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**BUREAU OF SUGAR EXPERIMENT STATIONS**

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# CALIBRATION OF A DENSITY METER

by


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## 1. INTRODUCTION

The Australian Sugar Milling Council (ASMC), as part of its continuing investigation into precision density meters, purchased an EXAC Model 8300EX 12A mass flow meter with density measuring capabilities. This instrument will be tested in four sugar mill laboratories in the 1992 crushing season comparing results with those obtained by the conventional cane payment method ie brix hydrometer. Prior to conducting these trials ASMC requested BSES assistance in calibrating the instrument against the standard brix hydrometer using pure refined sugar solution.

## 2. SPECIFIC REQUIREMENTS FOR INSTRUMENT CALIBRATION

(a) Perform necessary start-up checks and adjustments.

(b) Calibrate for zero flow.

(c) Configure the instrument:

- Determine repeatability on three standard solutions at approximately 15, 20 and 25°Bx.

(d) Provide a calibration curve using pure standard sugar solutions measuring brix with both hydrometer and pycnometer.

### 2.1 Start-up checks and adjustments

Considerable time was spent by the agents, Pacific Controls Pty Ltd, inputting various 'density factors' in an effort to obtain acceptable results. Each flow meter has two density constants $D_1$ and $D_2$ which are specific to that instrument. These constants determine the slope and offset of the density equation in the instrument's processor. Despite their efforts the instrument failed to give satisfactory results as shown later in this report. The agents have liaised continually with the manufacturers who are at a loss to explain these discrepancies.

### 2.2 Calibration for zero flow

The agents programmed the processor for zero flow. Samples were introduced to the instrument using a system similar to the 'continuous pol tube', ie a filter funnel mounted at the inlet end and a syphon-breaker at the outlet to keep the tube full of liquid.

### 2.3 Configuration of the instrument

Repeatability measurements were performed at approximately 15, 20 and 25°Bx using 100 mL of sample each time. The densities obtained at T°C were converted to degrees brix at 20°C. The results are shown in Table 1.

**Table 1**

**Repeatability of readings at different concentrations**

| **Brix @ 20°C** | | |
|---|---|---|
| 15.10 | 20.01 | 24.14 |
| 15.10 | 20.01 | 24.18 |
| 15.10 | 19.99 | 24.16 |
| 15.10 | 19.97 | 24.18 |
| 15.10 | 19.99 | 24.18 |
| 15.10 | 19.97 | 24.18 |
| 15.10 | 19.97 | 24.18 |
| 15.10 | 19.97 | 24.18 |
| 15.10 | 19.97 | 24.18 |
| 15.10 | 19.97 | 24.18 |
| 15.10 | 19.97 | 24.18 |
| 15.10 | 19.95 | 24.18 |
| 15.10 | 19.95 | 24.18 |
| 15.08 | 19.95 | 24.18 |
| 15.10 | 19.95 | 24.18 |
| 15.10 | 19.93 | 24.18 |
| 15.10 | 19.93 | 24.18 |
| 15.10 | 19.95 | 24.18 |
| 15.08 | 19.97 | 24.18 |
| n = 19 | n = 19 | n = 19 |
| $\bar{x}$ = 15.10 | $\bar{x}$ = 19.97 | $\bar{x}$ = 24.18 |
| S.D. = 0.006 | S.D. = 0.022 | S.D. = 0.010 |
| S.E. = 0.001 | S.E. = 0.005 | S.E. = 0.002 |
| Maximum difference between successive readings: | | |
| 0.02 | 0.02 | 0.04 |

Two trials were conducted to show the repeatability of reading when two samples of widely differing concentrations were introduced alternately into the instrument. Concentrations of 10 and 30°Bx and sample sizes of 100 and 200 mL were used. The results are shown in Tables 2 and 3.

**Table 2**

**Repeatability of alternate readings using 100 ml sample size**

| Brix @ 20°C | Brix @ 20°C |
|---|---|
| 10.79 | 28.16 |
| 10.82 | 28.15 |
| 11.08 | 28.10 |
| 10.84 | 28.12 |
| 10.82 | 28.15 |
| 10.84 | 28.15 |
| 10.89 | 28.15 |
| 10.86 | 28.15 |
| 10.95 | 28.17 |
| 10.86 | 28.16 |
| 10.86 | 28.16 |
| 10.88 | 28.16 |
| 10.93 | 28.16 |
| 10.98 | 28.16 |
| 10.90 | 28.18 |
| 10.90 | 28.18 |
| 10.92 | 28.14 |
| n = 17 | n = 17 |
| $\bar{x}$ = 10.89 | $\bar{x}$ = 28.15 |
| S.D. = 0.070 | S.D. = 0.020 |
| S.E. = 0.017 | S.E. = 0.005 |
| Maximum difference between successive readings: | |
| 0.26 | 0.05 |

**Table 3**

**Repeatability of alternate readings using 200 ml sample size**

| **Brix @ 20°C** | **Brix @ 20°C** |
|---|---|
| 10.37 | 28.63 |
| 10.36 | 28.57 |
| 10.40 | 28.55 |
| 10.36 | 28.48 |
| 10.37 | 28.49 |
| 10.35 | 28.49 |
| 10.35 | 28.48 |
| 10.29 | 28.51 |
| 10.24 | 28.51 |
| 10.26 | 28.53 |
| n = 10 | n = 10 |
| $\bar{x}$ = 10.34 | $\bar{x}$ = 28.52 |
| S.D. = 0.053 | S.D. = 0.048 |
| S.E. = 0.017 | S.E. = 0.015 |
| Maximum difference between successive readings: | |
| 0.06 | 0.07 |

### 2.4 Calibration curve

Sugar solutions in the range 5-30°Bx were prepared from pure refined sugar. Brix determination by hydrometer was done using primary standard hydrometers calibrated by CSIRO National Measurement Laboratory. Scale and temperature corrections were added to obtain brix corrected to 20°C. Following the spindle brix determination, the solution from the brix cylinder was quickly added to both the density meter and the pycnometer. Table 4 shows the values obtained by the three methods.

**Table 4**

**Comparison of brix readings by hydrometer, pycnometer and density meter**

| | **Brix @ 20°C** | | | | | |
|---|---|---|---|---|---|---|
| Brix hydrometer | 5.37 | 9.85 | 14.32 | 19.37 | 22.78 | 27.82 |
| Pycnometer | 5.34 | 9.80 | 14.28 | 19.35 | 22.77 | 27.77 |
| EXAC 12A | 5.34 | 9.91 | 14.63 | 19.97 | 23.54 | 28.72 |
| (EXAC-hydrometer) | -0.03 | +0.06 | +0.31 | +0.60 | +0.76 | +0.90 |

Clearly, brix as determined by the density meter is unacceptable. However, by plotting spindle brix vs EXAC brix (Figure 1) a regression equation is obtained ($R_2$ = 0.9999998) which brings the brix as determined by the density meter into line with the brix as determined by the conventional spindle method.

Equation 1 takes the form:

$$B_s = C_0 + C_1{*}E + C_2{*}E^2 + C_3{*}E^3 + C_4{*}E^4$$

where $B_s$ = Brix by spindle

$E$ = Brix by EXAC density meter

$C_0$ = -0.612985

$C_1$ = 1.269992

$C_2$ = -0.03176504

$C_3$ = 0.001257671

$C_4$ = -0.00001710025

Further analyses as in Table 4 at brixes of 12, 17, 21 and 25 were carried out to see if the results fitted the regression equation 1: they did not. The results are given in Table 5.

**Table 5**

**Effect of regression equation[1] on EXAC reading**

| | Brix @ 20°C | | | |
|---|---|---|---|---|
| (a) Brix hydrometer | 12.07 | 17.16 | 20.97 | 25.27 |
| (b) Pycnometer | 12.04 | 17.13 | 20.98 | 25.29 |
| (c) EXAC 12A | 12.50 | 17.71 | 21.71 | 26.13 |
| (d) EXAC adjusted with regression equation 1 | 12.34 | 17.22 | 21.06 | 25.35 |
| Difference (d)-(a) | +0.27 | +0.06 | +0.09 | +0.08 |

The data from Table 5 were combined with those from Table 4 to give a further regression equation ($R_2$ = 0.9999465) incorporating the 10 readings.

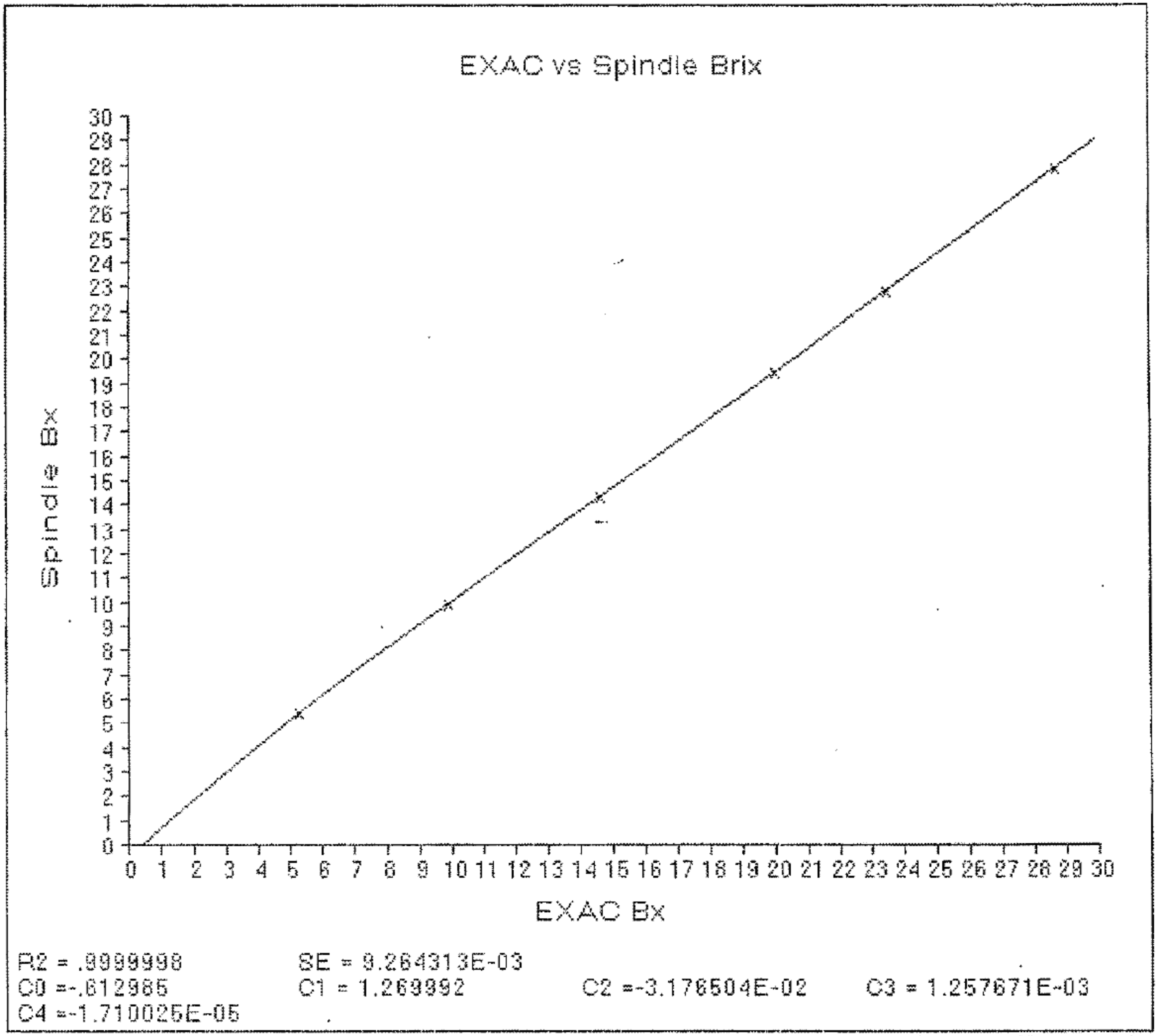


Figure 1: Correlation between EXAC and spindle brix using six observations

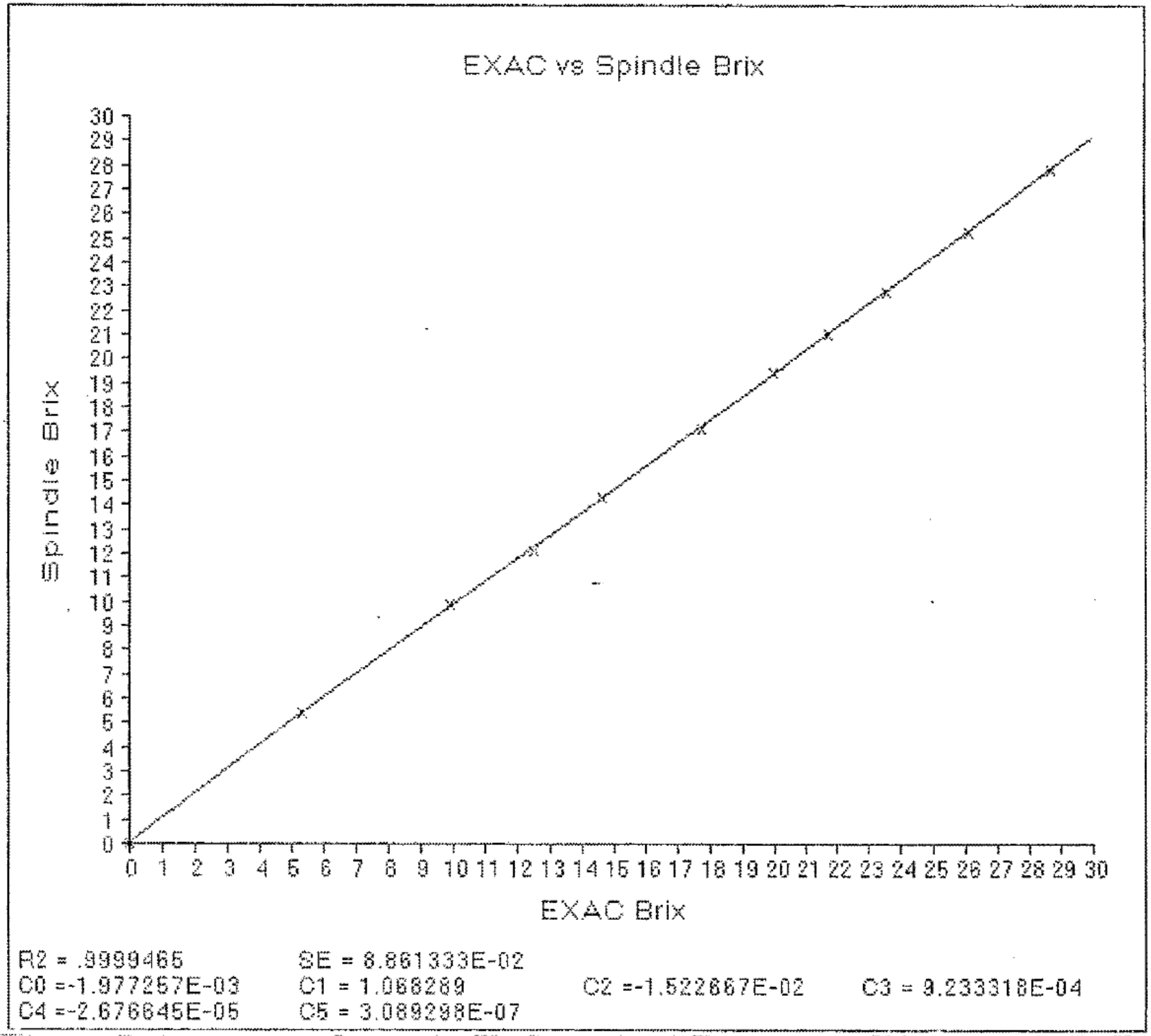


Figure 2: Correlation between EXAC and spindle brix using 10 observations

Equation 2 takes the form:

$$B_s = C_0 + C_1{*}E + C_2{*}E^2 + C_3{*}E^3 + C_4{*}E^4 + C_5{*}E^5$$

where $B_s$ = Brix by spindle

$E$ = Brix by EXAC density meter

$C_0$ = -0.001977257

$C_1$ = 1.068289

$C_2$ = -0.01522667

$C_3$ = 0.0009233318

$C_4$ = -0.00002676645

$C_5$ = 0.0000003089298

The graph of this data is shown in Figure 2.

## CONCLUSIONS

1. The precision of the density meter is acceptable as evidenced from the statistics in Table 1.

2. From Tables 2 and 3 it would seem little is to be gained in terms of accuracy by having a sample size greater than 100 mL.

3. The accuracy of the meter is poor with the current density factors and zero flow calibration. The manufacturer quotes an accuracy of 0.15%. This is equivalent to 0.0015 units of density or 0.3-0.4 units of brix. This alone is outside the accuracy necessary for cane payment analysis. Tables 4 and 5 suggest that the accuracy is outside the manufacturer's specification.

4. Perhaps the brix as determined by density meter can be aligned with spindle brix by use of a regression equation.