POSTER SUMMARY

# AN SMRI-CTS COMPARISON OF NEAR INFRARED (NIR) POLARIMETRY

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## Abstract

Leaded pol analyses are used in sugar factories as an estimation of sucrose for factory control purposes and for cane payment. Health and environmental concerns associated with the use of lead for sample clarification has put pressure on the use of this method. Near infrared (NIR) polarimetry has been investigated as an alternative worldwide and the South African sugar industry has decided to replace the leaded pol analysis with NIR polarimetry for factory control purposes. The new method uses a NIR light source which does not require the test solution to be of low colour. Lead clarification can therefore be replaced by a simple filtration step to remove solution turbidity.

Suitability of the use of NIR polarimetry by the Cane Testing Service (CTS) was established using a number of comparisons between CTS centres and the SMRI during the 2004/05 season. Previous comparisons were done only at the SMRI. Results are presented and discussed together with the change in method, equipment and other considerations.

*Keywords:* analysis, polarimetry, near infrared, NIR, lead

## Introduction

The South African sugar factory laboratories are in the process of converting from the use of leaded pol to NIR pol. Health, safety and environmental reasons dictate that the use of lead will not be viable for much longer. NIR polarimetry allows for the pol and Brix sample preparations to be combined; sample preparation is simpler (reduced workload on staff) and quicker due to the introduction of pressure filtration which should reduce the possibility of sample degradation. The costs of lead subacetate and waste disposal will be eliminated and result variations and biases due to 'batch' variations of lead are eliminated, giving more consistent results.

A number of seasonal and other comparisons have been reported since 1997 (van Staden and Mdlalose, 2000; Schoonees, 2003a,b) to compare the use of leaded pol and NIR pol. Although a small systemic difference in results was reported, the Factory Control Advisory Committee agreed that it was acceptable for factory performance purposes (Anon, 2004a). Accordingly factory laboratories are now implementing the new method. Since the South African cane payment system is based on sucrose calculated from pol to sucrose ratios, payment should not be affected (Schäffler and Smith, 1978). For cane payment, the number of analyses done in a season for most commercial growers is large enough to still give a representative average for field and seasonal data. The cane quality of smaller growers is highly variable and averages are often applied. Even in cases where payments are based on pol, differences should not preclude NIR pol from being

used. Certainly, the high purity of raw sugar ensures that leaded and NIR pol values are virtually the same for sugar analysis (Paton *et al.*, 1993).

A final comparison using the most recent method and equipment available in the South African industry was conducted between the SMRI and CTS at Noodsberg (NB) and at Darnall (DL) in the 2004/05 season. NB experienced exceptionally high viscosities (gums) during 2004/05 due to a drought in the previous year and severe frost with resulting infections around October 2003.

## Experimental

Samples were allowed to reach room temperature. A 200 $cm^3$ sample was mixed with 6 g Celite Filtercel and pressure filtered through a Postslip medium white filter paper in the Schmidt and Haensch (S&H) Autofilt filtration unit at 1-2 bar pressure. The filtration procedure was repeated with a fresh sample and filter paper to avoid contamination in the filtration unit. A prefilter was used for the second filtration where the initial filtration time exceeded 30 seconds. The first 20 $cm^3$ of filtrate was discarded and about 80 $cm^3$ of filtrate collected for analysis of pol and Brix. The S&H NIR Polartronic and Bellingham and Stanley RFM 510 refractometer were used at the SMRI and the S&H NIR Saccharimeter and DUR-W refractometer at the CTS.

## Results

Samples collected at the factories were subsampled and frozen for analysis at the SMRI. Four sets of samples composed of DAC, mixed juice and bagasse were collected and analysed at the factory for leaded pol and NIR pol. Frozen sub-samples were sent to the SMRI where they were analysed for NIR pol only. Set 1 was collected in July (NB), set 2 in September (NB), set 3 in October (NB) and set 4 in November (DL). A summary of the result statistics is shown in Table 1.

**Table 1. Summary of result statistics.**

| Sample set | n | NIR pol difference (SMRI - CTS) | | |
|---|---|---|---|---|
| | | Average | Standard deviation | Sum of squares |
| DAC combined | 75 | -0.02 | 0.09 | 0.63 |
| MJ combined | 85 | -0.02 | 0.16 | 1.25 |
| Bag combined | 73 | 0.00 | 0.05 | 0.18 |

DAC = direct analysis of cane extract, MJ = mixed juice, Bag = bagasse extract,
SD = standard deviation, SS = sum of squares

When evaluating method uncertainty ICUMSA recommends the use of the Horwitz formula, which takes into account the close correlation between the relative standard deviation (rsd), and the concentration of the analyte (Godshall, 2002). The calculated Horwitz ratio should generally not exceed a value of 2. Horwitz ratios were calculated for the SMRI and CTS NIR pol values based on time of the season and overall and all ratios were less than 1. The NIR pol method is therefore reproducible.

The absolute reproducibility for NIR pol on raw sugar recommended by ICUMSA (0.27 units) (Anon, 2004b) is slightly higher than that of leaded pol (0.25 units) (Anon, 1994), indicating that a bigger inherent method variation can be expected. Still, results are encouraging and will probably improve with experience and proper training of the testers. Based on these values the absolute difference between results from SMRI and CTS was 0.18 for DAC, 0.23 for mixed juice and 0.10 for bagasse. The recommended tolerances are therefore ±0.09, 0.12 and 0.05 for DAC, mixed juice

and bagasse respectively. Omission of some of the DL NIR pol results (>0.30 difference) for mixed juice resulted in a recommended tolerance of ±0.09 units (absolute difference of 0.17 units; 72 samples).

## Discussion

Since 1982 the major factory performance figures and overall cane payment in the South African sugar industry have been based on sucrose content measured by gas chromatography, rather than pol, since pol is not always a very good estimate of sucrose. The type of polarimetry method used will therefore not affect either the cane payment system or sucrose factory performance figures.

The recommended tolerances are well below internationally recognized repeatabilities for raw sugar and should be acceptable for use by the industry. Tolerances are expected to decrease with training and experience of the testers.

Initial problems with the filtration of highly viscous samples under pressure were solved by the use of a glass fibre prefilter. In general, a prefilter was used with samples that took more than 30 seconds to filter.

A significant reduction in cost of analysis, safety risks, disposal costs, time for sample preparation and negative environmental effects should provide substantial motivation for adoption of the method.

## Conclusions

NIR pol results obtained using the most recent methods and equipment available were found to be reproducible between the SMRI and two CTS centers for DAC, mixed juice and bagasse samples.

## REFERENCES


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