

USE OF THE SRI SUCROSE ANALYSER TO ANALYSE FOR SUCROSE IN A FACTORY LABORATORY

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Abstract

The Sugar Research Institute (SRI) in Australia provided the SMRI with a Sucrose Analyser for evaluation. Although the Sucrose Analyser is designed for online measurement of sucrose concentrations, this application describes the direct measurement of sucrose in factory streams in a laboratory environment. The Sucrose Analyser provides a quick and cost effective way of estimating the sucrose concentrations in factory steams.

The Sucrose Analyser is supplied with a Programmable Logic Controller (PLC) interface including a serial output used for transferring the data to a personal computer. A suite of software programmes was developed to acquire the data from the analyser to be saved for spreadsheet analysis, transferred to a Laboratory Information Management System (LIMS) or to be printed. A calibration procedure was developed to set up calibrations for different factory products. This procedure involved measuring samples with known sucrose (or pol) content taken from the factory stream of interest. A linear regression performed on the data provides the coefficients for estimating the unknown sucrose concentration in a sample from the same stream.

A calibration curve of measured absorbance against sucrose must be set up for each individual factory stream. Correlations for various streams can provide sucrose estimates within acceptable experimental error limits. It was shown that a temperature compensation factor is not needed.

Keywords: sucrose, analysis, analyser, laboratory

Introduction

The Sugar Research Institute (SRI) in Australia has developed a spectroscopic technique for online measurement of the soluble sucrose concentrations in process streams (Bullock *et al*, 2001; Edey, 2002; Edey *et al*, 2003). A patented 2-wavelength near-infrared (NIR) spectroscopy technique is used to determine the optical absorbance of soluble sucrose, although the instrument can be tuned to determine the content of any sugar (*e.g.* fructose or glucose) in solution (Edey and Huberts, 2003). The authors reported that the instrument response when analysing factory process streams is not adversely affected by low background impurities such as particulate or fibrous matter. However, increased levels of mud in mixed juice had a deleterious effect on the repeatability of the measurement as did gas bubbles in the stream.

This Sucrose Analyser was made available for evaluation in the South African sugar industry (Gooch *et al*, 2004). Preliminary findings were positive both in a factory (final evaporator

syrup) and laboratory (clear juice) environment. It was suggested that the solution temperature had an effect on the readings and that the effect would most likely be dependent on the product purity and measurement range. It was further suggested that calibrations be done on a weekly basis to compensate for instrument drift.

The Sucrose Analyser was also evaluated in a batch set-up to be used as a laboratory instrument. The South African sugar industry makes use of polarimetry (factory laboratories) and chromatography (a central laboratory) techniques to determine sucrose values in factory streams and products. A quick supplementary method to estimate sucrose in factory streams for control purposes in order to limit the use of lead-clarified polarimetry to calibrations and daily controls would be invaluable. It was concluded that the requirements in the South African sugar industry are currently such that the benefit in using the SRI Sucrose Analyser would lie in a quick laboratory analysis rather than direct online measurement of factory streams. Initial studies indicated the potential of this application but further work was needed to facilitate the proper implementation and successful transfer of the unit to a factory laboratory environment.

Experimental setup

The Sucrose Analyser was mounted onto a stand and a tap was connected to the outlet of the sample tube. With the tap closed, the analyser tube could hold *ca.* 70 cm³ of sample. The tube was rinsed twice with sample before introduction of the test portion of the sample solution to avoid contamination. Sucrose Analyser absorbances were compared to pol values determined using lead clarification at 589 nm and/or sucrose values determined by high performance liquid chromatography (HPLC).

The Programmable Logic Controller (PLC) interface was connected to a laboratory PC through a RS-323C port and Microsoft HyperTerminal or the SMRI Sugar Baby software suite (see below) was used to collect data. The absorbance value and the analyser housing temperature were recorded.

The SMRI Sugar Baby software suite

A suite of software programmes was developed to acquire the data from the analyser to be saved for spreadsheet analysis, transferred to a LIMS or to be printed. The software monitors the stability of readings before a sample is taken to a relative standard deviation of 0.5%. A calibration procedure was developed to set up calibrations for different factory products. This procedure involved measuring samples with known sucrose (or pol) content taken from the factory stream of interest. Linear regression analysis of the data provided the coefficients for estimating the unknown sucrose concentration in a sample from the same stream. The software also makes provision for temperature compensation.

Results and Discussion

Correlation estimation for calibrations

Three sets of first boiling refined sugar solutions with known sucrose concentrations in the range 0-14% (% m/m) were analysed a number of months apart. Correlation coefficients calculated for the data were consistently higher than 0.996 (maximum error 0.2% sucrose). The resulting slopes indicated a slight drift in the instrument response although no bias was evident, indicating a need for regular calibrations and the routine use of control samples.

C-molasses samples with known sucrose and pol values were diluted 2:1 and analysed. C-molasses is expected to give a lower correlation compared to other factory products since the impurities and therefore possible interferences would be the highest. Similarly, when using pol instead of sucrose C-molasses values would be the most affected since the pol to sucrose ratio of C-molasses is the lowest of all factory products.

The correlation coefficient calculated when using pol was 0.910. For pol values in the range of 27-35% (m/m) this resulted in a maximum estimated error of 2.2% sucrose on sample.

The pol ranges of the individual streams at a single factory tend to be narrow so that many of the differences are obscured by the experimental error. The SMRI has access to samples from various factories and can therefore work with a much wider pol range. To get around this problem it was demonstrated that a set of spiked samples might be used in the place of actual samples in order to manipulate the pol range.

Actual mixed juice samples in the range of 8-12% pol resulted in a correlation coefficient of 0.941 and an estimated error of 1.1% sucrose. Spiked mixed juice samples in the range of 9-12% pol resulted in a correlation coefficient of 0.997. The slope and intercept values for the actual and spiked samples were very similar.

Temperature study

A sample of mixed juice was analysed at temperatures ranging from room temperature (27°C) to 60°C. Each time the temperatures of the solution and the analyser housing were allowed to reach equilibrium while the absorbance values were averaged and collected every 10 seconds. Because of the large difference in the heat capacity of the liquid and the analyser housing the temperature of the sample did not change the temperature of the analyser by more than 2°C regardless of the initial temperature of the sample. Temperature equilibrium between the liquid and the analyser was reached within 90 seconds. The standard deviation of a set of absorbance values of the sample at temperature equilibrium between 30 and 40°C did not exceed 0.0012 AU (previously determined as being the maximum allowable SD) and is therefore acceptable without the need for a temperature compensation factor. However, to circumvent the observed heat transfer effects, reading of absorbance should be delayed for at least 90 seconds after sample introduction.

Conclusions and recommendations

The suite of software programmes make the sucrose analyser suitable for use in a laboratory and will facilitate the transfer of meaningful data to the LIMS.

The linear correlations between absorbances and pol or sucrose are sufficiently high to estimate the sucrose content of samples without significantly increasing the experimental error.

Due to the large difference in the heat capacities of the liquid and the analyser housing the introduction of hot samples in a batch setup is not expected to cause a significant increase in the analyser temperature. To circumvent the effect of this heat transfer on the absorbance reading a 90 second delay will be built into the software. It is still strongly recommended that the sample temperature not be more than 10°C above room temperature.

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