

ON-LINE ANALYSIS OF QUALITY PARAMETERS IN CONSIGNMENTS OF SHREDDED CANE BY NEAR INFRARED SPECTROSCOPY (NIR)

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Abstract

In South Africa (SA), individual consignments of shredded cane are analysed by the direct analysis of cane method (DAC). Although this procedure is capable of producing excellent sampling and accurate analysis of consignments, it is labour intensive.

NIR has been used by several sugar industries for determining cane quality for cane payment purposes. Earlier work in SA indicated that *At-line* NIR could produce excellent estimates of cane quality and could possibly replace the current DAC analysis for payment.

To be economically viable and to gain the best advantages, NIR has to be used on-line for automated analysis. This paper describes the progress made during the past two seasons.

Introduction

The Cane Testing Service (CTS) in South Africa is responsible for analysing consignments of incoming cane to determine the distribution of financial proceeds between growers. Cane is analysed using the direct analysis of cane method (DAC). Details of electronic cane tracking and full-hatch sampling can be found in the laboratory manual (Anon, 1985). Cane quality parameters of individual consignments include pol, brix, moisture, fibre and ash. Although it is accepted that this procedure is capable of producing excellent sampling and accurate analysis of consignments, it is labour intensive.

Laboratory and *At-line* NIR has been proposed for shredded cane analysis by Brotherton and Berding (1995), Edey and Clarke (1996), Schäffler (1996) and Peterson and da Silva (1998). However, NIR is expensive and, in South Africa, laboratory NIR cannot provide significant additional benefits. NIR can only be justified if the procedure can save money; this in turn means that NIR needs to be applied On-line.

Researchers at the Bureau of Sugar Experiment Stations in Australia have led the way with On-line NIR. Their CAS system has been commercialised and is used routinely in several Australian and Fijian raw sugar factories (Staunton *et al.*, 1999; Watson *et al.*, 1999; Habib *et al.*, 2001).

The DAC sub-sampling method, used in SA, provides a unique application of NIR. This is due to:

- the efficiency of sampling growers' cane. This has been demonstrated by Buchanan and Brokensha (1974). A system for identification and monitoring of each consignment is already in place.
- analysis of the samples of these consignments is also routinely available.

- implementation of On-line NIR at the DAC station is much simpler than sampling and analysing cane on the main carrier.
- calibration development for NIR equations and subsequent validation is much easier as DAC samples are routinely sent to the laboratory for analysis. No additional laboratory analyses are required and all results are available from the CTS Sample Manager, a laboratory information management system (LIMS).

Initial testing of a NIRSystems Direct-Light process NIRS was carried out At-line in the Maidstone mill CTS laboratory. The results were similar to those published by others (Schäffler, 2001).

This paper describes the transition from At-line to On-line NIR.

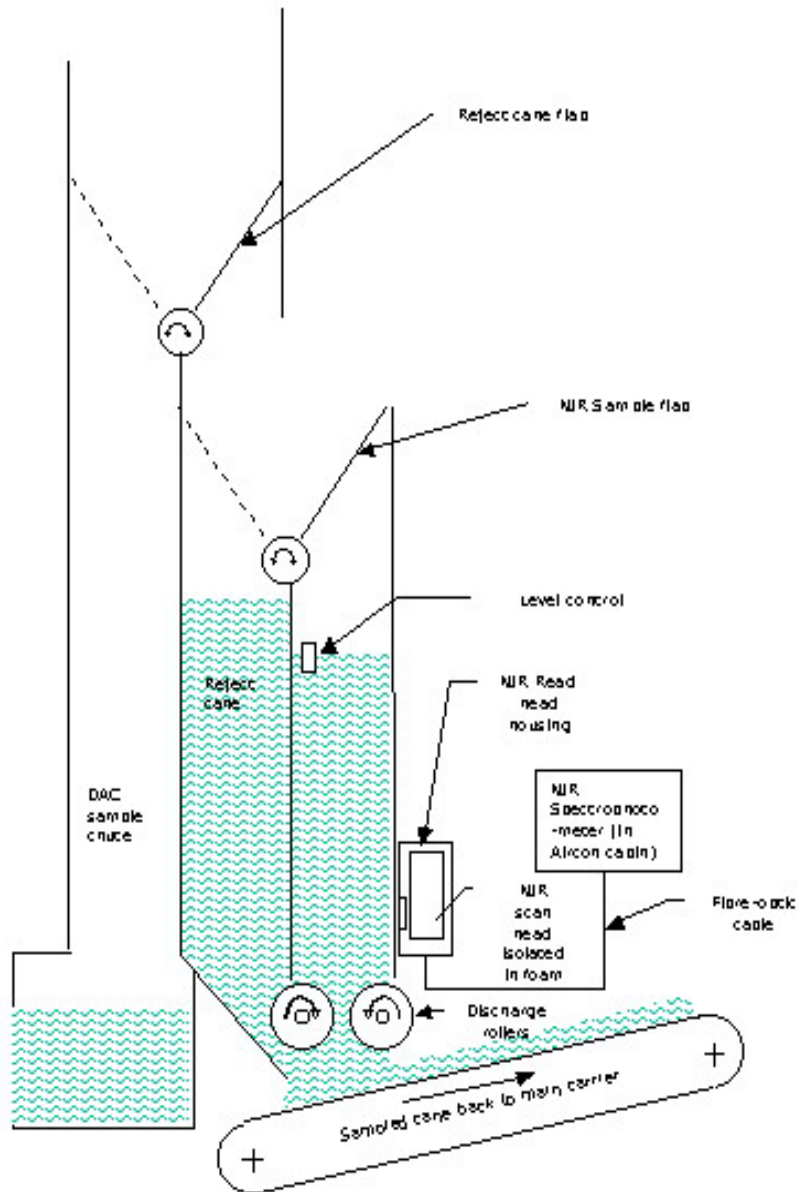


Figure 1. Schematic of On-line NIR scanning of shredded cane at Maidstone mill (see main text for details).

Experimental

DAC sampling station

Cane from the main carrier is sampled using a full-width hatch system as described by Buchanan and Brokensha (1974). The sub-sampling of cane is achieved by dropping it into one of two vertical chutes (sampler and reject chutes), the direction of the cane flow being controlled by a reciprocating flap. A timer and delay circuit controls the amount of cane received by the sampler chute; the overflow is despatched via the reject chute. A test-rig was developed and installed at one of the DAC sampling stations at Maidstone (MS) sugar factory. A third chute was added to sample the reject side of each consignment. This 'NIR' chute incorporates a set of metering rolls to ensure cane build-up in the chute. A glass window just above the rolls allows light from the NIR's scanhead to interact with the moving cane. A capacitive level sensor (Sensorik 50/10) has been installed about 1.5 metres above the rolls. This sensor activates a two-way pneumatic valve operating a third reciprocating hatch, ensuring that a constant head of cane is always in the chute. A schematic of the NIR sampling system is shown in Figure 1.

NIR sampling chute and discharge rolls

The NIR chute was fabricated from 3CR12, widening slightly from the inlet towards the exit end. Feed rollers with 'shark'-type teeth were fitted to the rolls (Figure 2). The rolls were driven by AC motors through separate gearboxes and controlled by a Xtravert PID controller.

Housing the NIR

A small fibreglass air-conditioned cabin (3x3x2 m) was installed next to the sampling station to house the Foss NIRSystems 6500 process spectrophotometer. The instrument was mounted on one of the walls using anti-vibration mountings allowing the scanhead, on a 2 m fibre-optic cable, to be mounted adjacent to the sampling chute. The cabin also contained a control PC, control logic for operating the rolls, and compressed air lines for running the NIR reference and cooling the scanhead (Figure 3).



Figure 2. Bottom of NIR chute at Maidstone DAC station, looking through the trapdoor at discharge rolls.



Figure 3. Inside the NIR cabin at Maidstone, showing the NIR Systems 6500 process spectrometer (mounted on rubber isolators). The PID controller for the discharge rolls is shown on the right.

Reducing the effects of factory vibrations on the NIR instrument

This was tackled in two ways:

Spectrophotometer

Air springs, from Firestone Industrial Products, were used to provide isolation from factory vibrations. A two-part steel frame was constructed with the four air springs mounted in each corner (see Figure 4). These were maintained at 250 kPa.



Figure 4. Two-part steel frame and air springs to reduce the effects of factory vibration on the NIR spectrometer.

Scanhead.

The head was housed in a fibreglass box and was surrounded by soft foam to reduce the effects of factory vibrations. A special mounting bracket ensured that, after routine maintenance, refitting was reproducible (see Figure 5). The unit was sealed against the chute, using anti-vibration foam, and positive air pressure was applied to prevent dirt or moisture ingress (Figure 6). As an additional safety precaution, a metal screen surrounded the box.



Figure 5. ‘Trapdoor’ in bottom of NIR chute with glass scanning window and mounting sill and bracket for scanhead box.



Figure 6. NIR readhead mounted in a fibreglass box bolted to the hinged window attached to the NIR chute. The window can easily be opened to clean the window or to clear bridged cane.

Setting up the Dac Sampling Station (Dacss) prior to NIR Scanning

The amount of cane moving through the station under normal sampling conditions is insufficient for On-line NIR scanning; higher throughputs are necessary. Prior to scanning, the NIR tester alters the timers, increasing the frequency of the main hatch opening and sending more cane to the station. In addition, the ratio of cane sent to the NIR chute relative to the manual sampling chute is also increased.

Collection of spectra

WINSCAN v1.04 from Foss NIRSystems/Tecatur was used for all NIR scanning. Although the NIR was On-line, scanning was done manually. A bell in the NIR cabin indicated that a cane consignment had arrived at the DAC sampling station. A monitor in the cabin graphically indicated tracker number and the progress of each consignment. The consignment number was manually entered into WINSCAN and after a lag period of two minutes scanning took place (it takes approximately two minutes for the NIR chute to fill). A scan takes approximately one minute (50 seconds for the sample and 10 seconds for the reference). This process was repeated until the bell signalled the end of the consignment. If the final sub-sample was still being scanned when the consignment ended it was included for subsequent averaging as the sample (due to the lag-time) would still be in the NIR chute. On average, four scans were obtained per consignment.

Melding spectra and laboratory data

At the end of each day, the tester retrieved pol, brix, moisture, fibre and ash data from the CTS laboratory for each consignment and manually merged this data with the relevant spectra.

Uploading Spectral and Laboratory Data

A procedure for uploading spectra and CTS laboratory data to the SMRI was developed using modems and PCAnywhere software (v8.01).

Results and discussion

Initial Design Problems

The first attempts at On-line scanning of shredded cane were prone to a number of problems, including:

- *Spurious spectral peaks.* Safety glass was inadvertently used for the window in the NIR chute. NIR spectra of cane were atypical and contained additional peaks at 1684 and 2156 nm. Toughened glass ensured that spectral quality was not compromised.
- *Discharge rolls.* The long, thin 'fingers' that were initially welded to the discharge rolls resulted in frequent chokes. The design shown in Figure 2 ensured that the shredded cane ran continuously through the NIR system without any chokes. A further improvement included a modified outlet.
- *High spectral noise.* Initial tests without any vibration damping resulted in noise levels above those recommended by the instrument manufacturer. The installation of air-mounted isolators on the cabin, mounting the NIR spectrometer on rubber isolation mounts and enclosing the readhead in soft foam reduced noise to acceptable levels. Average diagnostics for a two-month period are given in Table 1. These results are within the instrument's nominal limits.
- *Level sensor.* This capacitive sensor is relatively sensitive and initially the chute would not fill as moisture on the sensor kept the hatch closed.

Table 1. Foss 6500 Direct Light NIR Diagnostics for August and September 2002. Wavelength range 880 to 2300 nm.

| Statistic | Response | Wavelength accuracy (nm) | Repeatability (uA units) |
|------------------|-----------------|---------------------------------|---------------------------------|
| Mean | 25938 | 0.16 | 70 |
| RSD | 1.8% | 5.8% | 9.6% |

Once these problems had been resolved the system ran, virtually trouble-free, from July 2002 to the end of the season (mid-December 2002).

Procedure for scanning consignments

Maidstone has two diffusers. Throughput ranges from 170 to 220 tons cane per hour for each diffuser. Not all consignments are analysed and some four to five DAC analyses are carried out per hour for each diffuser. The size of the consignments varies from 8 to 25 tons. Using a scan time of one minute, consignments are scanned 2-8 times by the NIR, depending on consignment size. It is estimated that over 80% of each consignment is scanned. This together with the fact that during NIR scanning the amount of cane passing through the DAC station is at a maximum means that sampling errors are reduced when compared with the standard procedure.

Development of calibration equations

After the system had been optimised, collection of spectra began in July 2002 and continued until the end of November 2002. As the On-line procedure was only semi-automated, samples were collected during the day shift only. Spectra were scanned from 880 to 2300 nm. Sub-samples of each consignment were averaged (WINISI v1.04). The calibration set consisted of 650 consignments (from a total of 2225 scans) collected from 25 July to 15 October 2002. Spectral outliers were removed (Global Mahalanobis distance (GH) >3). The variation of analytes in the calibration set is shown in Table 2.

Table 2. Cane quality parameters in shredded cane: Summary of conventional laboratory data (N = 650 collected from July to October 2002).

| Analyte | Mean | Min | Max | SD |
|----------|------|------|------|-----|
| Pol | 12.6 | 9.6 | 16.1 | 1.0 |
| Brix | 14.8 | 12.3 | 18.2 | 0.9 |
| Moisture | 69.6 | 63.9 | 75.3 | 1.9 |
| Fibre | 15.6 | 11.6 | 21.4 | 1.8 |
| Ash | 1.2 | 0.3 | 7.0 | 0.7 |

Calibration equations were developed using the WINISI PLS process. The calibration statistics were compared with those from tests carried out in 2000 under 'ideal' conditions (off-line in an air-conditioned laboratory, cane packed manually into a sample compartment, no vibration problems) (Table 3).

Table 3. NIR calibration statistics for cane quality parameters in shredded cane: Comparing INLAB and On-line calibration results.

| Analyte | Laboratory trial 2000 | | On-line trial 2002 | |
|----------|-----------------------|------|--------------------|------|
| | SEC | RSQ | SEC | RSQ |
| Pol | 0.3 | 0.91 | 0.4 | 0.86 |
| Brix | 0.2 | 0.94 | 0.3 | 0.88 |
| Moisture | 0.8 | 0.86 | 0.7 | 0.85 |
| Fibre | 0.9 | 0.8 | 0.8 | 0.8 |
| Ash | 0.2 | 0.5 | 0.4 | 0.4 |

The calibration results from the two tests were remarkably similar, indicating that the transition strategy from laboratory to On-line was sound.

Validation of NIR equations

It is important when testing NIR equations that the calibration and validation sets be independent of each other. For this reason the test set consisted of samples scanned from 21 October to 29 November 2002. Figure 7 shows the distribution of the validation samples as a distance from the mean of the calibration score file. The mean GH value was 1.1, indicating that the prediction file was well within the limits of the calibration library. The predictions of the validation set using the equations summarised in Table 3 are shown in Table 4.

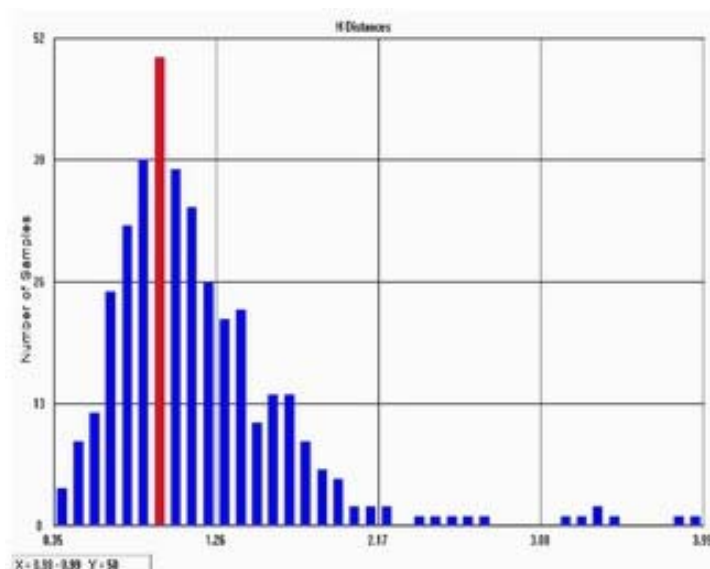


Figure 7. Histogram of Mahalanobis distances (GH) for the prediction set samples from the calibration mean, indicating that the two sets are similar.

Table 4. On-line prediction results for analytes in 371 consignments of shredded cane.

| Analyte | SEP | Bias | Slope | RSQ |
|----------|-----|------|-------|------|
| Pol | 0.5 | -0.3 | 0.84 | 0.72 |
| Brix | 0.4 | -0.1 | 0.90 | 0.74 |
| Moisture | 0.9 | 0.3 | 1.03 | 0.76 |
| Fibre | 0.9 | -0.1 | 0.92 | 0.67 |
| Ash | 0.6 | 0 | 1.23 | 0.26 |

The standard error of prediction (SEP) is a measure of the scatter between laboratory and NIR predictions. Bias is a measure of NIR accuracy; it is the mean difference between laboratory and NIR results.

The results in Table 4 show that:

- for on-line predictions, pol and brix results were very good, scatter and bias were less than half a unit. Buchanan and Brokensha (1974) established that DAC sampling and analysis produced errors of 0.4 units of pol.
- a SEP of less than one unit for moisture is most satisfactory. On average this is a relative error of less than 2%.
- A NIR SEP of 1 in 15 for fibre is therefore reasonable, since fibre % cane is derived from moisture and brix % cane. In practice, when using NIR, it is preferable to calculate fibre from the NIR estimates of brix and moisture.

Another way of evaluating the NIR prediction results is to compare the NIR precision (SEP) with the precision of the conventional laboratory methods. The precision of the DAC procedure was recently investigated by Schäffler and de Gaye (2002). The results are included in Table 5. It is obvious from these results that the NIR SEP are similar to the standard deviation (SD) of sub-sample replicates for all analytes, reinforcing the findings that On-line NIR predictions are suitable for routine cane analysis.

Table 5. Comparing the precision of NIR predictions with results obtained by conventional analysis (4 CTS laboratories, 274 samples).

| <i>Analyte</i> | <i>Laboratory SD</i> | <i>NIR SEP</i> |
|----------------|----------------------|----------------|
| Pol | 0.3 | 0.5 |
| Brix | 0.3 | 0.4 |
| Moisture | 1.1 | 0.9 |
| Fibre | 1.5 | 0.9 |
| Ash | 0.4 | 0.6 |

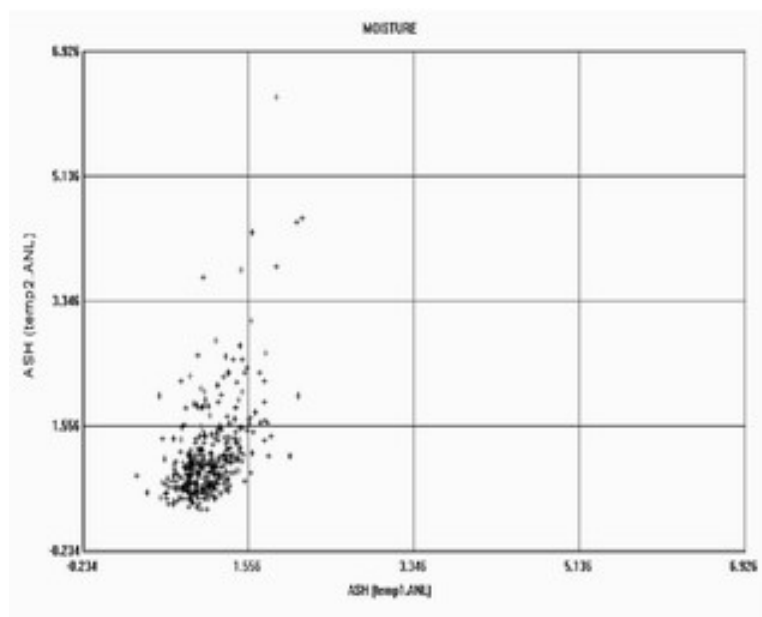
Ash Predictions

Total ash estimates are extremely poor (see Table 4 and Figure 8). These results are similar to those previously published (Schäffler, 2001). Schäffler *et al.* (1993) also found that ash correlations in mixed juice were disappointing (correlation coefficient squared (RSQ) = 0.6). In 1997, Schäffler and de Gaye found that, although NIR ash results for molasses (high ash content) were excellent, the same could not be said for mixed juice (RSQ = 0.55). This is in contrast to reports by other investigators. Madsen *et al.* (2002), using a Foss Direct Light NIR in a batch mode, found that a logarithmic function produced a reasonably strong correlation; however, the sand (dirt or mud) levels in Louisiana were extremely high (average ash = 5%, with levels peaking at 10 to 30%). In the current study, an equation was generated using log ash as a new analyte. The prediction was then converted using antilogs; the results did not show any improvements to those summarised in Table 4. Staunton *et al.* (1999) found reasonable ash predictions (SEP = 0.5, RSQ = 0.71 with a slope of 1.00). A slope of unity is in contrast to that found by Madsen *et al.* (2002) and this work.

The poor ash predictions in South African DAC samples are probably due to:

- low absorbance of ash, as inorganic substances have little absorbance in the NIR.
- low total ash levels in SA cane (0.5-1.5%). NIR is not a sensitive technique. In the prediction set, 90% of the samples had ash levels of less than 2%.
- the precision of the laboratory ash technique; a 50 gram cane sample is used. Brokensha and Mellet (1977) found that, under ideal conditions, ash precision was ± 0.4 units for an average ash content of 3.6%, this relates to a relative error of 11%. In routine CTS laboratories greater errors can be expected. As NIR calibrations are dependent on this laboratory procedure, the NIR estimates will include this scatter.

Ash predictions in SA DAC samples are more likely to be qualitative rather than quantitative. However, selection of samples for a future database needs to be considered carefully.



**Figure 8. Combustion ash analysis in shredded cane.
Comparing NIR estimate (x axis) with the laboratory data (y axis).**

Future work

Future work using the NIRSystems 6500 process analyser

On-line NIR predictions obtained during 2002 are considered to be sufficiently precise and free from bias to warrant further work automating the On-line process.

Future strategies include:

- updating WINSCAN 1.04 to ISISCAN v1.26, as this software supports automatic sampling and interfacing to an external process computer.
- installing a serial cable from the tracker cabin to the NIR cabin to provide the control computer with tracker numbers for each consignment.
- laying of fibre optic cables from the NIR cabin to the factory LIMS server. This will enable the LIMS system to receive NIR data and predictions.
- developing a control program integrating factory and NIR systems.
- testing the fully automated NIR system and analysing all DAC consignments from all three shifts.
- developing a procedure to cope with instrument/computer malfunctions.
- on-going comparison of laboratory and NIR results.

Future work using a Fourier-transform (FT) process NIR

One of the major concerns of long term NIR is the transfer of calibrations to future NIR instruments. Another concern is the possible inaccuracy of NIR instruments after servicing or repair. In 2001, a NIRS 5000 had its wavelength encoder rebuilt. GH values of molasses samples were vastly different after the re-build (GH >50) (Anon, 2002), and subsequent predictions were badly biased. Fortunately, molasses samples can be frozen or refrigerated and the NIR was re-standardised. After re-standardisation, GH values dropped to normal with excellent predictions, virtually free from bias. Berding (2002) recently gave two examples of dispersive instruments producing highly biased predictions after service and repair. The occurrence of these breakdowns has rendered calibrations, developed over several years, virtually useless, as reference materials for high moisture, unstable products are unavailable.

The cost of these breakdowns, in terms of rebuilding the databases, is substantial. For this reason it is imperative that research into NIR techniques be broadened to include instruments that guarantee greater wavelength accuracy and precision. Process FT NIR spectrometers are rapidly establishing a reputation for ruggedness and reliability. It is imperative that dual monitoring of the NIRSystems 6500 (established, proven with potential wavelength repeatability problems) and FT NIR (high speed scanning, impervious to factory vibrations, state of the art, reliable with high wavelength accuracy) be carried out on the same shredded cane samples. Negotiations for a trial system are at an advanced stage.

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