

REFEREED PAPER

LIGHT AT THE END: A SEASON OF COMPOSITE MJ NIRS ANALYSIS

WALFORD SN¹ AND NAIDOO S²¹*Sugar Milling Research Institute NPC, c/o University of KwaZulu-Natal, Durban, 4041, South Africa*²*Cane Testing Service, South African Sugar Association, PO Box 700,
Mount Edgecombe 4300, South Africa**swalford@smri.org seelan.naidoo@sasa.org.za*

Abstract

Near Infrared Spectroscopy (NIRS) has held the promise of being used as an alternative method for composite Mixed Juice (MJ) analysis at the Sugar Milling Research Institute NPC (SMRI) for a protracted length of time. The lengthy adoption uptake has been the result of technology issues and in part due to the importance of ensuring that the technology provides results equivalent to the conventional methods as a consequence of their use in the cane payment system. SMRI identified and resolved the technology issues and has developed robust prediction equations for the analysis of brix, pol and sucrose in weekly composite MJ samples. These equations were used for parallel testing of the NIRS analysis method against conventional methods at SMRI for all South African tandems throughout the 2014-2015 season. This paper briefly describes the technology issues and how they were overcome, compares the results obtained from the parallel testing and investigates what effect replacing conventional results with NIRS results would have had on the calculation of the mill-based Recoverable Value.

Keywords: NIRS, MJ, calibrations, predictions, RV

Introduction

Near Infrared Spectroscopy (NIRS) is a universally accepted secondary analysis technique that is used in many industries. The Sugar Milling Research Institute NPC (SMRI) has been investigating the use of NIRS as a possible alternative technique for the analysis of factory streams for more than twenty years (Schäffler *et al.*, 1993; Schäffler and de Gaye, 1997; Schäffler, 2000; Schäffler, 2001; Simpson and Oxley, 2008; Simpson and Naidoo, 2010; Simpson and Naidoo, 2011; Naidoo and Walford, 2013). The NIRS method was shown to be accurate and reproducible for analysis of brix, pol, sucrose, glucose, fructose and conductivity ash when used at SMRI for weekly composite Mixed Juice (MJ) samples (Simpson and Oxley, 2008). The attraction of NIRS as an alternative analysis technique is the rapid simultaneous estimation of multi-components in sample streams with minimal sample preparation and elimination of environmentally unfriendly chemicals such as lead acetate.

SMRI has analysed weekly tandem composite MJ samples for cane payment and factory control since 1996. Official Methods were used to report brix, pol and sucrose using both 'conventional' methods (refractometry, polarimetry and gas chromatography) and NIRS (during the 2011-2012 season). Methods for Factory Control were used to report glucose, fructose and ash using conventional methods (gas chromatography and either sulphated or conductivity ash) and NIRS (2011-2012 season). The brix, pol and sucrose results are supplied

to the South African Sugar Association Cane Testing Service (CTS) division for cane payment purposes whilst the glucose, fructose and ash results are supplied to factory personnel for mill control purposes.

With the introduction of the NIRS technology at multiple mill laboratories at the beginning of the 2012-2013 crushing season, untoward results were reported requiring the withdrawal of Official Method status for NIRS MJ analysis method. The conventional methods of analysis of weekly tandem composite MJ samples were therefore reintroduced at SMRI. The root cause of the untoward results at the mill laboratories was determined and a solution adopted toward the end of the 2012-2013 season. The results of the investigation and the adopted solution are presented in the first part of this paper. Prediction equation development continued during the early part of the 2013-2014 season with the equations being used in a preliminary comparison of the NIRS and conventional methods during the second half of the 2013-2014 season. Further updating of the prediction equations took place at the end of the 2013-2014 season and parallel analysis of composite MJ samples by both conventional and NIRS analysis methods was then undertaken for the complete 2014-2015 season. The results of the comparisons of the methods are presented in this paper.

Investigation of non-reproducible results

The NIRS technology was introduced to the mill laboratories at the beginning of the 2012-2013 season. The instrument and analysis methods were based on the successful procedures developed at SMRI over the previous five years. Prediction equations were developed using a Bruker MPA NIRS instrument in the transmission mode with samples introduced using a 1 mm OS flow-through cell. The same type of instrument and cell were supplied to the mill laboratories. With the reporting of untoward results at the beginning of the season, an investigation of possible reasons was instituted. Factors reviewed included possible instrument differences, sample introduction problems and a comparison of the flow-through cells. It was shown early in the investigation that the flow-through cells were the major contributor to the untoward results.

The OS cell type originally used by SMRI in the development of successful MJ analyte prediction equations is manufactured from 'special optical glass' made from exceptionally pure raw materials. Inspection of the background absorption spectra of the dry OS flow-through cell used in the development of the prediction equations at SMRI showed that the OS type glass absorbed in a broad range from 7500 to 4000 cm^{-1} (wavenumber). This is in the same range as the $-\text{OH}$ stretching band that is found in water. The background absorption of the OS cell occurs in an absorption region that was used in the development of the original prediction equations and was therefore incorporated into the partial least squares prediction equations. Inspection of the absorption spectra of the OS cells supplied to the mills showed that the shape of the broad absorption was not consistent across all the cells (Figure 1). As the prediction equations included the $-\text{OH}$ absorption background from the SMRI cell, differences in the background absorption of the cells supplied to the mills resulted in untoward predictions. The supplier of the cells confirmed that the broad absorption was due to residual water in the OS glass from the manufacturing processes. The insert in Figure 1 shows that cells that had similar absorption spectra still differed sufficiently to produce inconsistent results. SMRI, as part of its NIRS development program, had used two different OS flow-through cells over a three-year period with no apparent problems. Investigation showed that the two cells had come from one production batch and had almost identical absorption spectra in the region used in the development of the prediction equations.

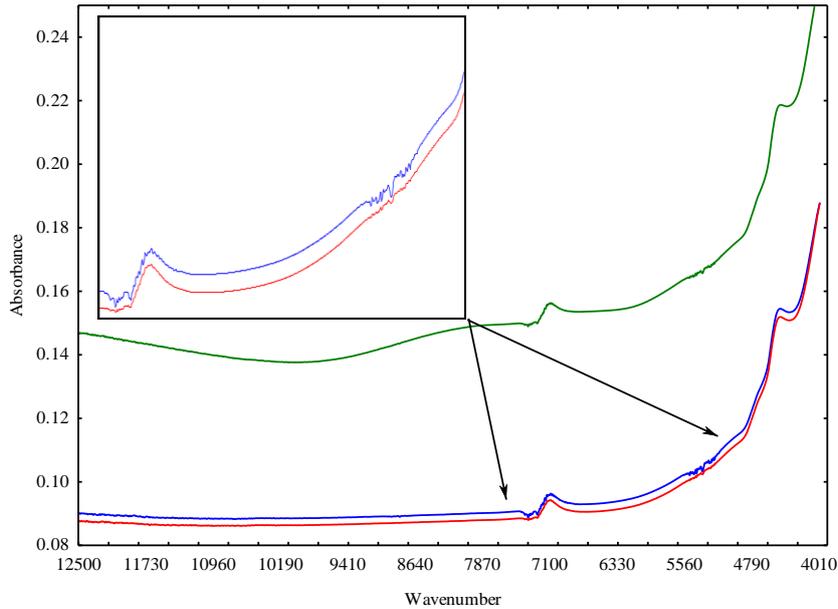


Figure 1. Absorption spectra of three OS type cells showing differences in the background absorbance of the cells. Insert shows the fine absorption differences between similar cells due to –OH absorption in the 7500 to 4000/cm region.

The instrument supplier (Bruker) suggested using an alternative QS type cell manufactured from synthetic Suprasil quartz (high purity and homogeneity) which they routinely supplied to their customers. Bruker supplied three ‘matched’ QS cells for checking at SMRI. Initial checks displayed unacceptable variability in the background absorption spectra of the cells due to distinct –OH peaks (Figure 2). In discussion with the cell supplier, it became apparent that the cells were ‘matched’ to within 1% in path length and that no check was performed as to the variance in cell absorption across the NIRS wavelength range.

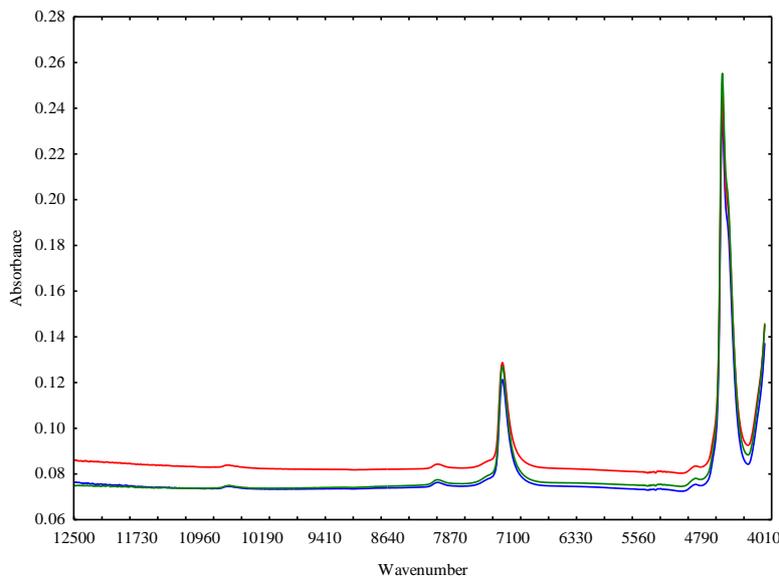


Figure 2. Absorption spectra of three ‘matched’ quartz QS type cells showing differences in the background absorbance of the cells. Peaks at 7100 and 4500/cm are due to absorption by –OH bonds.

Discussions with the cell supplier (Hellma) were initiated to determine the availability of ‘water-free’ quartz cells. Samples of QX cells (synthetic Quartz free from –OH absorption) were supplied for evaluation, found to have no absorbance due to –OH bonds, and proved suitable for MJ analysis (Figure 3). The OS cells initially supplied with the mill instruments were replaced with QX cells. Going forward, mills will be responsible for the replacement of damaged cells. A procedure was developed to ensure that suitable replacement cells are available for mill laboratories. Bruker will undertake to match all cells supplied to the South African sugar industry, whilst SMRI will check the cell absorption spectra and sample reproducibility of the cell before releasing the cell to the mill.

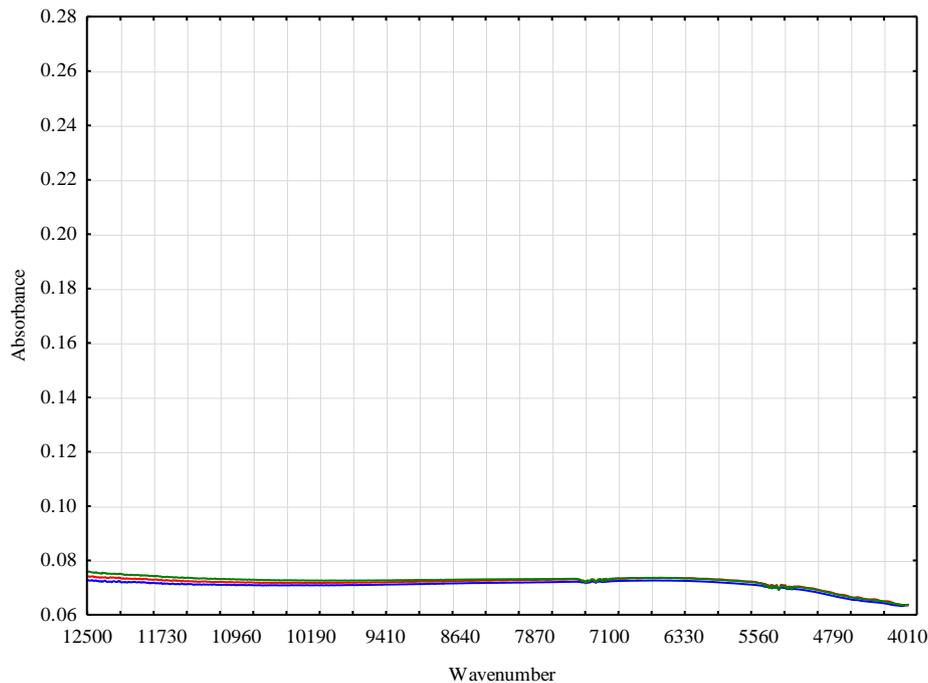


Figure 3. Absorption spectra of three QX quartz type cells showing an absence of background absorbance due to –OH bond absorption.

Development of updated composite MJ NIRS prediction equations

All analyte NIRS prediction equations prior to the 2012 season were developed using the OS flow-through cell type. With the adoption of the QX quartz flow-through cells a new set of MJ analyte prediction equations based on the new cell type were required to be developed. As considerable resources (conventional sample analysis, NIRS scanning and subsequent prediction equation development) had been used in the development of the original prediction equations over a five-year period, the possibility of transferring this information to the new set of equations was investigated. The most common method is conversion using spectral transfer methods. This procedure is routinely used in the pharmaceutical industry to convert spectra from one make of instrument to another. A spectra transfer method is developed to model the differences between the original and new spectra. During the spectral transfer process the differences are taken into consideration and the original spectra adjusted correspondingly. However, this procedure proved unsuccessful as it could not remove the background absorbances incorporated from the OS type transmission cells before transfer to the QX cell. This necessitated complete redevelopment of the prediction equations.

NIRS prediction equations are conventionally developed on one instrument in a central location. This requires the specific analyte analyses of the samples of interest (preferably following approved analysis methods) and suitable NIRS scans of the same samples using a single NIRS instrument, and is commonly referred to as a 'local prediction calibration'. The incorporation of variable measurement conditions such as different NIRS instruments or flow-through cells will improve the robustness of the calibration model and subsequent prediction equations (Swierenga *et al.*, 2000). This is commonly referred to as a 'global prediction calibration'. The initial NIRS prediction equations developed at SMRI were local prediction calibrations. The requirement for redevelopment of the predictions equations offered the opportunity to develop rugged, global prediction calibrations based on multiple NIRS instruments and QX transmission cells.

Thirteen mill laboratory MPA instruments were loaned to SMRI for a five-month period. A random weekly sampling schedule was developed which combined 15 instruments and 39 cells when scanning composite MJ samples. Replicate scans of composite MJ samples were made using all instrument/cell combinations. Weekly composite MJ samples were collected from the middle to the end of the 2012-2013 season (SASA week 27 to week 43) and from the beginning to the middle of the 2013-2014 season (SASA week 3 to week 23), scanned and analysed using Official and Accepted Methods of Analysis to determine the brix, pol sucrose, glucose, fructose and ash concentrations in the samples. The range of samples thus included beginning, middle and end of season sample variances. Samples from all South African sugar mills were included to encompass diffusers and milling tandems, geographic variation, rainfed and irrigated cane. Spectra of the composite MJ samples were recorded using a combination of 15 MPAs (13 mill and two SMRI instruments) and 38 QX flow-through transmission cells. A total of 276 composite MJ samples were scanned which resulted in a total combination of 24 559 spectra. A number of prediction models were developed for each analyte and the performances of the models were checked against a test set. Based on these results the best individual prediction models were validated against an independent sample set covering a complete season (Walford, 2014). The prediction equations were used for the parallel testing of the NIRS analysis method with the conventional methods for composite MJ brix, pol and sucrose for the 2014-2015 season.

Comparison of the conventional and NIRS analysis methods

The weekly pol and brix results reported by SMRI are not used directly for cane payment purposes but only as an audit check for the weekly total weighted hourly results reported by the CTS mill laboratories. The SMRI pol and sucrose values are used to calculate a pol:sucrose ratio which in turn is used to determine the quantity of sucrose (from the tandem pol values) in the cane payment calculations. It is therefore critical to determine whether the methods are equivalent, not only across a season, but also that there is no bias between tandems throughout the season and no bias between results reported between weeks. During the 2014-2015 crushing season, 594 weekly composite MJ samples from all South African tandems were analysed by conventional and NIRS methods at SMRI and the results used for the comparison of the methods. No samples were received or analysed for Weeks 14 and 15 during the industry strike.

Normal statistical analysis assumes that the data sets are normally distributed. In any set of analysis results there is the possibility that a particular result may be an observation that is an outlier. Outliers may have an appreciable effect on the mean and standard deviation of the data set. Random variation generates occasional extreme values by chance that could be perceived

as outliers, but these values are still part of a valid data set. It is important that any real outliers are noted and removed if necessary so as not to influence any conclusions that are to be drawn from the data set. As the weekly sample results naturally have a wide range of analyte values, it is difficult to apply an outlier test directly to the sets of results. However, if it is assumed that the methods give equivalent results, the differences between the results should be centred on zero. Outlier tests on the paired differences between conventional and NIRS results showed no outliers and all the data was used in the comparisons.

Overall comparison across the season

The results for the comparisons between the conventional and NIRS analyses at SMRI across the 2014-2015 season are shown in Table 1.

Table 1. Results of the comparison between conventional and near infrared spectroscopy analysis methods for brix, pol and sucrose across the season when analysed at the Sugar Milling Research Institute.

Analyte	Conventional			NIRS			Statistical	
	Valid N	Mean	Std. Dev.	Valid N	Mean	Std. Dev.	F _{calc}	t _{calc}
Brix	594	13.78	1.430	594	13.78	1.449	1.027	0.182
Pol	594	11.71	1.307	594	11.71	1.317	1.015	1.000
Sucrose	594	11.82	1.311	594	11.82	1.318	1.009	0.023

The F-test value F_{calc} is computed as a ratio of the square of the variances of the two methods with the larger variance used as the numerator. If the variances are the same, the minimum value will be one with the F-value increasing as the difference in the variances increases. As can be seen from Table 1, the F-values were very close to one, indicating that the two methods give equivalent precision for all three analytes. This is confirmed by comparing the standard deviations of the methods for each of the analytes, which are practically equivalent. For all three analytes F_{calc} was less than the $F_{crit(0.05)}$ of 1.145. Therefore, at the 95% confidence level there was no significant statistical difference between the precision of the two methods for brix, pol and sucrose across the season when analysed at SMRI. A paired t-test was used to compare the results from the two methods. For all three analytes t_{calc} is less than $t_{crit(0.05)}$ of 1.962 (Table 1). Therefore, at the 95% confidence level there was no significant statistical difference between the results produced by the two methods at SMRI for brix, pol and sucrose across the season when analysed at SMRI.

The differences between the conventional and the NIRS analysis results are plotted in Figures 4 to 6 (brix, pol and sucrose). The red lines on the graphs show the range within which 95% of all differences fall. This means that only one in 20 samples will have a difference greater than ± 0.08 units for brix, pol and sucrose. These differences are well within the currently accepted analysis method differences for replicate samples.

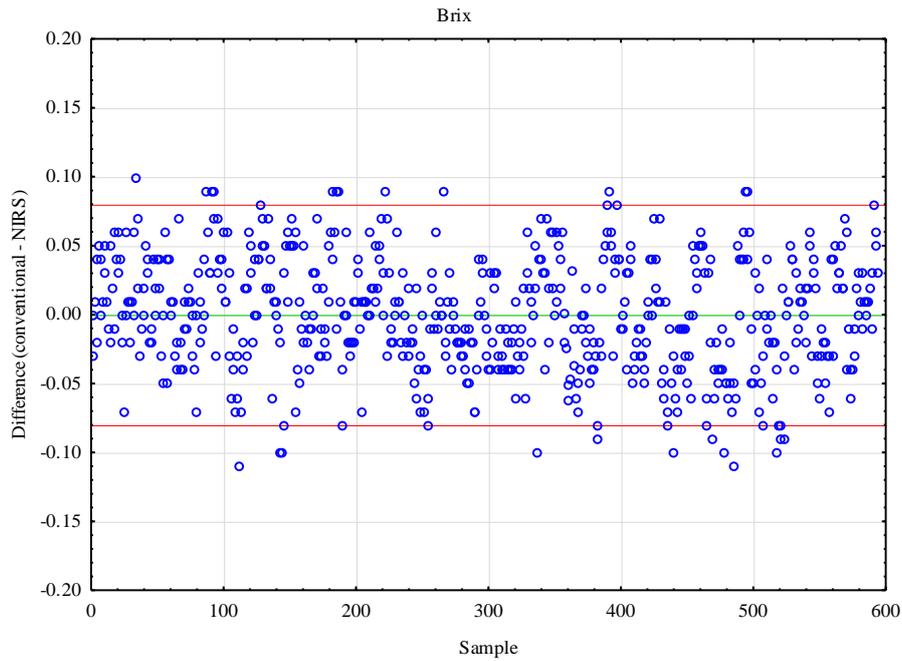


Figure 4. Differences between the brix results of the conventional analysis and near infrared spectroscopy (NIRS) analysis of composite mixed juice samples for all samples during the 2014-2015 season.

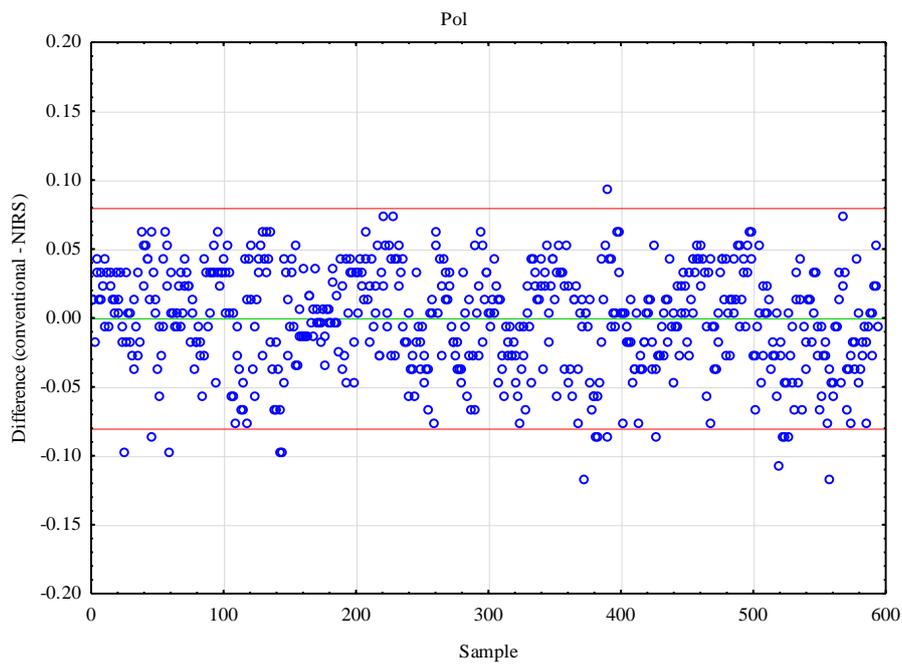


Figure 5. Differences between the pol results of the conventional analysis and near infrared spectroscopy (NIRS) analysis of composite mixed juice samples for all samples during the 2014-2015 season.

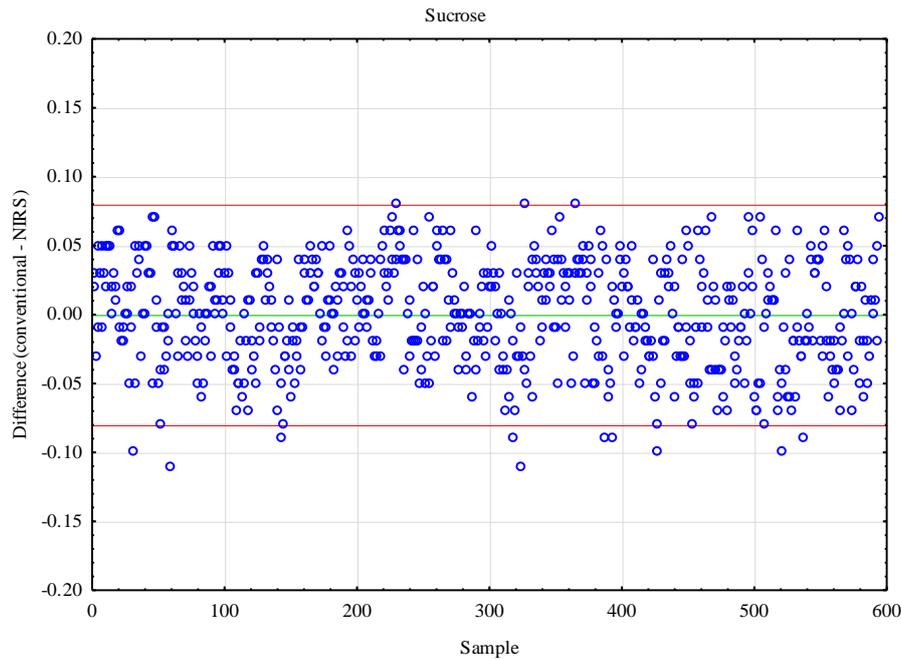


Figure 6. Differences between the sucrose results of the conventional analysis and near infrared spectroscopy (NIRS) analysis of composite mixed juice samples for all samples during the 2014-2015 season.

Comparison across mills

As stated above, the weekly pol:sucrose ratio results produced by SMRI for each tandem are used as an input into the cane payment system, and the brix and pol results as a check for the CTS mill laboratory results. It is therefore important to check that there is no statistically significant difference in the results (especially pol and sucrose) reported by conventional analysis compared to those by NIRS on a tandem-by-tandem basis if the NIRS results are to be used for the same purpose. The differences of the means are shown in Table 2. Note that the 18 tandems are randomly assigned a number between 101 and 118. On a mill-by-mill basis the maximum absolute difference between the conventional and NIRS analysis results is less than 0.02 for brix, pol and sucrose, and in the majority of cases 0.01 or less.

As in all analytical methods, the results given by the Official Methods for brix, pol and sucrose analysis have associated intermediate precision values. The intermediate precision value is a precision estimate obtained from replicate measurements of the same sample, by a different analyst, on different days, using different sets of equipment within the same laboratory (Ellison *et al.*, 2009). SMRI, as part of its ISO 17025 accreditation system, is required to determine the repeatability of all test methods on a regular basis. The intermediate precisions of the brix, pol and sucrose analysis methods have routinely been determined to be in the range of 0.07 to 0.09 at a confidence level of 95%. The absolute differences between the conventional and NIRS brix, pol and sucrose results for all tandems during the 2014-2015 season, grouped by week, are plotted in Figures 7, 8 and 9, respectively. The green line indicates the 0.08 currently accepted absolute difference intermediate precision value. The graphs show that for all three analytes, less than 5% of the difference in the results produced by the two methods was greater than 0.08 units. Therefore the graphs indicate that differences between the conventional analysis results and the NIRS analysis results are no worse than the existing accepted differences between replicate measurements made using conventional analysis methods.

Table 2. Results of the comparison between conventional and near infrared spectroscopy (NIRS) analysis methods for brix, pol and sucrose between tandems when analysed at the Sugar Milling Research Institute (SMRI).

Tandem	N	Brix			Pol			Sucrose		
		Conventional mean	NIRS mean	Diff	Conventional mean	NIRS mean	Diff	Conventional mean	NIRS mean	Diff
101	33	13.42	13.40	0.02	11.25	11.24	0.01	11.30	11.29	0.01
102	26	14.63	14.62	0.01	12.41	12.41	0.00	12.54	12.54	0.00
103	34	13.25	13.24	0.01	11.34	11.33	0.01	11.46	11.46	0.00
104	31	14.20	14.20	0.00	11.94	11.94	0.00	12.03	12.04	-0.01
105	30	14.13	14.12	0.01	11.83	11.82	0.01	11.91	11.92	-0.01
106	33	13.40	13.39	0.01	11.42	11.43	-0.01	11.50	11.49	0.01
107	33	13.09	13.08	0.01	11.14	11.13	0.01	11.24	11.23	0.01
108	36	14.35	14.36	-0.01	12.34	12.34	0.00	12.47	12.46	0.01
109	35	14.18	14.19	-0.01	12.18	12.18	0.00	12.31	12.31	0.00
110	36	13.95	13.97	-0.02	12.00	12.01	-0.01	12.12	12.13	-0.01
111	30	12.71	12.69	0.02	10.61	10.60	0.01	10.69	10.68	0.01
112	35	14.80	14.81	-0.01	12.52	12.54	-0.02	12.71	12.71	0.00
113	37	13.14	13.14	0.00	11.23	11.24	-0.01	11.33	11.34	-0.01
114	33	13.82	13.84	-0.02	11.75	11.74	0.01	11.86	11.86	0.00
115	34	13.94	13.96	-0.02	11.84	11.83	0.01	11.93	11.94	-0.01
116	36	14.87	14.89	-0.02	12.65	12.66	-0.01	12.80	12.82	-0.02
117	36	13.71	13.71	0.00	11.53	11.54	-0.01	11.70	11.71	-0.01
118	26	12.13	12.12	0.01	10.47	10.48	-0.01	10.51	10.50	0.01

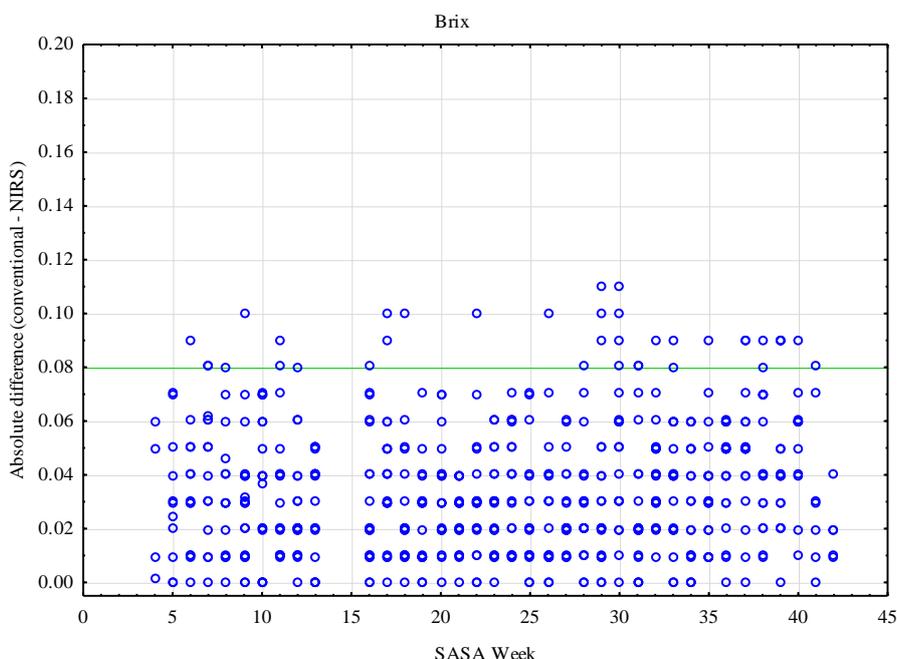


Figure 7. Absolute differences between the brix results of the conventional analysis and near infrared spectroscopy (NIRS) analysis of composite mixed juice samples for all samples during the 2014-2015 season grouped by week.

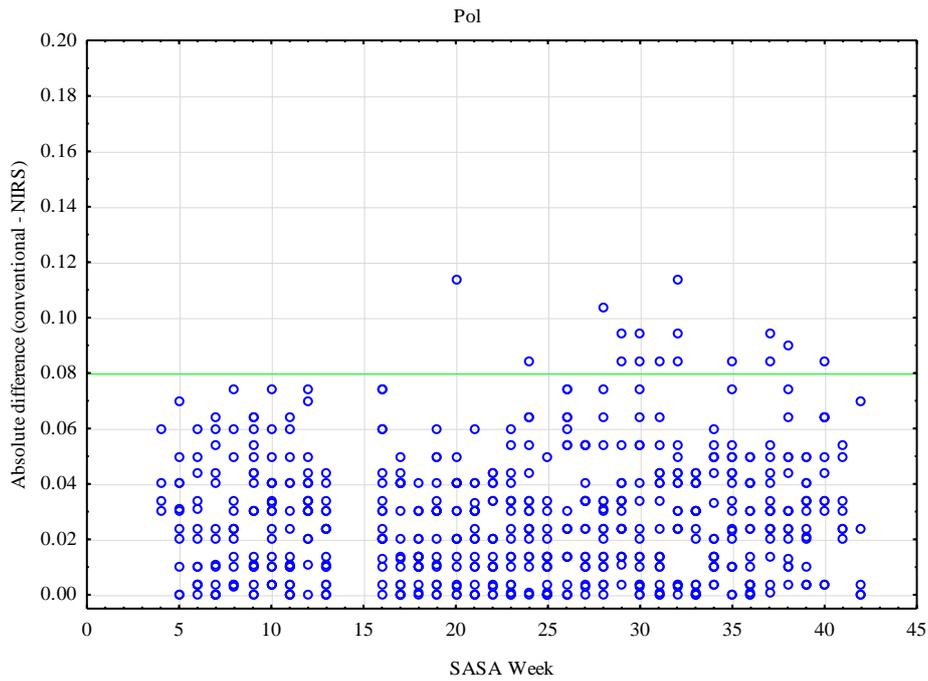


Figure 8. Absolute differences between the pol results of the conventional analysis and near infrared spectroscopy (NIRS) analysis of composite mixed juice samples for all samples during the 2014-2015 season grouped by week.

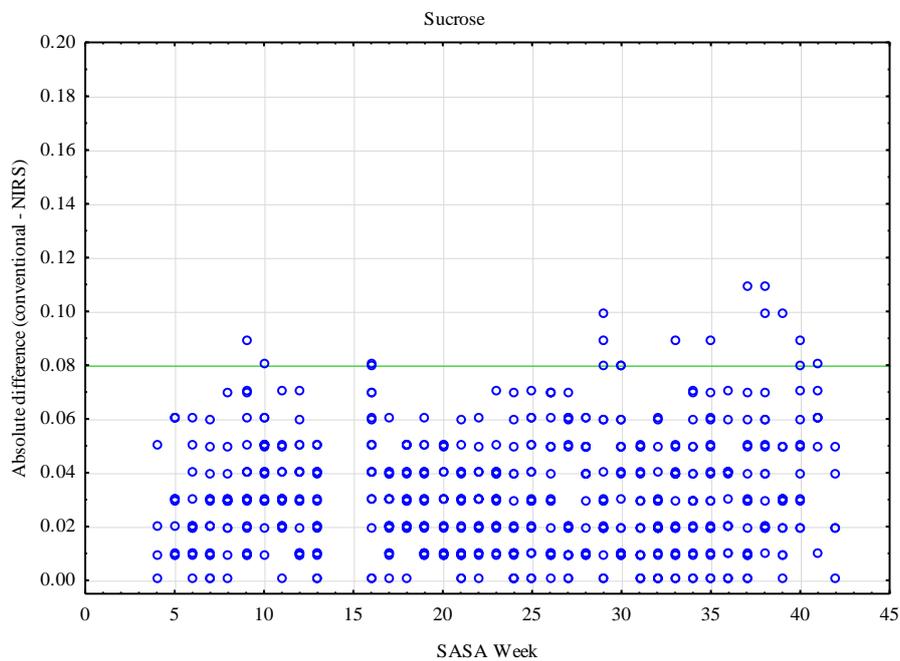


Figure 9. Absolute differences between the sucrose results of the conventional analysis and near infrared spectroscopy (NIRS) analysis of composite mixed juice samples for all samples during the 2014-2015 season grouped by week.

Effect of NIRS results on the calculation of the mill-based Recoverable Value

The choice between alternative methods of MJ analysis is only partially a technical question. Based on the results presented in this paper, the results predicted by the NIRS method are technically equivalent to those produced by the conventional methods. However, the results from these tests are used as inputs to the cane payment system and therefore the study required an investigation into the possible effect the use of NIRS results would have had on the system. The current system uses the pol:sucrose ratio provided by SMRI to convert the weighted tons pol determined at the mills into tons sucrose results. Cane payment is based on the Recoverable Value (RV) formula which includes the use of tons brix and tons sucrose. To investigate the effect the use of NIRS results may have on RV%, for the 2014-2015 season, the current RV values based on CTS pol (converted to sucrose using the conventional pol:sucrose ratio) were compared to RV values based on NIRS pol:sucrose ratio.

The results of this comparison are summarised in Table 3. Based on these results it appears that there is little difference between RV determined using the SMRI conventional results and SMRI NIRS results. This is to be expected as the differences between the conventional and NIRS results are very small, as shown in the earlier part of this paper. The effect of the calculated MJ mass balance RV on the mill RV% was calculated and is also shown in Table 3. Note that the 14 mills are randomly assigned a number between 101 and 114. The RV% differences between the conventional and NIRS based calculations were very small, typically between -0.03% and 0.04%, whilst one mill recorded a difference of 0.09%. It would be expected that due to the randomness of the differences between the conventional and NIRS pol:sucrose ratios, the differences recorded here for each mill would vary from season to season. The total RV% difference across the 2014-2015 season for all mills was 0.00%.

Table 3. Results of the comparison on a mill basis between conventional tons mixed juice (MJ) recoverable value (RV) calculated using the conventional pol:sucrose ratio and the near infrared spectroscopy (NIRS) pol:sucrose ratio for the 2014-2015 season.

Mill	Tons		Difference		Cane crushed (tons)	RV% (conv MJ RV)*	RV% (NIRS MJ RV)	Difference (%)
	MJ mass balance RV (CTS conv pol and conv pol:sucrose)*	MJ mass balance RV (CTS conv pol and NIRS pol:sucrose)*	Conventional - NIRS (tons)	Conventional - NIRS (%)				
101	147 014.0	146 924.6	89.4	0.06%	1 185 297	12.40%	12.40%	0.01
102	134 658.5	134 552.2	106.3	0.08%	1 105 079	12.19%	12.18%	0.01
103	228 223.5	228 617.0	-393.5	-0.17%	1 877 160	12.16%	12.18%	-0.02
104	128 859.8	128 820.0	39.8	0.03%	1 003 229	12.84%	12.84%	0.00
105	110 745.4	111 553.3	-807.9	-0.73%	860 545	12.87%	12.96%	-0.09
106	308 953.8	308 459.3	494.5	0.16%	2 330 859	13.25%	13.23%	0.02
107	159 504.0	159 502.1	1.9	0.00%	1 257 948	12.68%	12.68%	0.00
108	224 810.4	224 907.3	-97.0	-0.04%	1 655 413	13.58%	13.59%	-0.01
109	107 230.1	107 227.2	3.0	0.00%	849 936	12.62%	12.62%	0.00
110	150 663.1	150 666.7	-3.5	0.00%	1 124 488	13.40%	13.40%	0.00
111	225 813.2	226 332.7	-519.5	-0.23%	1 755 129	12.87%	12.90%	-0.03
112	99 312.4	99 096.9	215.5	0.22%	711 983	13.95%	13.92%	0.03
113	91 758.1	91 727.7	30.4	0.03%	712 256	12.88%	12.88%	0.00
114	172 911.7	172 333.9	577.7	0.33%	1 326 215	13.04%	12.99%	0.04
Total	2 290 457.9	2 290 720.9	-263.0	-0.01%	17 755 537	12.90%	12.90%	0.00

* conv = conventional

Conclusion

This study has shown that based on the results of the analysis of weekly composite MJ samples from the 2014-2015 season, the NIRS analysis method gave brix, pol and sucrose results that were statistically equivalent to the Official Methods. The use of the NIRS results for the calculation of RV% showed that there was overall no difference for the season. This suggests that in practice the conventional analysis methods for analysis of weekly composite MJ samples could be replaced by the NIRS MJ method.

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REFERENCES

- Ellison SLR, Barwick VJ and Farrant TJD (2009). *Practical Statistics for the Analytical Scientist. A Bench Guide*. 2nd Edition. Royal Society of Chemistry.
- Naidoo Y and Walford SN (2013). The development of juice NIRS predictions for minor constituents. *Proc Int Soc Sug Cane Technol* 28: 8 pp.
- Schäffler KJ (2000). Trials and tribulations of implementing NIRS for raw sugar factory liquors. *Proc S Afr Sug Technol Ass* 74: 361-368.
- Schäffler KJ (2001). Automated routine analysis of quality parameters in sugarcane juices and molasses by NIR. *Proc S Afr Sug Technol Ass* 75: 318-321.
- Schäffler KJ and de Gaye MTD (1997). Rapid near infra-red estimation of multi-components in mixed juice and final molasses: The possibility of day-to-day control of raw sugar factories using NIR. *Proc S Afr Sug Technol Ass* 71: 153-160.
- Schäffler KJ, Dunsmore AN and Meyer JH (1993). Rapid analysis of sugar products by near infra red spectroscopy. *Proc S Afr Sug Technol Ass* 67: 222-229.
- Simpson R and Naidoo Y (2010). Using near infra red spectroscopy for rapid quantification of intermediate sugar factory products. *Proc S Afr Sug Technol Ass* 83: 382-391.
- Simpson R and Naidoo Y (2011). Can near infrared spectroscopy (NIRS) be used to predict lactic acid, acetic acid and ethanol in sugarcane mixed juice? *Proc S Afr Sug Technol Ass* 84: 432-438.
- Simpson R and Oxley J (2008). Routine analyses of molasses and mixed juice by NIR spectroscopy. *Proc S Afr Sug Technol Ass* 81: 245-275.
- Swierenga H, Wülfert F, de Noord OE, de Weijer AP, Smilde AK and Buydens LMC (2000). Development of robust calibration models in near infra-red spectrometric applications. *Analytica Chimica Acta* 411: 121-135.
- Walford SN (2014). Development, validation and on-site testing of NIR calibrations for the prediction of brix, pol and sucrose in MJ samples. Sugar Milling Research Institute Technical Report No. 2182, 32 pp.