# SOME QUESTIONS AND ANSWERS CONCERNING ANALYSES IN FACTORY LABORATORIES

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## Abstract

It has been demonstrated that temperatures in the range 20 to 30°C have a slight but insignificant influence on the final pol values obtained for mixed juice, clarified juice, syrup and final molasses. The detrimental effect of inaccurate adjustment of sugar solutions to a pH of 7,0 when determining colour can be avoided by using triethanolamine buffer at a pH of 7,00 as solvent. No further adjustment of the pH is necessary. There are often wide differences between the Sugar Milling Research Institute and factory laboratories when analysing final molasses for pol and brix and sugars for pol. It has been shown that some laboratories can achieve good agreement and that close attention must be paid to these analyses in order to ensure correct results.

## Introduction

Many of the analyses performed to establish the content of a particular constituent in sugar and sugar products are not specific and/or quantitative. A short overview is given drawing attention to (a) mistakes often made when the pol and brix of sugar products are not read at 20°C, (b) the importance of adhering to certain analytical procedures during the preparation of sugar solutions being analysed for colour content, and (c) the discrepancies that can exist between inter laboratory analyses for pol and brix of final molasses and pol of sugars. Certain remedial steps to overcome or limit these errors are discussed.

**Table 1**

**The effect of temperature on the pol of intermediate sugar products as measured on the Polartronic Universal**

| Sample type and measuring temperature (°C) | Pol at indicated temperature (°Z) (a) | Pol at 20°C (°Z) (b) | Difference (a) - (b) |
|---|---|---|---|
| *Mixed juices* | | | |
| 1. (27,8) | 9,39 | 9,40 | −0,01 |
| 2. (28,5) | 10,24 | 10,25 | −0,01 |
| 3. (32,2) | 10,04 | 10,05 | −0,01 |
| 4. (32,4) | 10,32 | 10,35 | −0,03 |
| 5. (33,2) | 10,08 | 10,10 | −0,02 |
| 6. (33,4) | 9,47 | 9,50 | −0,03 |
| 7. (34,7) | 10,27 | 10,30 | −0,03 |
| *Clarified juices* | | | |
| 1. (26,8) | 11,14 | 11,15 | −0,01 |
| 2. (28,2) | 9,63 | 9,65 | −0,02 |
| 3. (32,7) | 11,17 | 11,20 | −0,03 |
| 4. (33,2) | 9,67 | 9,70 | −0,03 |
| 5. (33,6) | 11,02 | 11,05 | −0,03 |
| *Syrups* | | | |
| 1. (26,6) | 58,47 | 58,50 | −0,03 |
| 2. (27,6) | 60,45 | 60,45 | −0,00 |
| 3. (29,2) | 60,72 | 60,75 | −0,03 |
| 4. (30,0) | 57,57 | 57,60 | −0,03 |
| *Final molasses* | | | |
| 1. (27,4) | 29,55 | 29,50 | +0,05 |
| 2. (29,4) | 30,20 | 30,15 | +0,05 |
| 3. (30,0) | 29,78 | 29,75 | +0,03 |
| 4. (30,1) | 30,10 | 30,05 | +0,05 |

*Temperature Corrections to be Applied to the Pol and Brix Readings of Intermediate Sugar Solutions*

Equations exist to correct normal sugar solutions (i.e. 26,000 g sugar/100 cm³ of solution) for the effect of temperature when pol readings are taken at temperatures other than 20°C. This applies to both quartz-wedged or circular scale instruments. Because of the varying composition of intermediate sugar products no formulae exist that could be applied universally. Table 1 illustrates the average of 5 replicate results obtained when measuring the effect of temperature on the pol (°Z) of samples of South African sugar products. The Table also shows that for mixed juice, clarified juice and syrup the pol decreases with increasing temperature, while final molasses shows a consistent increase in polarisation with increasing temperature.

When analysing mixed juice, clarified juice and final molasses no justifiable correction for pol, when read at temperatures between 20-30°C, need be applied. It must be borne in mind that the concentration figures quoted in Table 1 are expressed on sample and that the temperature effect on the sample when read in the diluted form (*ca.* around 15° Bx) is often much less than the accuracy of the instruments or the repeatability of the method.

In most South African sugar mill laboratories, the brix of a solution will be read at 20°C, i.e. circulation of water at 20°C is used. However it was found that the brix of the solutions was sometimes read at temperatures other than 20°C and the refractometer was not equipped with temperature correction facilities. The following calculation was then erroneously performed on the basis that the purity at 20°C = the purity at say 26,5°C:

$$\frac{\text{Pol determined at 26,5°C} \times 100}{\text{Brix read at 26,5°C}} = \frac{\text{Pol determined at 20°C} \times 100}{\text{Brix read at 20°C}}$$

Because refractometers are calibrated at 20°C, the correct brix will be displayed only at 20°C and the reading obtained at 26,5°C will not be brix but brix reading. Brix cannot change, whether you consider it at 20°C or at any other ambient temperature and the above example would lead to an inflated pol e.g.:

$$\frac{31{,}85 \times 100}{82{,}25} = \frac{\text{Pol at 20°C} \times 100}{84{,}35}$$

Pol at 20°C = 32,66°Z, which is substantially different from the correct pol value of 31,85°Z.

*Measurement of Colour of Sugar*

The most important factor to consider when the ICUMSA 420 colour is determined is probably the proper adjustment

of the solution to a pH of 7,0. Over or under adjustment of the pH of sugar solutions often leads to irreversible formation or destruction of the colour present in the solution. The use of triethanolamine (TEA) buffer solution at pH 7,00 as solvent, in place of water, for refined and raw sugars was investigated.

TEA buffer was prepared as described elsewhere (Anon[1]) and the final pH was adjusted to 6,90 or 7,00, depending on the purpose for which the solution was to be used. In one instance 50° Bx solutions of sugars were prepared using the pH = 6,90 buffer and the final pH of the sugar solution was adjusted to 7,00 using 0,1 M NaOH solution, while in another series of tests the sugars were prepared (50°Bx) in the pH = 7,00 buffer and no further adjustment of pH was performed. The pH values of these solutions were noted in order to establish any drift from pH 7,00 as a result of the dissolution of the sugar in the buffer solution. The results of these tests are given in Table 2 and show that the drift away from pH 7,00 is within the specified ±0,1 pH units for raw or refined sugars.

**Table 2**

**Colours of sugars dissolved in pH 7,00 buffers and read directly and obtained by dissolving the sugars in pH 6,90 buffer and then adjusted to pH 7,00**

| Sample | Dissolved in pH 7,00 buffer | | Dissolved in pH buffer and adjusted to pH 7,00 |
|---|---|---|---|
| | Colour | pH | Colour |
| Refined | | | |
| 1 | 58 | 6,93 | 60 |
| 2 | 41 | 6,92 | 42 |
| 3 | 57 | 6,91 | 59 |
| 4 | 65 | 6,93 | 67 |
| 5 | 59 | 7,03 | 60 |
| 6 | 48 | 6,93 | 49 |
| 7 | 45 | 6,92 | 46 |
| 8 | 53 | 6,95 | 54 |
| 9 | 50 | 7,01 | 49 |
| 10 | 38 | 6,95 | 38 |
| SD calculated on difference = 0,63 ICUMSA units | | | |
| Raw | | | |
| 1 | 1201 | 6,92 | 1259 |
| 2 | 1189 | 6,94 | 1231 |
| 3 | 1241 | 6,94 | 1192 |
| 4 | 1160 | 6,95 | 1163 |
| 5 | 1180 | 6,97 | 1187 |
| 6 | 1362 | 6,92 | 1207 |
| 7 | 1172 | 6,99 | 1192 |
| 8 | 1151 | 6,97 | 1172 |
| 9 | 1222 | 6,95 | 1221 |
| 10 | 1144 | 6,97 | 1138 |
| SD calculated on difference = 46 ICUMSA units | | | |

The difference in colour values obtained between the sugars dissolved in pH 7,00 buffer and read without further pH adjustment and the colour values obtained when the sugars were dissolved in pH 6,90 buffer and the pH then set at 7,00 show that, although the colour of the non-adjusted sugars tends to be slightly lower, the standard deviations for refined and raw sugars are well within those reported elsewhere (Anon[1] and Mellet *et al.*[2]).

The effect of pH on the colour values of sugar solutions is demonstrated in Table 3 where average results for refined and raw sugars obtained on ten replicates for each sample are given at pH values of 6,90; 6,95; 7,00; 7,05 and 7,10.

Refined, VHP, HP and LP sugars were used to compare the colour values obtained using TEA buffer or water as solvent when measuring ICUMSA 420 colour. After dissolving the sugars in water or TEA buffer the colours were read at pH 7,00. The results are given in Table 4 and show that the differences between the two methods are well within experimental error. A t-test showed that there was no significant difference in the colour determined by the two methods.

**Table 3**

**Effect of pH on the colour values of various sugars**

| pH | Refined | VHP | HP | LP |
|---|---|---|---|---|
| 6,90 | 38 | 1183 | 1943 | 1585 |
| 6,95 | 39 | 1260 | 1944 | 1600 |
| 7,00 | 39 | 1263 | 1955 | 1651 |
| 7,05 | 41 | 1316 | 1983 | 1624 |
| 7,10 | 40 | 1379 | 2024 | 1688 |
| Max. Diff. % | 5,3 | 16,6 | 4,2 | 6,5 |

**Table 4**

**The effect of using TEA as solvent when measuring ICUMSA 420 colour**

| Sample | Distilled water | TEA buffer |
|---|---|---|
| Refined sugar | 45 | 46 |
| VHP sugar | 1314 | 1350 |
| HP sugar | 2253 | 2260 |
| LP sugar | 1750 | 1749 |

It is to be noted that if TEA buffer is used as solvent, the measured relative dissolved solids (RDS) of the sugar solution must be multiplied by a factor of 0,98912 to account for the RDS of the buffer.

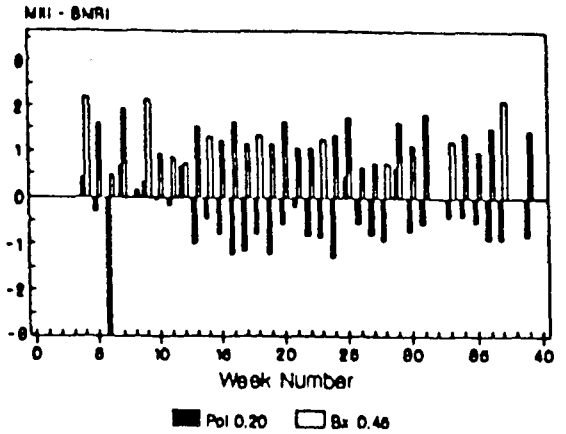
*Final Molasses Analyses*

Pol and brix analyses of final molasses are used by the factories for certain mass balances and recovery calculations. Over the past 8 - 10 years final molasses composites have been sent to the Sugar Milling Research Institute (SMRI) on a weekly basis for a full analysis which includes pol and brix. Each factory laboratory also determined brix and pol on the composites and the results are used for comparison with the SMRI, the discrepancies being summarised in bar graphs. Certain tolerances have been established for the reproducibility of pol and brix analyses (Mellet *et al.*[2]) and it is obvious from the bar graphs whether a particular laboratory is within the tolerance. Because the SMRI is able to compare its own pol and brix results with other parameters obtained on the same samples, e.g. dry solids and sucrose, and because a system of internal reference samples is used, it is generally accepted that the SMRI results are correct.

Figure 1 displays the discrepancies between the mills and SMRI analyses while Table 5 shows the percentage of cases within/out of tolerance.

If a 65% agreement with SMRI is taken as an acceptable figure, it is seen that only AK and DL achieved this percentage for pol while FX, GD, UC and SZ achieved it for brix analyses. If the out of tolerance figures for the factories had shown an equal scatter of positive and negative values, it could be argued that the season's average analysis figures would still be acceptable, but in most cases the out of tolerance analyses are either positive or negative, e.g. above tolerance for pols were: ME 94%, GD 73% and UK 74%, while below tolerance for pols were: ML 68%, PG 73%, EN 65% and SZ 71%. Good agreement between the factory and SMRI brix analyses was shown by FX 79%, GD 65%, UC 71% and SZ 68%. Above tolerance brixes were found for

## ML

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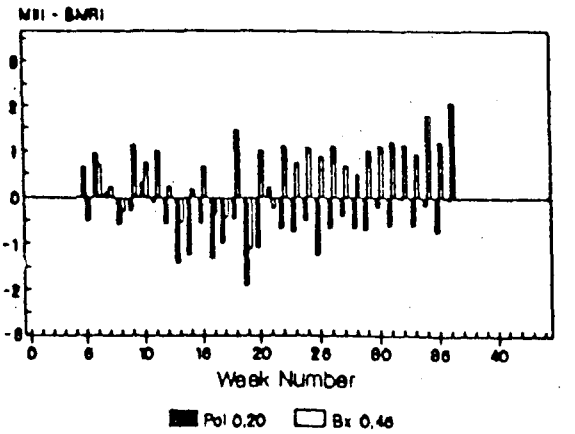


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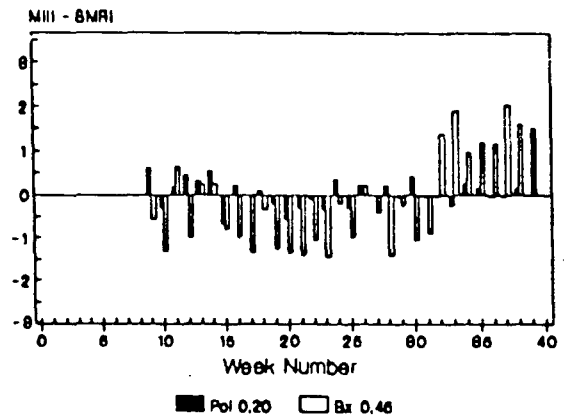


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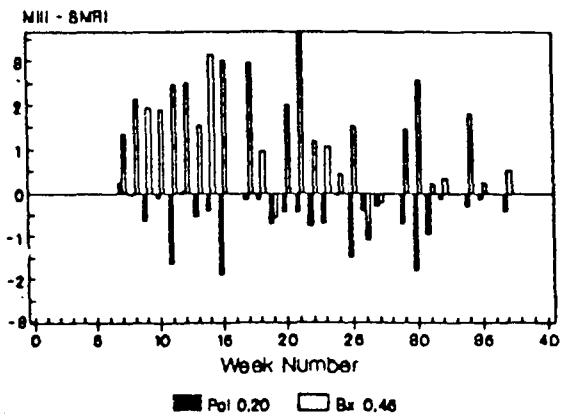
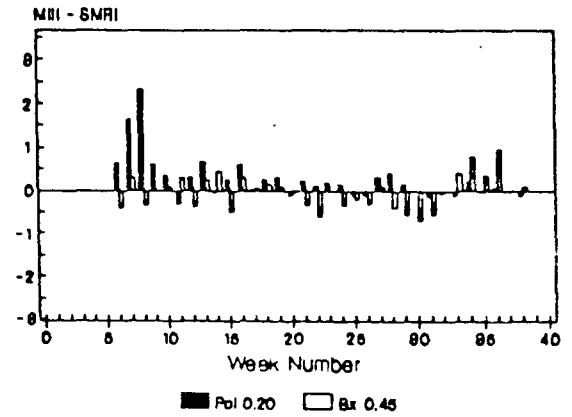
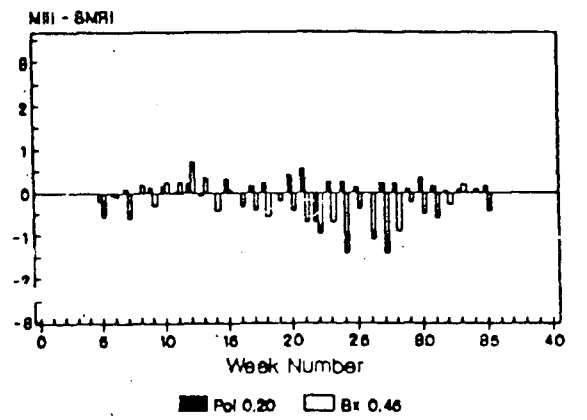


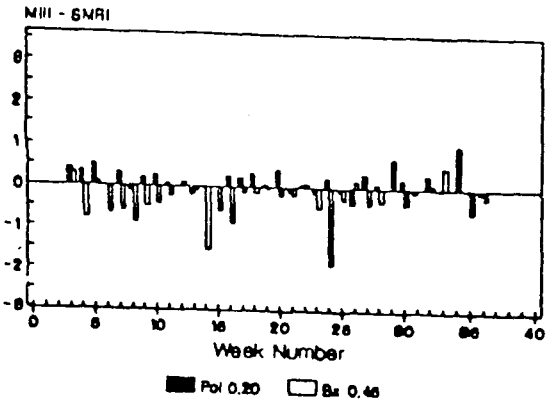

## FX



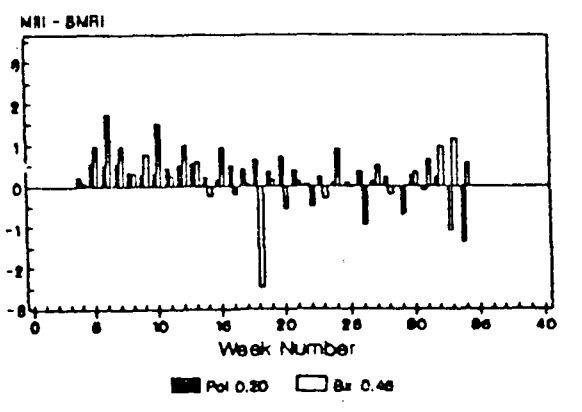

## AK




## DL




## MS



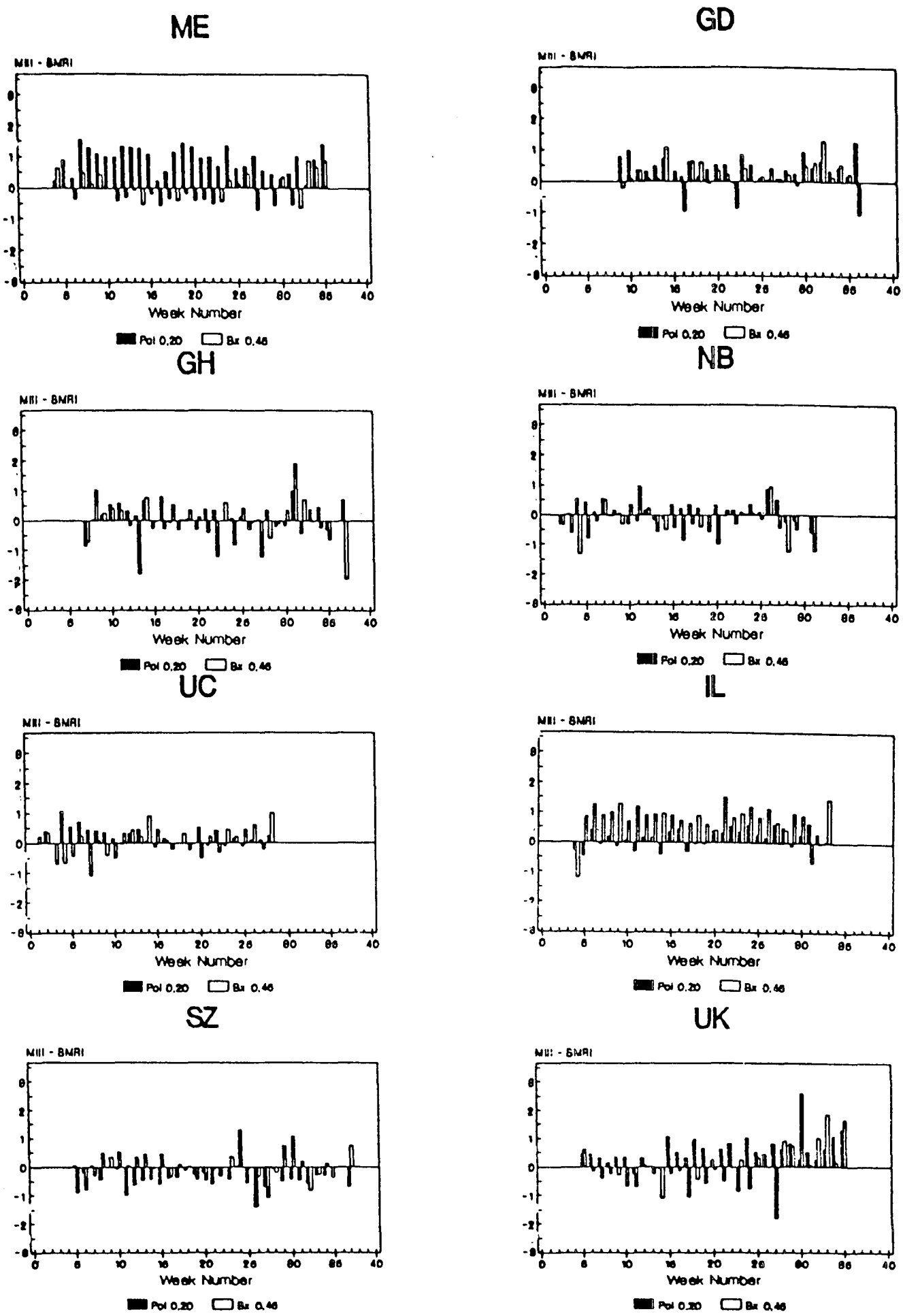



**FIGURE 1** Bar graphs illustrating pol and brix differences between factory laboratories and SMRI for the 1989/90 season.

**Table 5**

**Comparative pol and brix analyses between the SMRI and factory laboratories**

| Mill | Pol | | | Brix | | |
|---|---|---|---|---|---|---|
| | % of analyses falling within the tolerance | % of analyses above the maximum limit | % of analyses below the minimum limit | % of analyses falling within the tolerance | % of analyses above the maximum limit | % of analyses below the minimum limit |
| ML | 13 | 19 | 68 | 3 | 97 | 0 |
| PG | 17 | 10 | 73 | 20 | 70 | 10 |
| UF | 37 | 37 | 26 | 26 | 19 | 55 |
| EN | 35 | 0 | 65 | 8 | 85 | 7 |
| FX | 46 | 50 | 4 | 79* | 4 | 18 |
| AK | 71* | 23 | 6 | 58 | 3 | 39 |
| DL | 67* | 30 | 3 | 58 | 9 | 33 |
| MS | 37 | 57 | 6 | 40 | 47 | 13 |
| ME | 6 | 94 | 0 | 61 | 13 | 26 |
| GD | 27 | 73 | 0 | 65* | 27 | 8 |
| GH | 36 | 39 | 25 | 54 | 21 | 25 |
| NB | 55 | 31 | 14 | 52 | 10 | 38 |
| UC | 57 | 39 | 4 | 71* | 11 | 18 |
| IL | 43 | 40 | 17 | 10 | 83 | 7 |
| SZ | 29 | 0 | 71 | 68* | 16 | 16 |
| UK | 23 | 74 | 3 | 55 | 23 | 22 |
| SZ (82/83) | 70** | 27 | 3 | 80** | 15 | 5 |
| SZ (83/84) | 66** | 25 | 9 | 81** | 16 | 3 |
| SZ (84/85) | 67** | 19 | 14 | 72** | 22 | 6 |

* Analyses showing 65%, or more, agreement with the control values.
** SZ pol and brix analyses showing good agreement with the control values.

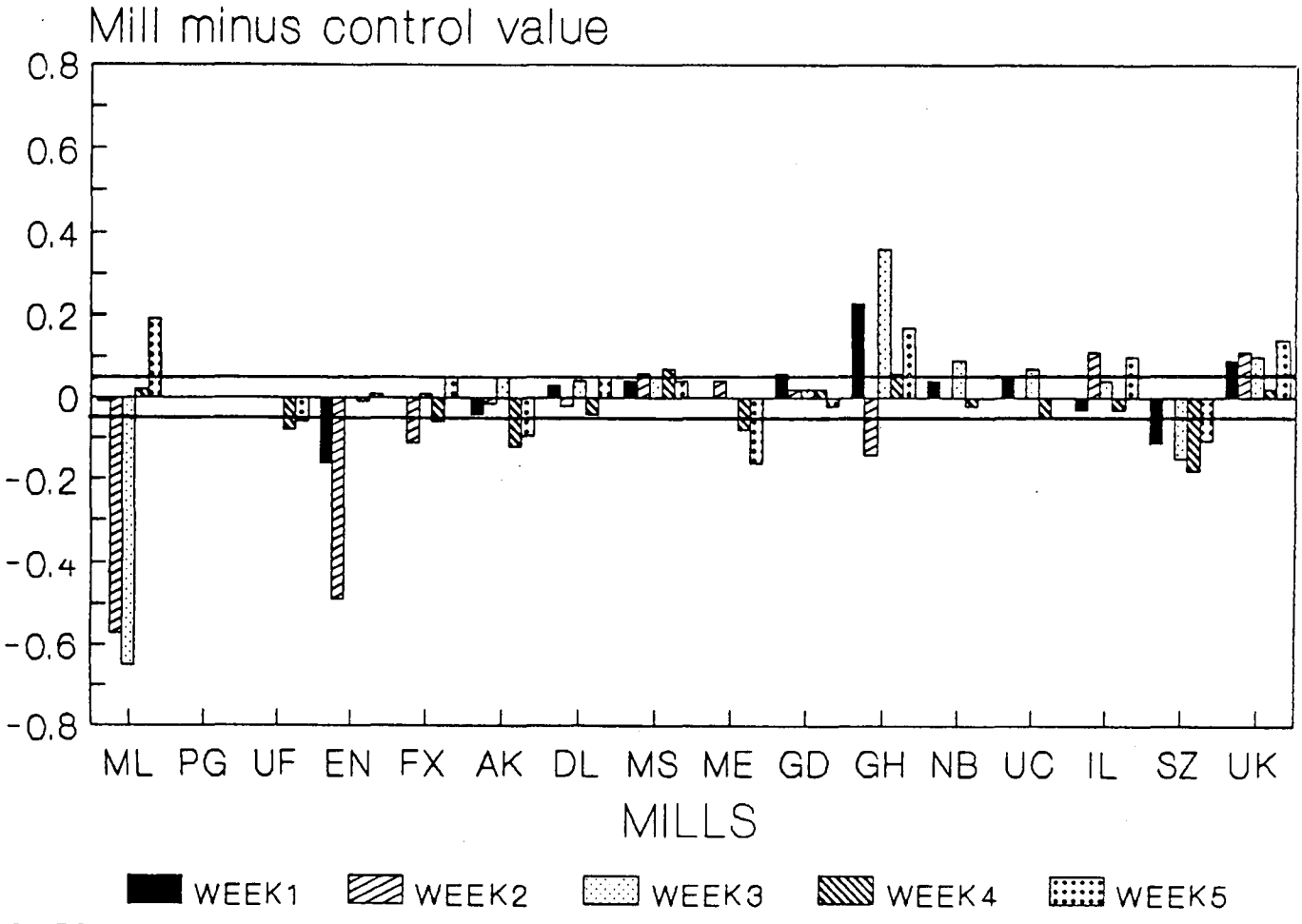



**FIGURE 2** Bar graphs illustrating pol differences between mill laboratories and control values for the month of July 1989.

ML 97%, PG 70%, EN 85% and IL 83%. In general brix analyses are seldom found to be below the lower tolerance limit. Pol and brix figures for SZ over the three seasons 1983 - 1985 are listed at the end of Table 5 to show that, with the necessary attention by the laboratory staff, both pol and brix analyses can be performed correctly.

*Sugar Analyses*

Analysis by SAST and Hulett Refineries laboratories on VHP sugar is used for penalty purposes, e.g. on pol, colour, starch and grain size. To check the analyses an inter-laboratory exchange of VHP reference samples was introduced over the 1989/90 season. The scheme included the central (SAST, Hulref, SMRI and STD) as well as the factory laboratories. The mean result obtained by the four central laboratories is taken to be the control result with which the mill laboratory results are compared.

There was a marked improvement in the mill laboratory results between the first set of results received in July 1989 and those of November 1989. It is also noticeable that the Tongaat-Hulett laboratories, which have been audited by STD for some time, generally show a much closer agreement with the control samples than most other laboratories as illustrated in Figure 2 for pol analyses. This is due to the consistent effort of the mill laboratory staff.

## Conclusions

(a) The effects of temperature on the pol readings of mixed juice, clarified juice, syrup and final molasses solutions over the temperature range tested are so small that it is not considered justifiable to apply any temperature corrections in the range of about 20 - 30°C.

(b) To avoid unnecessary colour changes in sugar solutions when attempts are made to adjust the pH of the solution to 7,0 prior to ICUMSA 420 colour determination, the use of a triethanolamine buffer solution, adjusted to pH 7,00, is recommended as solvent for refined and raw sugars.

(c) Some mill laboratories achieve pol and brix results on final molasses which generally agree well with the SMRI results, but most factories show discrepancies which are often large, and in some cases are consistently too high or too low and would therefore appreciably affect the overall season's balance. Weekly comparison of pol results obtained on check samples of VHP sugar has proved beneficial in improving analytical accuracy in mill laboratories.

## REFERENCES

1. Anon. (1986). *ICUMSA Proceedings*, 19th Session 377-389.
2. Mellet, P, Lionnet, GRE, Kimmerling, ZJ and Bennet, PJ (1982). Standards for the analytical precision of sugar and molasses analyses. *Proc S Afr Sug Technol Ass* 56: 55-57.