

APPLICATIONS FOR A NEW SUCROSE ANALYSER: PRELIMINARY FINDINGS

M A GOOCH¹, B M SCHOONEES¹ and G R E LIONNET²

¹*Sugar Milling Research Institute, University of KwaZulu-Natal, Durban, 4041, South Africa*

²*Tongaat Hulett Sugar, Durban, South Africa. E-mail: mgooch@smri.org,
bschoonees@smri.org and raoul.lionnet@hulett.co.za*

Abstract

A Sucrose Analyser, developed by the Sugar Research Institute in Australia, was made available for evaluation in the South African sugar industry. The analyser was installed in line on evaporator syrup, at a raw sugar factory. After an original *in situ* calibration with sucrose solutions of known concentrations it performed satisfactorily and repeated calibrations showed no evidence of drift or bias. Tests were carried out for six weeks during which the sucrose values were logged by the factory data capturing system.

The Sucrose Analyser was then set up for laboratory applications at the Sugar Milling Research Institute (SMRI). Both continuous and batch operations were evaluated using juices, syrups and molasses with the appropriate calibrations. Again the analyser performed well. The paper describes how the analyser was calibrated and comments on the results obtained.

Keywords: analysis, sucrose, instrument, NIRS, spectroscopy

Introduction

Sugar Research Institute (SRI) in Australia have developed a spectroscopic technique for online measurement of sucrose concentration in process streams (Edye *et al.*, 2003). A patented 2-wavelength near-infrared spectroscopic (NIRS) technique is used to determine the optical absorbance of sucrose in factory products. This absorbance measurement is not adversely affected by background impurities like particulate or fibrous matter. A tungsten filament lamp is used as the near-infrared source. The sucrose absorbance is measured in a flow through sample cell connected to the process stream using one-inch Swagelok tube fittings. If the process stream contains large amounts of particulate or fibrous matter it is recommended that a filter should be installed upstream of the Sucrose Analyser to prevent damage to or blockage of the sample cell.

The instrument requires a 15 to 24V DC, 0.5A power supply. It can be linked to a factory PLC system or to a laboratory computer via a PLC interface using a two-pair twisted cable via RS422 communication protocol. The PLC interface stores calibrations, converts the digital inputs to 4-20 mA analogue outputs and converts RS422 to RS232C for connection to a laboratory computer.

Online experiments at Darnall

Installation

The SRI Sucrose Analyser was installed at Darnall sugar mill during mid-October 2003, to analyse evaporator syrup online. The trial lasted until the end of November 2003. The

analyser was coupled to a syrup by-pass pipe delivering final effect syrup to a batch pan. The signal from the analyser was routed to the factory data-logging system. A general view of the analyser as installed in the factory is shown in Figure 1.



Figure 1. The SRI Sucrose Analyser (yellow unit) installed in Darnall sugar mill, monitoring syrup.

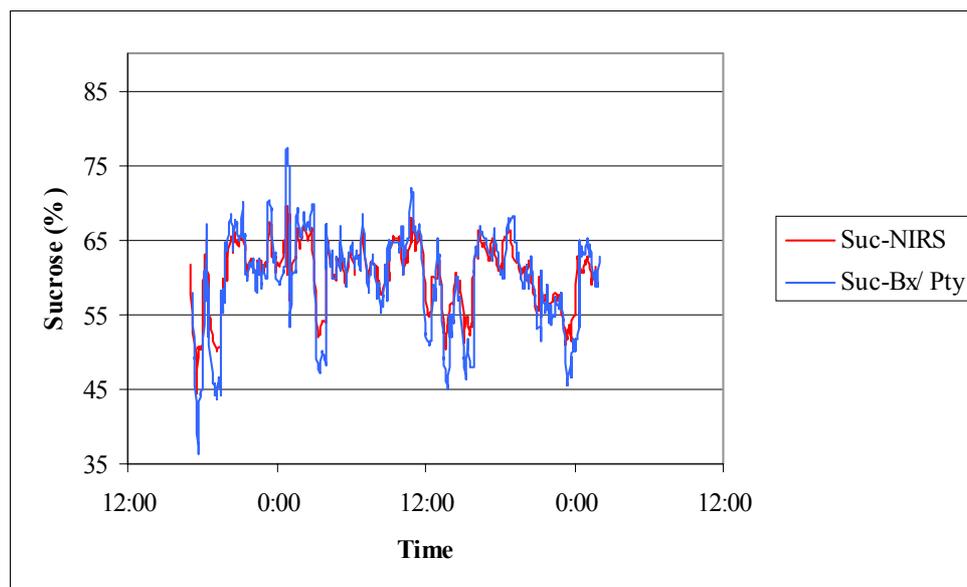


Figure 2. Comparison of calculated and NIRS sucrose.

A typical example of the results obtained from the analyser is shown in Figure 2. These results cover a period of 32 hours from November 5th to 6th, 2003. The factory also measures syrup Brix online using another instrument. The NIRS sucrose values as well as the calculated sucrose (Brix multiplied by the syrup composite purity value for this period) for the same syrup are shown in Figure 2. This figure shows a good correlation between measured NIRS sucrose and calculated sucrose.

Calibrations

The first calibration was done after installation in mid-October. The syrup feed to the analyser was shut off and cold water was flushed through to remove any remaining syrup. Two sucrose solutions (35.0 and 55.5 g sucrose per 100 g solution) were used. These solutions were poured through the analyser at room temperature and the readings were recorded directly on a laptop computer. The following calibration equation was obtained:

$$\text{Sucrose \% (syrup)} = 159.6 \times \text{absorbance reading} - 26.4 \quad (1)$$

Equation (1) was entered into the analyser to predict sucrose in syrup. This was logged on the factory data system. A second calibration was carried out after two weeks of operation (Equation 2).

$$\text{Sucrose \% (syrup)} = 155.8 \times \text{absorbance reading} - 23.7 \quad (2)$$

Equation (2) yielded sucrose values differing from those obtained using equation (1) by about 1%, which was considered acceptable for factory control. Another calibration (Equation 3) was done three weeks later.

$$\text{Sucrose \% (syrup)} = 159 \times \text{absorbance reading} - 23.9 \quad (3)$$

At this stage it is recommended that weekly calibrations be carried out involving two sucrose solutions. It is important that these calibrations are done at constant temperature. This should give an equation accurate within 0.5% sucrose, which is acceptable for the online sucrose measurement of syrup.

Operations

The analyser operated continuously for about six weeks at Darnall. The experiment was discontinued due to shut down for off-crop maintenance. The analyser required no attention from operating staff and was only cleaned before the calibrations were done. There was no visible evidence of fouling or other problems, and the data logging was reliable and error free.

Pseudo-online laboratory trials

Clear juice

The Sucrose Analyser was assembled and set-up in the SMRI laboratory as shown in Figure 3. A tank capable of holding 30 litres, with electrical heaters installed into its side, was coupled to the Sucrose Analyser via a pump and in-line filter. The outflow from the Sucrose Analyser was returned to the tank below the juice level to prevent entrained air from entering the Sucrose Analyser. A T-piece was installed before the Sucrose Analyser to reduce the flow rate by splitting the flow of the juice between the Sucrose Analyser and the return line to the tank. The RS232C output from the Sucrose Analyser was connected to a PC for logging the data.



Figure 3. Sucrose Analyser installed at SMRI.

Clear juices obtained from three different raw sugar factories were used to determine whether geographical location had any effect on the prediction of sucrose results. For each mill, 20 litres of juice was added to the test apparatus and heated to 50°C and a sample taken for HPLC analysis. Two litres of juice were removed from the tank and replaced with an equal quantity of water. When the temperature reached 50°C, another sample was taken for HPLC analysis. This cycle was repeated until the sucrose concentration in the tank was below 4%.

The results from these tests are shown in Figure 4.

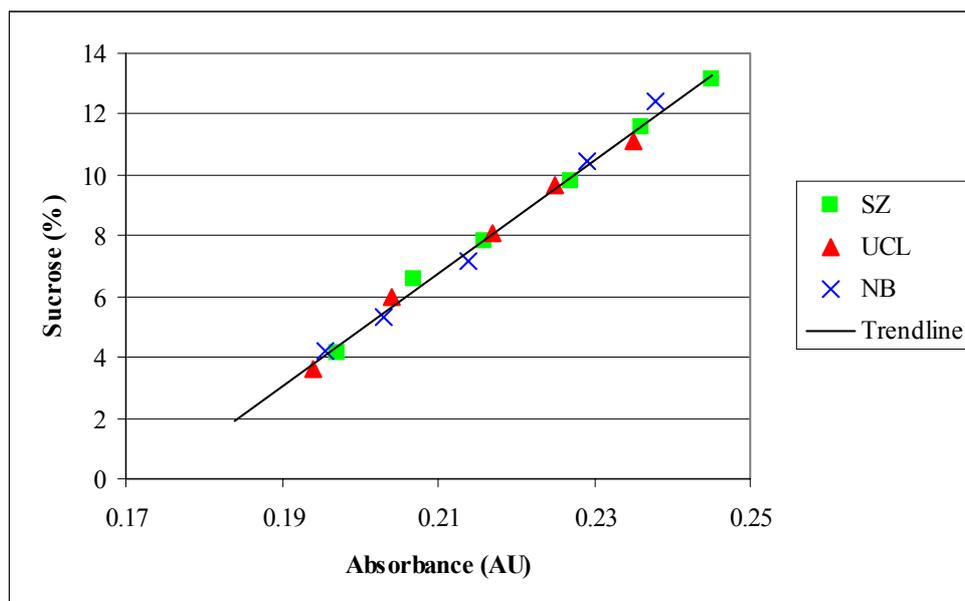


Figure 4. Sucrose – absorbance relationship for selected factories.

The following calibration equation was derived from this data:

$$\text{Sucrose \% (clear juice)} = 186.5 \times \text{absorbance reading} - 32.4 \quad (4)$$

The recorded absorbances from the three different clear juices (Equation 4) indicated no drift and predicted results were within 0.3% sucrose.

Temperature effects

Clear juice at 12% sucrose (HPLC analysis) and clear juice at 20% sucrose (HPLC analysis) concentration were used to determine equations for a temperature range between 20°C and 50°C. Figure 5 shows the absorbance readings logged while increasing the temperature of the juice during its circulation through the Sucrose Analyser. Combining this data with Equation 4 resulted in the following calibration equations for clear juice at 12% sucrose (Equation 5) and clear juice at 20% sucrose (Equation 6):

$$\text{Sucrose \%} = 186.5 \times (\text{absorbance reading} - 0.0005 \times (\text{temp-ref temp})) - 32.4 \quad (5)$$

$$\text{Sucrose \%} = 186.5 \times (\text{absorbance reading} - 0.0004 \times (\text{temp-ref temp})) - 32.4 \quad (6)$$

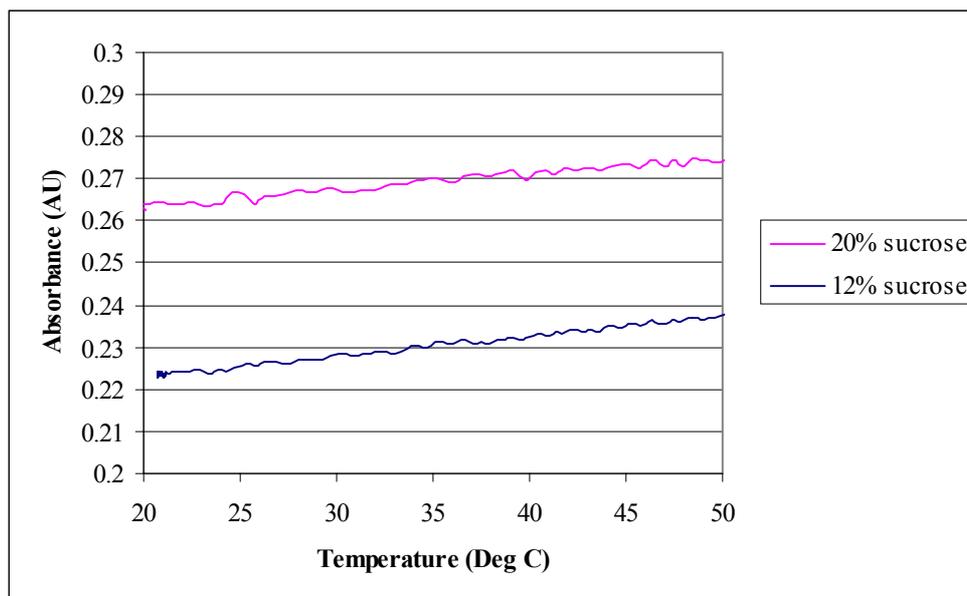


Figure 5. Effects of temperature on the use of the Sucrose Analyser.

Some results published at Sugar Industry Technologists Inc (SIT) (Edye, 2002; Edye *et al.*, 2003) on this Sucrose Analyser have indicated that process stream temperature had no effect on the measurement. This appears to apply only if a temperature compensation factor is programmed into the PLC interface which is provided with the meter. If the analyser RS232 output is directly connected to a computer, Equations 5 or 6 can be used to calculate the sucrose concentration with temperature compensation. Care should be taken to ensure that the temperature of the Sucrose Analyser is the same as the temperature of the product that is measured. In addition, it is important to note that these equations would need to be adjusted depending on the product purity and measurement range.

Batch laboratory trials

The Sucrose Analyser was assembled in a batch set-up in order to be used in a mill laboratory to determine the sucrose content of juices and other factory products. Mounting the Sucrose Analyser in a stand and connected via a funnel to the inlet of the Sucrose Analyser and a tap to the outlet achieved this. Each measurement required approximately 70 cm³ of sample. Estimation of the sucrose content of diluted A-, B- and C-molasses, A-masseccuite and some refinery liquors were evaluated for response linearity and repeatability.

The Sucrose Analyser was rinsed twice each time with the solution before the absorbance was determined. Readings were recorded and averaged over one minute.

Linearity for C-molasses

Thirteen samples of C-molasses with known sucrose values (HPLC analysis) were analysed using the above procedure. A 1:2 dilution gave the best R² value and resulted in an absorbance range of the same magnitude as that obtained with the pseudo-online laboratory trials of clear juice. The molasses samples at no dilution or 1:1 dilution took too long to stabilise due to entrained air as it was poured into the Sucrose Analyser. The 1:2 dilutions were therefore used throughout for molasses measurements. The resulting absorbances were plotted against known sucrose values, as indicated in Figure 6. (Three outliers were removed for statistical purposes according to the Cochran and the Grubbs tests.) The following calibration equation was derived from these data:

$$\text{Sucrose \% (C-molasses)} = (103 \times \text{absorbance reading} - 13.4) \times \text{dilution} \quad (7)$$

The linear equation resulted in a standard deviation of 0.3% (undiluted molasses) and the recommended tolerance based on these data is therefore $\pm 0.9\%$ (2.8 x standard deviation). In cases where such a tolerance is acceptable (e.g. factory control) use of this instrument is ideal. The simplicity of sample preparation (the absence of a filtration/clarification step) and analysis is unmistakable.

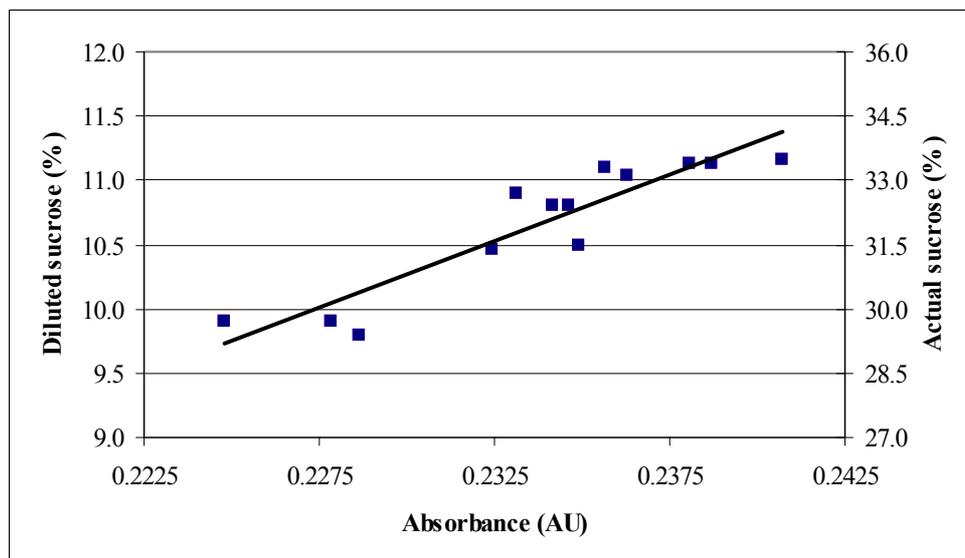


Figure 6. First calibration graph for C-molasses diluted 1:2.

At this stage the light source became defective and another lamp was installed. The new lamp needed some time (approximately 24 hours) to stabilise and, as expected, the new calibration equation was significantly different. Thirty-one samples with known sucrose values (HPLC

analysis) were analysed using the indicated procedure. The resulting absorbances were plotted against the sucrose values to obtain the calibration graph indicated in Figure 7. (Two outliers were removed for statistical purposes according to the Cochran and the Grubbs tests.) The following calibration equation was derived from these data:

$$\text{Sucrose \% (C-molasses)} = (133.7 \times \text{absorbance reading} - 22.1) \times \text{dilution} \quad (8)$$

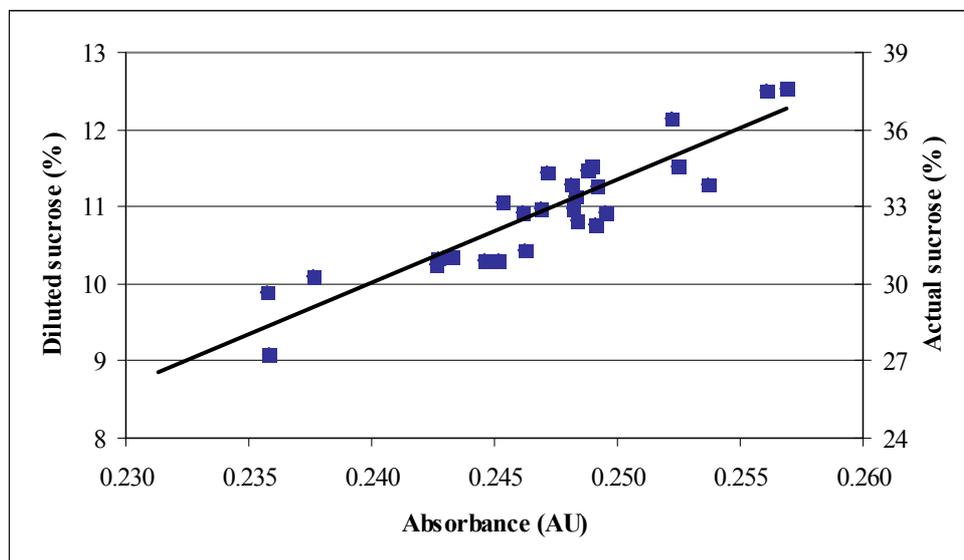


Figure 7. Second calibration graph for C-molasses.

The change in calibration was to be expected with a new light source because of the light intensity increase of the new lamp. However, the new light source required the instrument to be on for some time prior to analysis in order to give reliable readings, and the best results were obtained when the Sucrose Analyser was left on permanently.

Linearity for refined sugar

In order to establish the true linearity of the new light source, eight sucrose solutions were prepared in the concentration range in question and analysed. Figure 8 indicates the excellent correlation that was obtained between the absorbances recorded and the calculated sucrose values. This correlation is a clear indication of the potential of the analyser to be used for other factory products. Since C-molasses generally has the lowest purity of all factory products, it is expected to display the poorest correlation. The following calibration equation was derived using the data from Figure 8:

$$\text{Sucrose \% (refined sugar)} = 195.3 \times \text{absorbance reading} - 31.3 \quad (9)$$

Repeatability

Samples of A-, B- and C-molasses, A-massecuite and two refinery liquors were prepared and analysed 10 times consecutively in order to establish repeatability. The recorded absorbances indicated no drift and results were well within 0.1% sucrose. The Sucrose Analyser can therefore be used to determine the sucrose of all of these products. However, it is imperative, as has been indicated in the pseudo-online laboratory trials, that each product be calibrated separately due to the effect of inherent impurities of the product being measured.

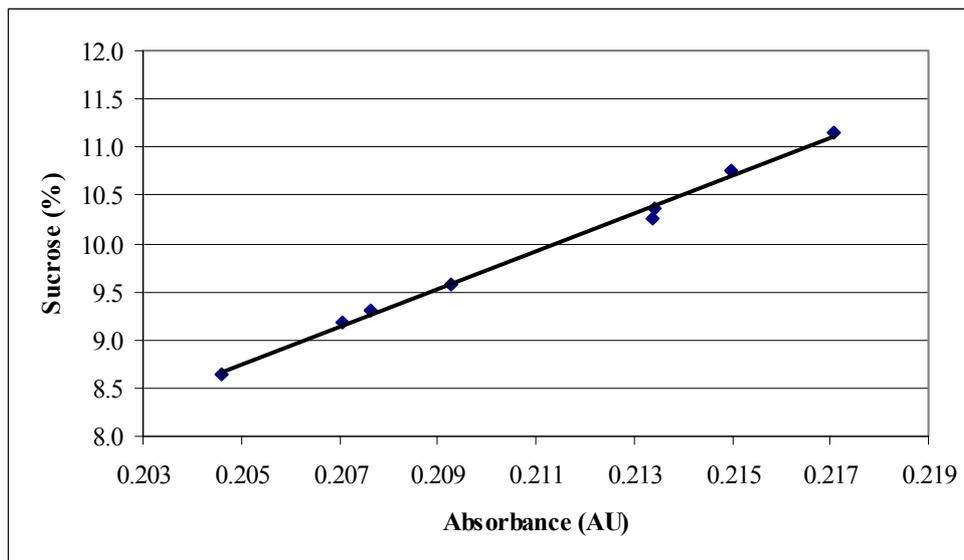


Figure 8. Instrument linearity with refined sugar.

Conclusions

The Sucrose Analyser was installed online at Darnall monitoring syrup and in the SMRI laboratory for pseudo-online monitoring of clear juice. It was also set up for batch laboratory trials at the SMRI. The results from these experiments showed that the Sucrose Analyser could adequately measure sucrose in factory products for factory control purposes. A temperature compensation factor must be programmed into the PLC interface (provided with the meter) to minimise the effects of temperature. It is imperative that the Sucrose Analyser be calibrated separately for each factory product to accommodate the effect of inherent impurities. For online measurements an inline filter must be installed to prevent blockages in the measuring cell.

The Sucrose Analyser can also be used in the laboratory to simplify the measurement of sucrose. Filtration and clarification would not be required, which would result in time and cost savings. It provides a RS232C interface, which would be ideal for laboratory instrument automation. If the RS232 interface is used the temperature compensation equation must be used to avoid errors due to temperature fluctuations.

Acknowledgements

The authors would like to acknowledge the contribution made by the Darnall mill personnel. At short notice and nearing the end of the season, the analyser and peripheral equipment were assembled, installed in the factory and linked to the data logging system. The authors also thank Mr D Joseph (SMRI workshop) for setting up the laboratory test apparatus, and Ms E Ramphal and Ms R de Jager (SMRI) for carrying out the laboratory tests and analyses.

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