# ASSESSMENT OF SUGAR POLARISATION USING AN ELECTRICAL CONDUCTIVITY METHOD

By

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**Abstract**

A STUDY of the potential for using an electrical conductivity (EC) method for sugar polarisation (pol) assessment was undertaken during 2004. The method involves measuring EC in a 27$^{o}$ brix solution of the sugar being assessed, and using the result to calculate the purity. This in turn is used in conjunction with an infrared moisture analysis to calculate the pol. The results of this study were encouraging and the decision was taken to implement the method routinely for shift sugar analysis. The method has been in use for three seasons and has performed well. Over this period the standard error of prediction (SEP, calculated as the standard deviation of the difference between the results for conventional and EC pol methods) has been 0.093$^{o}$Z, with 95% of results being within 0.189$^{o}$Z. The best performance was for the 2006 season when the SEP was 0.077$^{o}$Z, with 95% of results being within 0.152$^{o}$Z. This compares favourably with conventional sugar pol analysis repeatability of 0.147$^{o}$Z. Maintaining a calibration equation that adequately represents the relationship between EC and sugar purity has been found to be integral to achieving good results. The precision of results returned using the EC method has been shown to be related to the precision of the analyst undertaking the analysis used for calibration. As such, having a competent analyst undertake this work is advantageous. The EC sugar pol assessment method offers advantages over the conventional method for routine shift analysis. It is simple, requires little analytical skill, experience or training to undertake, and has a high degree of repeatability. It is also significantly faster to complete than the conventional method. The adoption of this method in conjunction with an EC method for the assessment of pan product true purity has allowed a significant reduction in laboratory manning without a reduction in the analytical output. Both methods also have the advantage of allowing large numbers of samples to be analysed during trials with far less work than conventional methods.

## Introduction

An electrical conductivity (EC) method for the assessment of true purity in factory products has previously been described by Snoad (2005) and Snoad *et al*. (2007). In addition to the EC pan product analysis method, Mulgrave Mill has also been using an EC method for routine shift assessment of sugar pol. The implementation of EC analysis methods for pan product and sugar analysis has allowed laboratory manning to be reduced while maintaining laboratory output.

This paper describes the evaluation, implementation, and use of the EC sugar analysis method over three crushing seasons.

**Definitions**

For the purposes of this paper, the following definitions apply:

- EC refers to EC corrected for temperature. The coefficient of correction used was 2% per 1°C.
- Conventional sugar pol shall refer to analysis done by BSES Method 30, Polarisation—Determination in raw sugar using visual or automatic polarimeters. Sugar purity shall refer to the apparent purity of sugar calculated from the pol and the moisture.
- Standard error of prediction (SEP) is calculated as the standard deviation of the difference between results obtained with conventional and alternate analytical methods.
- Precision will refer to the closeness of the results of replicate analyses.
- Accuracy will refer to the closeness of results to the true value.

**Initial studies**

Evaluation of the potential for an EC method for sugar pol assessment began following a description by Broadfoot (2004, pers. comm.) of a technique used by Sugar Research Institute personnel for sugar analysis in fugal trial work (Broadfoot and Muddle 2001).

The method involved determining the EC of a 27° brix solution of sugar, and using this in conjunction with moisture to calculate the pol.

Initial evaluation involved determining the EC of 27° brix solutions using sugar from terminal delivery samples. Examination of the data gathered indicated the following.

- There was a closer relationship between the EC data and purity than there was with pol.
- While there was variation in the relationship between EC and purity across the crushing season and between brands of sugar, over shorter timeframes the variation was of a manageable degree.

A decision was made to implement the use of an EC sugar analysis method for the 2005 season.

**Basis of the method**

The relationship between EC and brix for process materials has previously been discussed (Snoad, 1996). A peak in EC occurs around 27° brix. Snoad (2005) described the use of this peak in the EC of process material solutions to assess true purity.

Sugar samples exhibit similar behaviour, and the same methodology applies to assessing sugar purity. The sugar pol can then be calculated using sugar purity and moisture as below.

$$pol = purity \left( \frac{100 - moisture}{100} \right)$$

**Method employed**

**Make a 27° brix solution of the sugar being analysed**

A container has 27g +/– 0.03 g of sugar weighed into it, and water added to make the weight up to 100 g +/– 0.05 g. The container is placed on an orbital stirrer and mixed for 15 minutes to dissolve the sugar.

**Determine the EC**

Once dissolved the conductivity is measured using a Mettler Toledo SevenEasy™ conductivity meter hooked to a computer using Mettler Toledo BalanceLink™ software. The conductivity results are read directly into a Microsoft Excel spreadsheet.

**Determine the sugar moisture**

Moisture determination is undertaken using an infrared (IR) balance. This allows a result to be returned in a relatively short timeframe. Issues relating to the accuracy and precision of IR moisture analysis will be discussed later.

**Calculate the sugar purity and pol**

The sugar purity is calculated from the EC using a predetermined calibration equation written into the Excel spreadsheet. The moisture is entered, and in conjunction with the purity is used to calculate the pol using the equation previously given.

**Method of calibration**

An automatic sampler is used to sample across 200 tonne batches of sugar. Each sample is taken to the lab, thoroughly mixed, and two subsamples packaged. One subsample is analysed using the EC method and IR moisture, before being returned to the fugals with the analytical results.

The other subsample is kept in the laboratory. Usually four of these are selected each day, and along with a daily composite analysed in duplicate for pol using the conventional method, moisture by IR and oven methods, and EC at $27^{o}$ brix. Moisture data are examined to ensure that IR moisture results are in reasonably similar to oven moistures.

The IR moisture, conventional pol, and EC data are added to data already accumulated through the season, and the behaviour of the relationship between sugar purity and EC examined. This information is used in examining and deriving calibration equations.

**Potential influences on the accuracy of results**

Potential influences on the reliability of results derived using the EC method include:

- reliability of EC measurements;
- reliability of IR moisture measurements;
- validity of the calibration equation.

With analysis for the purposes of calibration usually undertaken in duplicate, much of the data can be used to examine the effects of these influences. The 2007 season saw analysis for calibration being undertaken by two relatively inexperienced analysts in their first year of performing these methods. As this is a situation which may become more common, 2007 information will primarily be discussed.

**Reliability of EC measurements**

Reliability of the EC measurements appears most likely to be influenced by errors in dilution of the sugar sample, variation in EC measurement, and calibration issues with the measurement equipment. These will be discussed in turn.

***Errors in dilution and EC measurement***

With EC peaking at around $27^{o}$ brix the shape of the curve representing the relationship between concentration and EC provides some protection against errors in dilution. As such a small error in dilution is unlikely to result in a significant error in the EC result.

Experience has shown that there is little variation between duplicate analyses. By way of example, an examination of the 581 data sets used during 2007 for calibration purposes shows a standard deviation of the difference between duplicates of 4.45µS/cm, which would equate to a variation in the sugar result of less than 0.01°Z. For this data the 95% repeatability was 0.026°Z. The same analysts completed 601 duplicate sets of sugar pols using the conventional method, returning a 95% repeatability of 0.151°Z. This compares to the accepted repeatability for sugar pols of 0.147°Z (BSES, 2001a). These data indicate that the dilution and measurement methods used return results with a far higher degree of repeatability than the conventional methods.

***Meter calibration issues***

The only issues with meter calibration occurred towards the beginning of the 2006 season. This was believed to be related to power supply fluctuations. The occurrence led to daily meter calibration checks becoming normal practice and it was extremely rare to find a significant calibration issue.

**Reliability of IR moisture measurements**

During 2007, 623 sets of duplicate moisture analyses were undertaken for calibration purposes using IR methods. Examination of results showed a 95% repeatability of 0.040%, identical to the accepted repeatability (BSES, 2001b). This compared to 614 sets undertaken by the oven method, which returned a repeatability of 0.047%.

Of the samples mentioned above, 603 were analysed using both IR and oven methods. Comparison of these showed that when the duplicates are averaged, 18.4% returned a result where the IR analyses were higher, and 8.0% where the analyses were identical. As such, 73.6% of oven moisture results were higher than the IR moisture. The mean difference between IR and oven methods was 0.021%, with the median difference 0.015%.

If for the purposes of this examination the oven moisture is assumed to be accurate, then on average the error from using IR moisture in the calculation of sugar purity would have been around 0.02%. However this is compensated by the use of IR moisture in deriving the calibration equation. As such, the error in moisture results from using IR moisture should not affect the calculated pol result unless for some reason there is a significant variation between the two methods.

**Validity of the calibration equation**

By far the greatest influence on the accuracy of the EC pol analysis method is the calibration equation. While based on data from previous analysis, there is a certain amount of subjective judgement with respect to what data will be included in the derivation of the equation. In addition, judgements are made based upon what has been analysed previously, which may not reflect adequately the circumstances of the time. Variations in the relationship between sugar purity and EC do occur throughout the season, and result in errors in calculated sugar pol assessment until the relationship can be redefined.

**Experiences in using the method**

The method has been used over three seasons, and some experiences in using it are described below.

**2005 season**

The 2005 season was the first in which the method was used for routine on shift assessment of sugar pol. Figure 1 shows the relationship between EC and sugar purity for 360 samples analysed in duplicate for the purposes of calibration across the 2005 season.

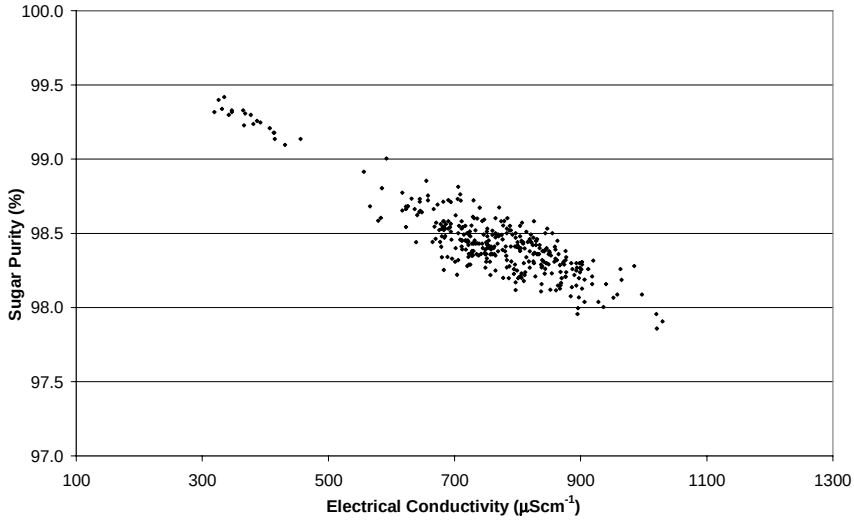


Fig. 1—Relationship between EC and sugar purity for the 2005 season.

Some spread can be seen in the data, which is the result of normal analytical variation and shifts in the relationship between EC and sugar purity. The analyses represented were undertaken by a competent analyst with a number of years sugar industry experience.

Shifts in the relationship between EC and pan product true purity have previously been noted by Snoad (2005) and Snoad *et al.* (2007). The same variation is also seen in the relationship between EC and sugar purity, although the relationship with sugar tends to be less stable than that found with pan products. As a result a number of changes are required through the season in order to keep the calibration equation representative of the EC/sugar purity relationship. In 2005, 21 different calibration equations were used.

Figure 2 shows the conventional pol results against the calculated results for the 360 samples used for calibration.

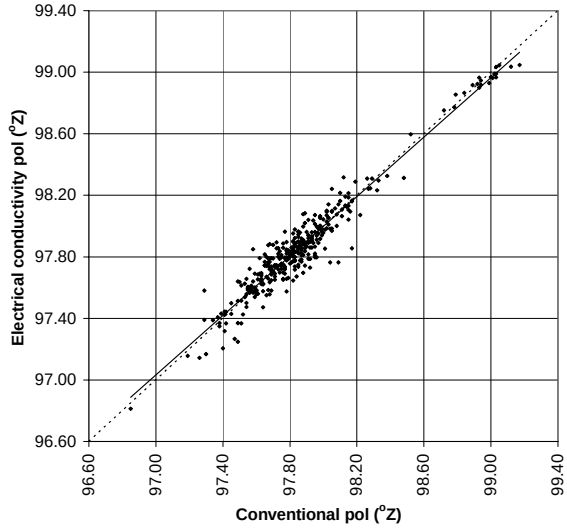


Fig. 2—Conventional pol results against pol calculated using EC method, 2005 season.

The calculated results are derived using the calibration equation in use at the time of analysis.

As such the results indicate how the method was actually performing in operation. Analysis of data indicated that when duplicates were averaged, and comparison made between the two methods, a SEP of 0.086°Z was returned, with 95% of calculated sugar pol results being within 0.177°Z of the conventional lab method.

**2006 season**

Figure 3 shows the 346 samples analysed for calibration during the 2006 season.

Some data generated early in the season that may have been affected by previously mentioned calibration issues have been removed.

The vast majority of the analysis was undertaken by a highly experienced and competent analyst without any prior sugar industry experience. Of note is that most of the data presented was the result of single analysis, rather than the average of duplicates as in the case of data presented for the 2005 and 2007 seasons.

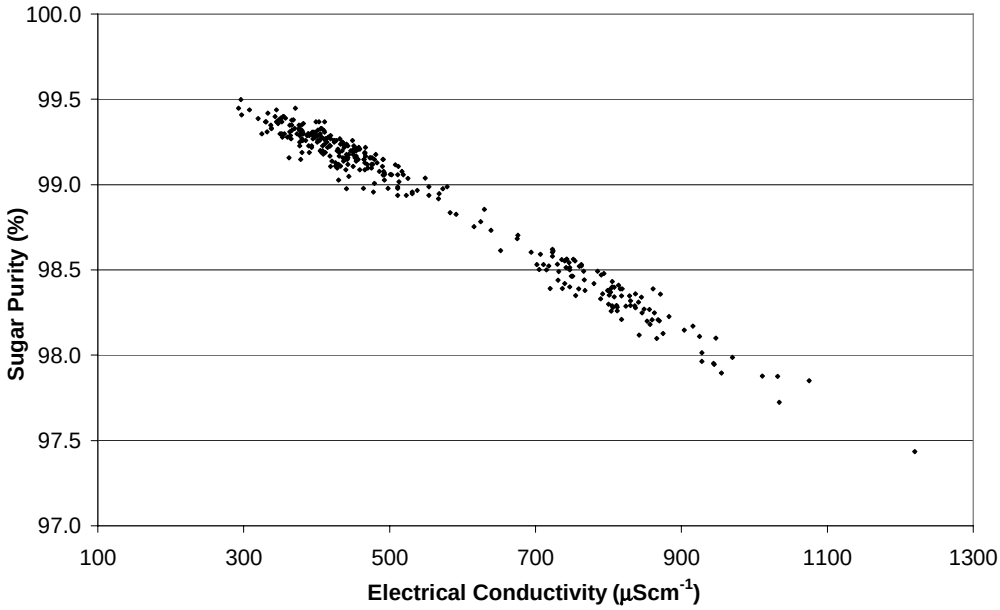


Fig. 3—Relationship between EC and sugar purity for the 2006 season.

While the data is more closely grouped than that from 2005, the spread was sufficient to require 12 different calibration equations to be used over the period represented.

Figure 4 shows the conventional pol results against the calculated results for the samples used in calibration. Analysis of data returned a SEP of 0.077°Z, with 95% of calculated sugar pol results being within 0.152°Z of the conventional lab method.

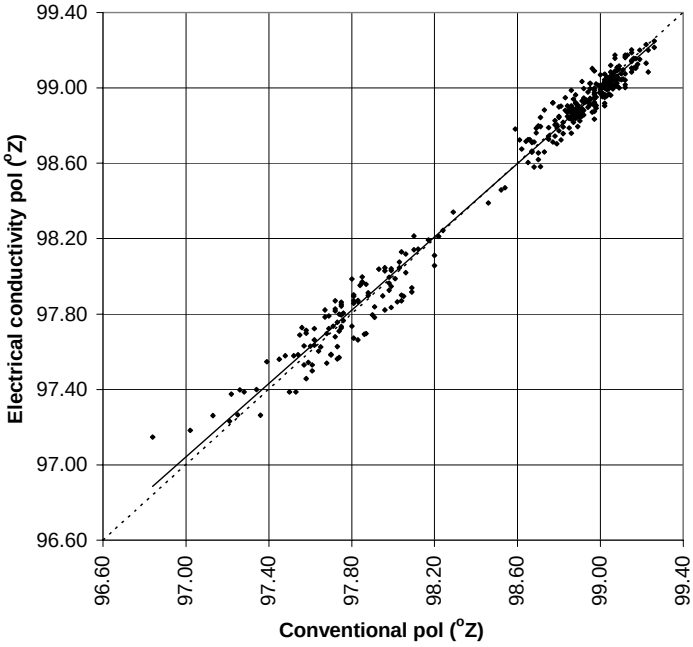


Fig. 4—Conventional pol results against pol calculated using EC method, 2006 season.

**2007 season**

During the 2007 season 547 samples were analysed in duplicate for calibration purposes and the data are shown in Figure 5. The vast majority of these were done by two analysts with limited experience.

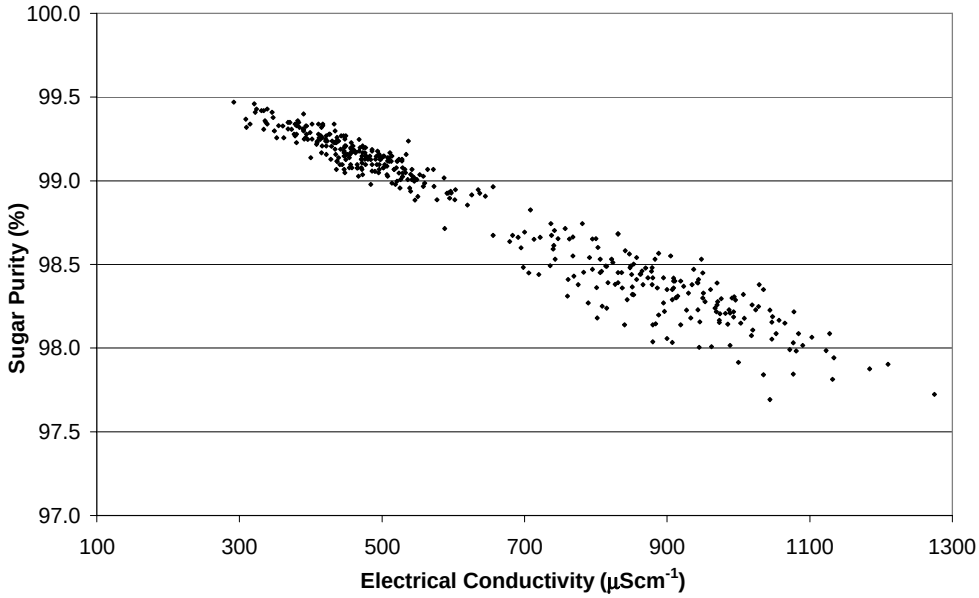


Fig. 5—Relationship between EC and sugar purity for the 2007 season.

The spread of data in 2007 is partly influenced by the processing of additional syrup from another mill during the season. This material had significantly different EC characteristics to Mulgrave syrup, and resulted in problems both with sugar and pan product EC analysis until a system for metering the additional syrup into the process was established. The situation was further exacerbated by a number of brand changes in the first half of the season.

Seventeen different calibration equations were used in 2007. Figure 6 shows the conventional pol results against the calculated results for the samples used in calibration. Analysis of data indicated that when duplicates were averaged, and comparison made between the two methods, a SEP of 0.106°Z was returned, with 95% of calculated sugar pol results being within 0.223°Z of the conventional lab method.

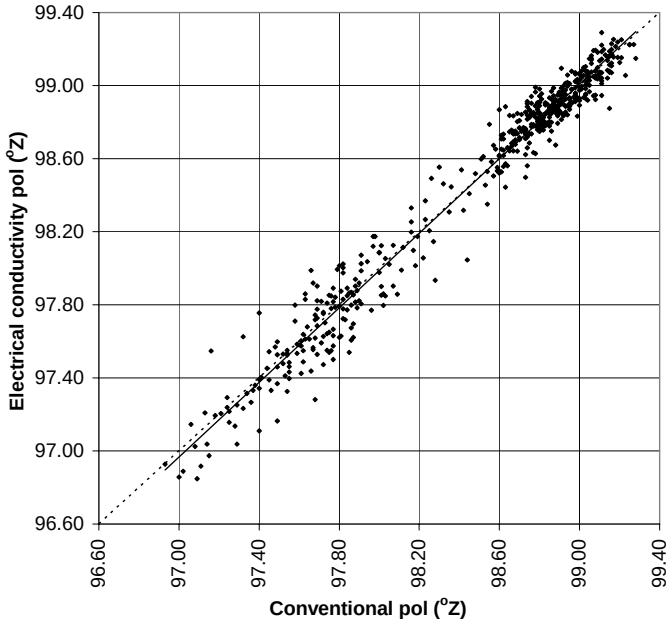


Fig. 6—Conventional pol results against pol calculated using EC method, 2007 season.

**Effect of brand change on calibration**

Calibration equations are linear, taking the form of:

$$y = -mx + c\,.$$

Changes in brand affect the calibration equation most notably through changes in the slope. For a given set of conditions, on average the slope tends to increase with an increase in pol, and vice versa.

**Discussion**

A summary of results across the three years of the method's use is given in Table 1. Calculated results used in deriving this data utilised the calibration equation current the time of analysis, without any adjustment for changes in the EC / sugar purity relationship that may have occurred prior. As such the results indicate how the method was actually performing in operation.

Because a comparison is being made between two different analytical methods, the conventional methodology for arriving at a 95% repeatability figure is invalid. Instead the difference between conventional and EC pol results for each sample have been calculated, and then the range within which 95% of the results fell determined. This data is given in the third column of Table 1.

**Table 1**—Summary of results from use of EC sugar assessment method.

| Year | SEP ($^oZ$) | Variation 95% of results within ($^oZ$) | Number of samples |
|---|---|---|---|
| 2005 | 0.086 | 0.177 | 360 |
| 2006 | 0.077 | 0.152 | 346 |
| 2007 | 0.106 | 0.223 | 547 |
| All years | 0.093 | 0.189 | 1249 |

In examining the data in Table 1, it should be remembered that analyses in 2005 and 2007 were determined from the average of duplicate analysis. As such the figures doe not necessarily represent what would be found if single analyses by each method had been compared.

The best performance of the EC method occurred in 2006, and the poorest in 2007. This correlates directly with the analytical experience and skill levels of the analysts. While the performance of the method in 2007 was no doubt influenced by the impacts of imported syrup, the effect of having inexperienced analysts generating data used for calibration should not be underestimated. More precise analysis means less chance of variation between the two methods for three main reasons.

Firstly, the EC assessment method has been shown to be highly repeatable with little variation between duplicate analyses. Therefore normal analytical variation in the conventional method is a more significant factor in the variation between results generated using the different methods. Assuming that the calibration equation in use is accurate, a higher level of precision in conventional analysis will keep the results of the two methods closer together and make performance of the EC method look better.

Secondly, more precise analysis means that better data is available for use in deriving calibration equations. Having calibration equations that accurately reflect the relationship between EC and sugar purity is a key element in getting the EC method to produce accurate results.

Thirdly, precise analysis makes it easier to define when a shift in the EC / sugar purity relationship has occurred, and allows more rapid and accurate derivation of a new calibration equation.

Analysis of sugar by the EC method described in this paper takes around 25 minutes. It is estimated that the average time between receipt of a sample and return of a result to the fugal station is approximately 45 minutes. This is approximately one hour faster than estimates of the average time previously required using conventional sugar pol analysis and an additional analyst on each shift.

Overall the performance of the EC sugar analysis method has been considered good. It is easy to undertake, requires minimal training, and returns highly repeatable results. This is of particular importance in circumstances where finding appropriately skilled or experienced personnel to undertake laboratory work on shift is becoming problematic. The adoption of this method in conjunction with an EC method for the assessment of pan product

true purity has allowed a significant reduction in laboratory manning without a reduction in the analytical output.

## Conclusion

A relatively simple and rapid method of determining the polarisation of sugar using EC and IR moisture has been described. It requires minimal analytical skill and training, and has a high degree of repeatability. The most important element in maintaining the accuracy of the method is ensuring good quality analysis for calibration purposes.

Overall the performance of the method is considered good, and its adoption in conjunction with a similar method for purity analysis of pan products has allowed reductions in laboratory manning without a reduction in analytical output.

## Acknowledgements

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## REFERENCES

Broadfoot R, Muddle CP (2001) Studies to improve the purging and washing efficiency of continuous high grade fugals. SRI Technical Report.

BSES (2001a). Laboratory Manual for Queensland Sugar Mills, Volume 2. Method 30, Polarisation—Determination in raw sugar using visual or automatic polarimeters. (BSES, Brisbane).

BSES (2001b). Laboratory Manual for Queensland Sugar Mills, Volume 2. Method 31, Moisture—Determination in raw sugar by drying at atmospheric pressure. (BSES, Brisbane).

Snoad JD **(**1996) Further modifications to the Mulgrave Mill evaporator station for continuous crushing. *Proceedings of the Australian Society of Sugar Cane Technologists* **18**, 361–367.

Snoad JD (2005) Investigation of rapid methods for the assessment of true purity and dry substance in factory product streams. *Proceedings of the Australian Society of Sugar Cane Technologists* **27**, 367–377.

Snoad JD, Pope GM, Thomas NS (2007) Experiences with application of rapid methods for true purity and dry substance assessment in factory product streams. *Proceedings of the Australian Society of Sugar Cane Technologists* **28**, 460–471.