapped in flow-cell, acid leaks etc).
   The factory environment is also extremely aggressive to sensitive instrumentation and despite protection, equipment was damaged (electronics, switches, pump).
4) As condenser and other factory waters are turbid, filtration is necessary. A continuous belt-type filter (Technicon, paper 2,5 cm wide, length of roll 185 metres) fitted with high and low porosity papers proved unsuccessful.

TABLE 1

Effect of filtration and flocculation on condenser water (Mount Edgecombe Mill).

Sample	% Transmission / 514 nm
Untreated	20%
Filtered (sxs 410)	31%
Filtered (sxs 2043B)	42%
Flocculation	95-97%

Flocculation with basic lead acetate solution produced a water-white solution (Table 1). Lead acetate is also an excellent preservative and inactivates bacteria.[3] Factory water samples treated with lead produced repeatable results 3 days after treatment. The effect of lead acetate addition on calibration standards was also tested and found to be insignificant.

Sequential auto analysis –

The development of a sterile sequential procedure has the following advantages:

1) A blank is unnecessary.
2) As the flocculation process removes most of the coloured and suspended material, flow-cell coating and blockages are no longer experienced.
3) During entrainment studies and/or losses, laboratory staff at factories do not normally have the time to carry out large numbers of manual trace sugar determinations. Samples are collected at the required intervals (every 5 or 15 minutes) from standard sampling points and preserved with lead (see appendix). The samples (30-200) in standard test tubes are then despatched to Huletts R & D for analysis. The Technicon AA II in its present form has a throughput of 30 samples/hour.
4) When heavy losses are being experienced, samples from a variety of sources can be analysed (dunder water, condensates and condenser waters).
5) Sequential analysis can produce almost as much information as continuous analysis. Although the Technicon AA II is reasonably expensive it is at present providing a "same day" service to 5 raw sugar factories.
6) Although the autoanalyser is operated by unskilled staff, downtime is reduced to a minimum due to the presence of experienced analysts.

Applications – During the latter half of the past season the factory water analysis service was used by 4 Huletts factories.

1) Mount Edgecombe – (159 samples) – *Entrainment at Evaporator Station.* The sucrose carry-over in the evaporator tail pipe was monitored using the sequential procedure. It was established that injection water has negligible amounts of sucrose (2 - 4 ppm over a three week test period). It was also established that although the tailpipe water temperature is reasonably steady, the absolute pressure varies quite considerably, causing entrainment.
2) Empangeni – (68 samples) Water samples from high and low pressure boiler feeds, evaporator and pan condensates, juice heaters, cooling tower, tail pipes and dunder water were analysed on several occasions. Sucrose levels were acceptably low in all cases.

3) Darnall – (180 samples) During the latter part of August, this factory experienced abnormally high undetermined losses. Analysis of water samples confirmed this (average ppm 250-350). Despite the high background level, process staff were able to pin point the cause with the aid of the auto-resorcinol/HC1. technique. Remedial action was taken.

The water service was again used in determining average levels of entrainment in individual evaporator effects. Multistage angle iron savealls were subsequently installed with a dramatic reduction in entrainment.[4]

Summary

A comparison is made between indirect and sugar specific analysers. Continuous and sequential analyses are also compared. A sequentially operated autoanalyser with a sucrose-specific methodology offers the most convenient, flexible and economic procedure for monitoring sugar entrainment.

REFERENCES

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3. Scott, R. P. and Falconer, D. (1976). The preservation of juice samples in the laboratory. Huletts Sugar Ltd., Research and Development. Internal Report.
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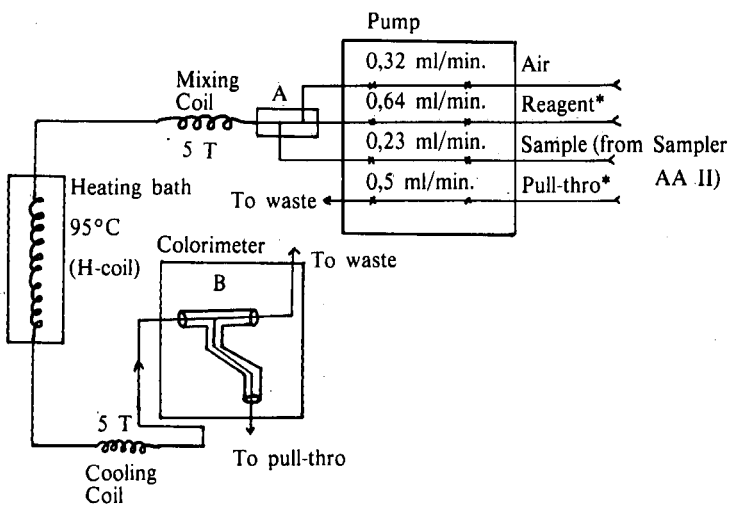

Performance

Linear Range: 0 - 250 ppm without dilution loop ± 7%
Repeatability: better than 2%
on 50 ppm
(n = 20)

Detection limit : less than 1 ppm **

* Acidflex pump tube
A – T-connector (P/N 116-0489-01E)
B – 15 mm x 2,0 mm industrial flow cell and 520 nm filter
** – for example see Fig 2.

Figure 1: Sucrose in factory waters using Resorcinol/HC1 Methodology

Appendix

Analysis of sugar traces in factory water.

Sampling procedure

1) Samples are collected manually at the required frequency by the mill laboratory.
2) An aliquot (20 ml) is added to a labelled test tube (150 mm x 15 mm), containing 10 drops of basic lead acetate solution.
3) The test tubes are corked, packed and dispatched to Huletts R & D.

Auto Analysis – A Technicon AA II is used for the analysis, sampling rate is 30/hour with a 2:1 sample/wash ratio. The methodology for condenser water is outlined in the flow diagram. (Fig. 1)

Preparation of reagents –

1) Basic lead acetate solution as cited in Proceedings of ICUMSA, 16th Session 1974.
2) Resorcinol/HC1 –

Mass of resorcinol = 0,29
Mass of ferric sulphate = 0,39

Dissolve in 500 ml concentrate HC1 by stirring, make-up with HC1 to 1 litre and store in amber bottle.

3) Sucrose stock and working solutions – Prepare in 0,01% benzoic acid solution.

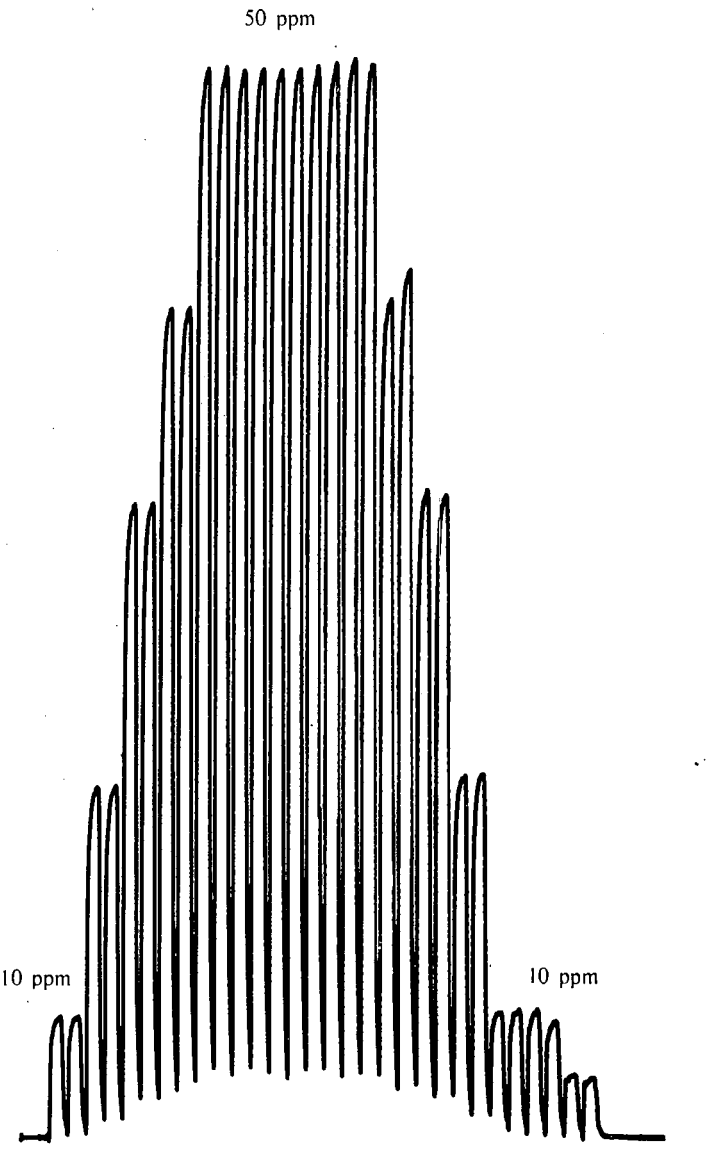

Figure 2: Automated analysis of sucrose in factory waters, calibration standards – linearity and repeatability tests. (range 5 to 50 ppm).