

ESTIMATING DRY SOLIDS AND TRUE PURITY FROM BRIX AND APPARENT PURITY

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Abstract

Brix and apparent purity are well known to be biased estimators of dry solids and true purity respectively, particularly on lower purity streams. Correlations which estimate dry solids from brix and apparent purity and estimate true purity also from brix and apparent purity are presented. The suitability of these correlations is evaluated in the light of available historical data.

Keywords: pol, brix, dry solids, sucrose, purity

Introduction

Perhaps the two most important measurements in the sugar manufacturing process are those to quantify the sucrose and total dissolved solids concentrations of the various process streams. There is a range of analytical techniques available for making these measurements which are well described in texts on analytical procedures for the sugar industry (e.g. Anon, 1985; Anon, 1991). Unfortunately when selecting the most appropriate technique, there is the conventional tradeoff between those techniques which are rapid and convenient (but less accurate) and those that are accurate (but demanding in terms of time, equipment and technique).

Within the South African sugar industry, both types of measurement (rapid and accurate) are used, depending on the particular circumstances.

The present convention for rapid and convenient measurements is:

- for dissolved solids concentration : brix as measured by refractometer
- for sucrose concentration : pol as measured by polarimeter.

These are most commonly presented as brix and apparent purity (being the ratio of pol to brix expressed as a percentage).

The present convention for accurate measurements is:

- for dissolved solids concentration : dry solids as measured by vacuum oven drying or Karl Fischer titration
- for sucrose concentration : sucrose as measured by high pressure gas liquid chromatography (HPGLC).

These are most commonly presented as dry solids and true purity (being the ratio of sucrose to dry solids expressed as a percentage).

These selections have not always been the preferred choice and may well change in the future. For example, in the past “spindle brix” - measured using a hydrometer - was used as a rapid measure of dissolved solids concentration. Near infra-red spectroscopy has recently been the subject of extensive research as a possible tool for rapid and convenient measurements of both dissolved solids and sucrose (Schaffler and De Gay, 1997).

Both pol and brix are accurate for pure sucrose solutions (since the measurements are calibrated against pure sucrose solutions) but are increasingly in error as the level of impurities increases. These errors are also more severe when the solids concentrations are high. The magnitude of the error depends on the nature of impurities which in turn depends on seasonal, climatic and geographic variations. The effect of the impurities can thus be expected to vary slowly at a given sugar factory, raising the possibility of devising a correction factor determined from detailed analyses carried out weekly for a particular factory which can then be used to correct the pol and brix data.

The need to estimate sucrose and dry solids from pol and brix

For day to day production control, pol and brix measurements are usually adequate. They provide a sufficient level of repeatability to monitor variability. Target values for critical aspects of the process (e.g. C-masseccite apparent purity) can also be set and monitored even though the measurements are known to be biased.

Pol and brix measurements are, in most instances, not suitable for performing accurate mass balances. It is for this reason that the South African sugar industry uses analyses of sucrose and dry solids in the cane payment system and for the measurement of overall factory performance (e.g. the estimation of undetermined loss). Accurate analyses are also important to be able to quantify the level of sucrose and impurities accurately when measuring or estimating the solubility of sucrose in impure solutions (e.g. the calculation of target purity).

For mass balances across evaporators, such as that devised by Hoekstra (1981), pol and brix measurements will normally be sufficiently accurate (the streams being relatively high in purity and low in solids concentration). However mass balances over pans (e.g. Hoekstra, 1984), mass balances over the complete boiling house (Rein and Reid, 1983) or complete factory steam balances (e.g. Rein and Hoekstra, 1994) all require measurements of sucrose and dry solids for meaningful calculations.

Often the only data that are available or practical are based on pol and brix measurements. In these instances it is necessary to make an estimate of the true sucrose and dry solids measurements, based on some estimate of the degree of bias, before useful mass balance calculations can be made.

Whilst it is clear that there is substantial bias in the measurements of pol and brix in low purity streams encountered in C-Pan boiling, it is not as clear whether the bias on the streams encountered in A-Pan boiling is substantial or not. During the early development of high grade continuous pans by Tongaat Hulett Sugar (Rein *et al.*, 1985), numerous samples of syrup, masseccite and molasses were analysed by both the approximate and accurate analytical procedures (pol, brix, sucrose and dry solids). This (unpublished) data showed that:

- within the range of variability, pol and sucrose values were equal (i.e. no offset)
- the dry solids results were slightly greater than the brix results (contrary to accepted behaviour).

Although this data indicated that pol and brix measurements were sufficiently accurate for mass balance calculations, the anomaly in the dry solids results was a concern. The anomaly was subsequently attributed to problems with the vacuum oven drying procedure, indicating that the assumption of minimal bias on the brix measurements could also not be trusted.

Published information on conversions between measurements

The issue of developing correlations to estimate one type of measurement from another has been addressed by a number of authors. Archibald and Smith (1975) developed correlations to estimate refractometric brix from the earlier analytical technique of spindle brix. Batterham *et al.* (1974) proposed the following correlation for estimating dry solids concentration from the brix output of an on-line refractometer:

$$DS = brix - 8.68 \cdot \left(1 - \frac{Pty}{100}\right) \quad (1)$$

Matthesius and Mellet (1976) proposed a correlation to estimate true purity from refractometer gravity purity (ie the ratio of sucrose to refractometer brix expressed as a percentage). Saska and Oubrahim (1989) subsequently used this relationship to estimate dry solids content from refractometric brix according to the relationship:

$$DS = \left(\frac{0.0093}{Suc} + \frac{1.013}{brix}\right)^{-1} \quad (2)$$

Maurandi *et al.* (1988) proposed a simple proportionality constant to relate water content by the Karl Fisher procedure to that estimated by refractometric brix.

Unfortunately none of these efforts apply directly to the present situation in the South African sugar industry.

Estimating dry solids concentration from brix

Hoekstra¹ suggested the correlation given by equation 3 for correcting brix readings to provide an estimate of dry solids content.

$$DS = brix \cdot (1 - 0.00066 \cdot (brix - pol)) \quad (3)$$

This equation was developed by analysing the results of other researchers. One set of data was collected by Durgueil as part of his study of C centrifugal performance and the rheology of low purity streams (Durgueil, 1987). A second set of data was collected by Sahadeo during an extensive project on the optimisation of C-centrifugal performance in the late 1980s - referred to in his paper on high capacity continuous centrifugals (Sahadeo, 1992). Hoekstra also included data on average C-molasses analyses for the entire South African sugar industry from the 1987 and 1988 seasons.

The equation has the correct format in that it predicts that dry solids is equal to brix for pure sucrose solutions (i.e. those for which $pol = brix$). The errors between the prediction of equation 3 and the original raw data (covering the low purity range) are shown in Figure 1. The errors are calculated as the predicted dry solids from equation 3 (termed DSp) less the measured dry solids concentration (termed DS). All of these dry solids concentrations are expressed as mass percent of total mass.

¹ Tongaat Hulett Sugar Ltd, Internal Reports

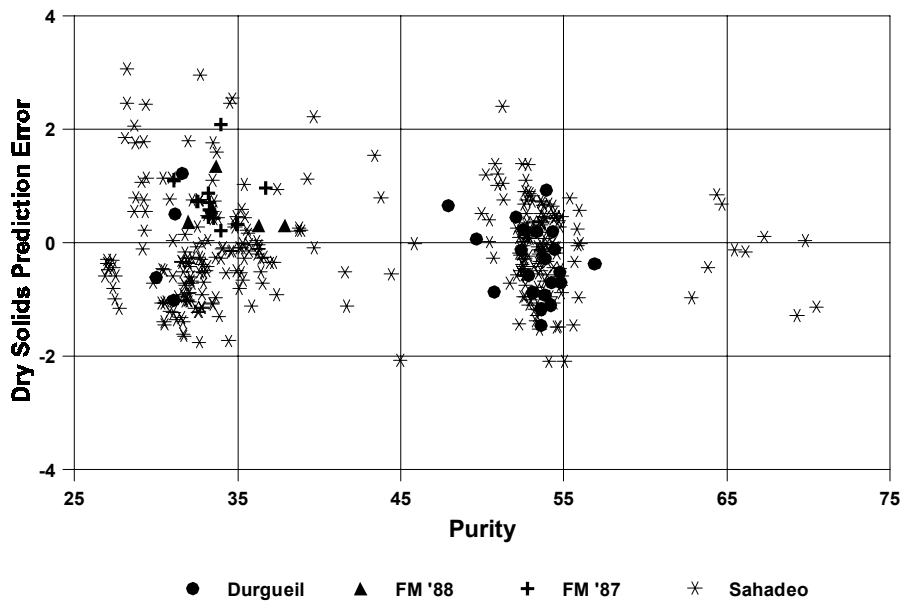


Figure 1. Errors in predicting dry solids from brix and pol measurements.

However the ability of the equation to accurately correct measurements in the range of A-massecuite boiling was not checked when it was formulated. Subsequently some data became available which could check the validity of equation 3 at higher purities. This data came from a sugar industry sponsored study undertaken at the Felixton factory to compare the magnitude of sucrose losses which occurred in the front end of the factory (from cane supply to syrup) with those that occurred in the back end of the factory (from syrup to raw sugar and final molasses). The particular data used were weekly average syrup samples for the 1990 season which were analysed for dry solids as well as brix and pol. The prediction error of equation 3 applied to these data is shown in Figure 2, where the scale of the y-axis, which shows the magnitude of the error, has been kept the same as in Figure 1. It is clear that the equation makes appropriate corrections in this high purity range and is thus suitable for estimating dry solids in high purity streams.

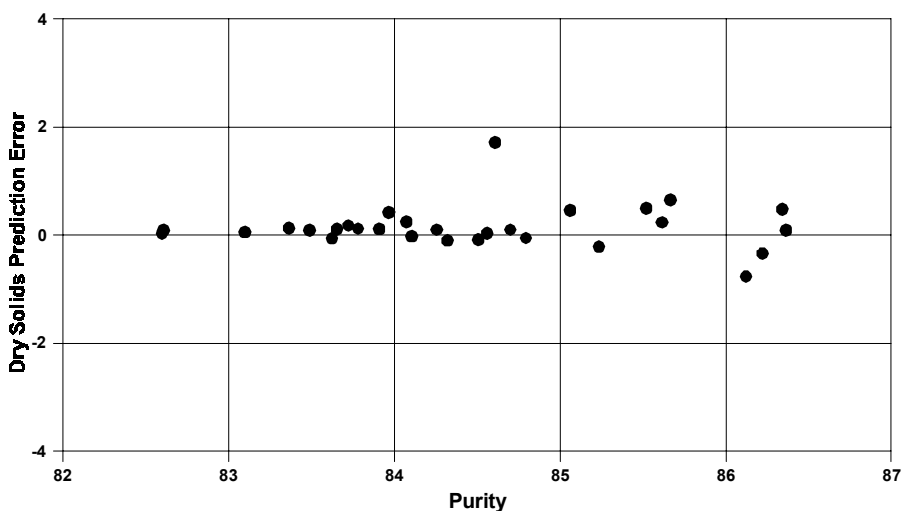


Figure 2. Errors in predicting dry solids in syrup from brix and pol measurements.

Equation 3 has been used by Hubbard and Love (1998) for performing mass balances over a high grade centrifuge. It has also been used for performing mass balances on continuous A-Pans at Felixton (Love, 2002).

Estimating sucrose from pol

Hoekstra¹ also proposed a correlation for correcting pol readings to obtain an estimate of the true sucrose content. This proposal was not based on any analysis of data but relied on the following assumptions:

1. The difference between pol and sucrose remains in a constant ratio to the amount of non-sucrose (i.e. the difference between dry solids and sucrose is in a constant ratio to the difference between dry solids and sucrose).
2. The nature of this relationship can be determined for the final molasses from a factory (available throughout the local industry on a weekly basis) and then applied to any other process stream in the factory. This is based on the assumption that this relationship will be specific to the cane quality from a particular supply area and that it will only vary slowly with time.
3. The impurities are known to change through the factory and the high purity products, towards the front end of the factory, will have impurities which are most different from the impurities in final molasses. Fortunately, these high purity streams need the smallest corrections and thus the importance of the difference in impurities is greatly reduced.

Mathematically, assumption 1 can be expressed in terms of a constant, K , as follows:

$$K = \frac{pol - Suc}{DS - Suc} \quad (4)$$

This relationship can be manipulated to give another constant, R , which is given by:

$$\begin{aligned} R &= \frac{Suc - pol}{DS - pol} \\ &= \frac{-K}{1 - K} \end{aligned} \quad (5)$$

This constant, R , can be included in the following identity:

$$\begin{aligned} Suc &= pol + (DS - pol) \cdot \frac{(Suc - pol)}{(DS - pol)} \\ &= pol + (DS - pol) \cdot R \end{aligned} \quad (6)$$

Using dry solids (DS) estimated from equation 3, and the value of R which can be calculated from the weekly data published for final molasses, it is possible to estimate the true sucrose value from pol data using equation 6.

The data from the industry sponsored survey mentioned above has been used to check the ability of this technique to estimate the sucrose content of a high purity streams (syrup and mixed juice). The degree of offset between the pol and sucrose measurements, which needs to be corrected, is shown in Figure 3, plotted against the value of the parameter R for the particular week. The offset is expressed as a percent of the pol measurement so that measurements on syrup and mixed juice are directly comparable.

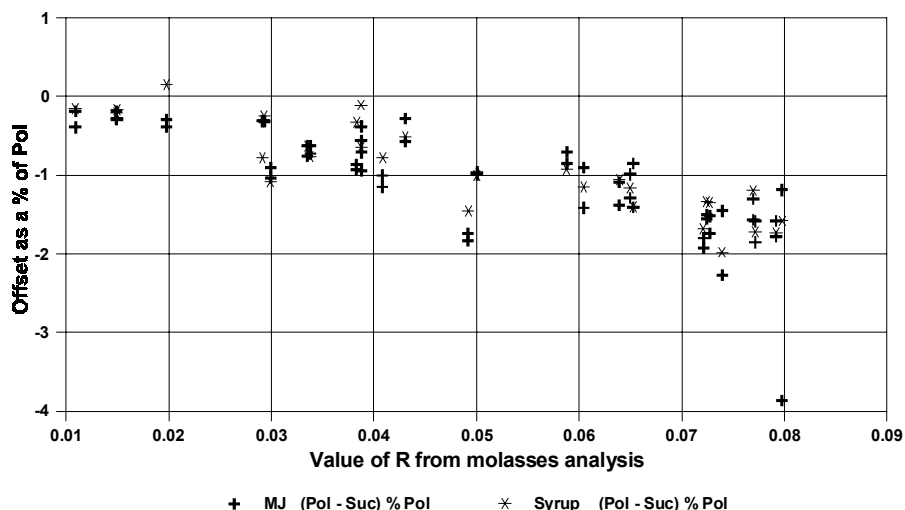


Figure 3. Offset between pol and sucrose readings expressed as a percentage of the pol measurement.

For comparison, the offset between the estimated sucrose value and the measured sucrose value is shown in Figure 4. The offset is also expressed as a percent of the pol measurement and the results are plotted on the same scale as used in Figure 3 for a clear indication of the benefit of the correction.

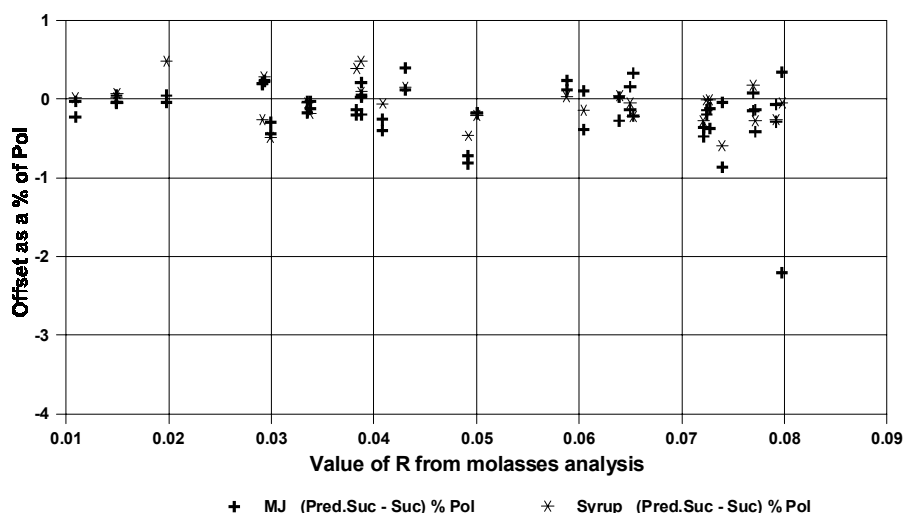


Figure 4. Offset between predicted and measured sucrose readings expressed as a percentage of the pol measurement.

These results demonstrate the ability of equations 3 and 6 to estimate the sucrose concentration from pol and brix measurements despite the off-set between pol and sucrose measurements varying from as low as about 0.1 units to as much as 2 units. The outlying point (at a value of R approximately equal to 0.08) appears to be a result of an error in transcribing the results).

Conclusions

Although based on a limited quantity of somewhat dated historical data, the correlations presented in this paper provide a convenient means of estimating sucrose and dry solids from pol and brix measurements. Although the correlations were developed for low purity streams, they have been shown to be applicable to high purity streams.

Ideally, correlations should be developed on a more comprehensive body of data. This data should be subjected to a full statistical analysis taking into account available data on the repeatability and accuracy of the individual analyses. This would provide more accurate correlations with quantitative measures of their level of statistical significance.

Acknowledgements

The mathematical correlations presented in this paper were originally developed by Rik Hoekstra whilst employed at Tongaat Hulett Sugar. They are characteristic of his work, showing careful thought in their formulation and having been selected so that they have the correct asymptotic form, i.e. they give the correct predictions in the extreme case of pure solutions. Raoul Lionnet provided the data on the analysis of mixed juice and syrup at Felixton. Thanks are also due to the management of Tongaat Hulett Sugar for providing access to information used in this paper and for allowing its publication.

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