

LABORATORY CONTROL PROCEDURES FOR  
SOUTH AFRICAN SUGAR FACTORIES

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## S U M M A R Y

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## 1. INTRODUCTION

Over a period of many years, the SMRI has been involved in laboratory audits in both South African and Affiliated mills. It now seems appropriate to suggest certain procedures to be followed by each mill laboratory so that it can become self sufficient in the control of sample handling and the correctness of analyses performed. This report therefore deals with sampling and sub-sampling of products, control and checking procedures to be followed regarding instrumentation and the systematic use of reference samples in the daily/weekly programme of analysis. A list of reference samples and how they should be prepared is included.

The comments are to be considered supplementary to the Laboratory Manual for South African Sugar Factories (1985 Ed.) and are therefore to be read in conjunction with it.

## 2. SAMPLING

Detailed instructions as to the size, type and frequency of sampling of factory materials are given in the Laboratory Manual for South African Sugar Factories (1985 Ed.), Table 14, pp. 413 - 415. In addition close attention must be paid to all aspects discussed in Chapter 7 of this manual.

Systematic inspection of the sampling points, the type of sampling and the frequency of sampling of the various products must be undertaken by accompanying the sampler when the sample is collected. Special attention must be paid to the cleanliness of apparatus, covering of receptacles with lids, cooling of samples where necessary and proper sub-sampling where applicable.

It is recommended that the sampling of products be inspected and reported on once per month.

## 3. INSTRUMENTATION

### 3.1. General

A permanent record should be kept of all checks performed. These should be clearly set out in terms of date, actual readings versus observed readings together with the Serial Numbers of the instruments and any other information/remarks deemed necessary.

### 3.2. Balances

A set of standard weights should be available to check all balances once per month. Those laboratories that don't have standard weights may, on request, make use of the weights from SMRI or the Sugar Industry Central Board. The weights should range from 1,0000 g to 150,0000 g for checking analytical balances and thereafter in 100,00 g steps up to 2000,00 g to check the light duty top pan balances. For checking the top

pan balances used for weighing larger samples, standard weights ranging from 1 000 to 10 000 g in steps of 1 000 g and calibrated to  $\pm 0,1$  g should be available. Rather than having set Service Contracts with a supplier, it is more practical (and also much cheaper) to have all the balances serviced during the off-crop and call for balance technicians only when necessary.

### 3.3. Polarimeters and Saccharimeters

#### Note:

A new international sugar scale was officially recognised by ICUMSA at its 1986 session and is to go into effect July 1, 1988. Until that time the old scale remains valid. The new value increases the 100°S point by 0,03%, thus lowering the new values by 0,03% compared with the present values. At the same time the notation °S will be replaced by °Z to exclude any confusion between the old and the new scales.

#### 3.3.1. Temperature corrections

It is preferable that the polarimeter or saccharimeter as well as the quartz calibration plates are kept in a temperature controlled room ( $20 \pm 0,5^\circ\text{C}$ ).

If this facility is not available then the appropriate temperature correction must be applied to the quartz plate's calibrated value at  $20^\circ\text{C}$ .

##### (a) For saccharimeters

For quartz wedge saccharimeters, e.g. the Saccharomat III, there is no temperature correction to be applied at temperatures other than  $20^\circ\text{C}$  because the quartz wedge(s) in the instrument will, for all practical purposes, be affected to the same degree as that of the quartz plate (both being at very similar temperatures).

##### (b) For polarimeters

The polarimeter, which is not a quartz compensated instrument but is based on circular-scale compensation, has however to be corrected for temperatures other than  $20^\circ\text{C}$ . The correction to be applied can be calculated by the formula:

$$\text{Correction} = (t-20) \times 0,00014 \times Q_{20}$$

where

- t = temperature of the quartz plate when the reading is taken
- $Q_{20}$  = calibrated value of the quartz plate at 20°C.

This correction will be positive and must be added to the calibrated value at 20°C if the temperature of reading is above 20°C and be subtracted from the calibrated value at 20°C if the temperature is below 20°C, because in this case the correction will be negative.

The Polartronic, which is at present the only polarimeter in use in the South African sugar industry, can be adjusted manually to display the correct reading of the quartz plate. If the instrument and quartz plate are both at 20 + 0,5°C, then the instrument is set to display the certified value of the plate at 20°C (e.g. 99,5°S). If the temperature is different from 20°C, e.g. 23,5°C, then

$$\begin{aligned} \text{Correction} &= (t-20) \times 0,00014 \times 99,50 \\ &= 0,049^\circ \\ &= 0,05^\circ \end{aligned}$$

The correct value of the quartz plate is therefore 99,50 + 0,05 = 99,55°S at 23,5°C and the Polartronic can be adjusted to display 99,55°S.

### 3.3.2. Instrument correction

#### (a) For saccharimeters

The difference in value engraved on the quartz plate and that shown by the saccharimeter must be subtracted from the observed value of the sample.

#### Examples

- (i) Quartz plate certified value at 20°C  
= 99,50°S  
Quartz plate displayed value at 24°C  
= 99,55°S  
Difference, which is the instrument  
correction = + 0,05°S

Therefore the correct sample reading:  
Sample reading observed - (+ 0,05°S)  
i.e. Sample reading observed - 0,05°S.

- (ii) Quartz plate certified value at 20°C  
= 99,50°S  
Quartz plate displayed value at 18°C  
= 99,40°S  
Difference, which is the instrument  
correction = - 0,10°S

Therefore the correct sample reading:  
 Sample reading observed - (- 0,10°S)  
 i.e. Sample reading observed + 0,10°S.

(b) For polarimeters

Provided that the instrument was set to display the correct quartz plate reading at the existing temperature then there is no instrument correction to be applied to the sample reading.

3.3.3. Temperature correction for normal sugar solutions

The temperature correction to be applied to a normal (i.e. 26,000 ± 0,002 g per 100 cm<sup>3</sup> of solution) VHP sugar solutions is :

(a) For saccharimeters

Correct the reading for the effect of temperature according to

$$P_{20} = P_t + 0,033 (t_r - 20).$$

where

$$P_{20} = \text{pol at } 20^\circ\text{C}$$

$$P_t = \text{pol as measured}$$

$$t_r = \text{temperature of solution in } ^\circ\text{C}.$$

(b) For polarimeters

Correct the reading for the effect of temperature according to

$$P_{20} = P_t + 0,019 (t_r - 20)$$

The correction must be added to the reading of the sugar solution if the temperature of the sample at reading is above 20°C. For temperatures below 20°C, the correction must be subtracted from the observed polarisation.

3.3.4. Temperature correction for other sugar products

For solutions with lower sugar concentrations, e.g. mixed juice, diluted syrups and molasses, etc. no temperature correction is applied to the sample reading provided the temperature of the sample at reading falls between 15 - 27°C.

3.3.5. Linearity

It is necessary to check the linearity of the instrument before the start of the season and at 6 monthly intervals throughout the season or when any doubt as to its accuracy arises, e.g. when a quartz plate is suspect. This is done

by preparing accurate solutions from pure sucrose or a good first boiling refined sugar, calculating the expected values and comparing them with the actual readings obtained. All masses should be determined on an analytical balance and all solutions must be prepared in scrupulously clean vessels with tight fitting stoppers.

For example

Flask A contains 5,1234 g sucrose and 94,6789 g water, i.e. 99,8023 g in total.

Flask B contains 15,0146 g sucrose and 85,9172 g water, i.e. 100,9318 g in total.

Flask C contains 23,5176 g sucrose and 76,4814 g water, i.e. 99,9990 g in total.

Flask A contains 5,1234 g sucrose per 99,8023g solution which is equivalent to

$$\frac{5,1234}{99,8023} \times \frac{100}{1} = 5,13^{\circ}\text{Bx}$$

Flask B contains 15,0146 g sucrose per 100,9318 g solution which is equivalent to

$$\frac{15,0146}{100,9318} \times \frac{100}{1} = 14,88^{\circ}\text{Bx}$$

Flask C contains 23,5176 sucrose per 99,9990 g solution which is equivalent to

$$\frac{23,5176}{99,9990} \times \frac{100}{1} = 23,52^{\circ}\text{Bx}$$

From Column (2) in Table 8 in the Laboratory Manual for South African Sugar Factories (1985 Ed.) the above brix values can be converted to grams per 100 cm<sup>3</sup> of solution in air at 20°C by multiplying the brix value by the apparent density from which figure an expected saccharimeter reading can be calculated as follows:

$$\begin{aligned} \text{Solution A} &= 5,13^{\circ}\text{Bx} \times 1,01731 \text{ (by interpolation)} \\ &= 5,219 \text{ g sucrose per } 100 \text{ cm}^3 \end{aligned}$$

$$\begin{aligned} \text{Solution B} &= 14,88^{\circ}\text{Bx} \times 1,057596 \text{ (by interpolation)} \\ &= 15,737 \text{ g sucrose per } 100 \text{ cm}^3 \end{aligned}$$

$$\begin{aligned} \text{Solution C} &= 23,52^{\circ}\text{Bx} \times 1,095732 \text{ (by interpolation)} \\ &= 25,772 \text{ g sucrose per } 100 \text{ cm}^3 \end{aligned}$$

$$\begin{aligned} \text{Since } 26,0000 \text{ g sucrose per } 100 \text{ cm}^3 \text{ solution} \\ &= 100,00^{\circ}\text{S} \end{aligned}$$



then

$$\text{Solution A} = \frac{5,219 \times 100}{26} = 20,07^{\circ}\text{S}$$

$$\text{Solution B} = \frac{15,737 \times 100}{26} = 60,53^{\circ}\text{S}$$

$$\text{Solution C} = \frac{25,772 \times 100}{26} = 99,12^{\circ}\text{S}$$

Keeping in mind that the Polartronic reads to  $0,01^{\circ}\text{S}$  and the Saccharomat III to  $0,05^{\circ}\text{S}$ :

solution A should read  $20,07^{\circ}\text{S}$  on the Polartronic and  $20,05^{\circ}\text{S}$  on the Saccharomat III,

solution B should read  $60,53^{\circ}\text{S}$  on the Polartronic and  $60,55^{\circ}\text{S}$  on the Saccharomat III

and solution C should read  $99,12^{\circ}\text{S}$  on a Polartronic and  $99,10^{\circ}\text{S}$  on a Saccharomat III.

These values are measured at  $20^{\circ}\text{C}$ . If the measurements are made at temperatures other than  $20^{\circ}\text{C}$  then the necessary corrections should be applied as under 3.3.1. - 3.3.3. above. These same solutions may also be used to calibrate a refractometer since their accurate brix values are known (see 3.3.5. above).

### 3.4. Refractometers

If at all possible refractometers must be operated at  $20 \pm 0,5^{\circ}\text{C}$ . If temperature control facilities are not available the necessary corrections must be applied by making use of brix temperature correction tables, e.g. Table 2 in the Laboratory Manual for South African Sugar Factories (1985 Ed.).

#### 3.4.1. Calibration

Using a first boiling refined sugar, weigh accurately about 5, 10, 15, 20 and 25 g to 3 decimal places into 250 cm<sup>3</sup> Erlenmeyer flasks and add distilled water to give approximately 100,000 g weighed to 3 decimal places. The correct brix of each solution can then be calculated, i.e.

<u>Weight of sugar, g</u>	<u>Weight of solution, g</u>	<u>Brix of solution, °Bx</u>
4,837	100,100	$\frac{4,837 \times 100}{100,100} = 4,83$
10,213	100,008	$\frac{10,213 \times 100}{100,008} = 10,21$

14,876	100,209	$\frac{14,876 \times 100}{100,209} = 14,84$
21,011	100,110	$\frac{21,011 \times 100}{100,110} = 20,99$
24,988	100,200	$\frac{24,988 \times 100}{100,200} = 24,94$

Before checking the brix readings of these solutions on the refractometer, make sure that the prism(s) and sample cell are scrupulously clean by washing with distilled water and detergent.

If the span needs readjustment, set it by making use of the 24,94°Bx solution. Once the span has been set, check the brix readings of the intermediate solutions. If these values do not agree with the calculated values, prepare a new 25°Bx standard solution in duplicate and re-check the correctness of the original span setting. If the intermediate standard solutions still give brix readings different from the calculated values, the instrument should be serviced by an approved instrument technician. The checking procedure using the ca. 25°Bx solution should be performed once daily.

#### 3.4.2. Maintenance

This should be done according to the instructions in the Laboratory Manual for South African Sugar Factories (1985 Ed.).

#### 3.5. pH Meters

The laboratory should stock three standard buffer solutions of pH 4,0; pH 7,00 and pH 9,00. If the pH range to be measured is large then the pH meter has to be standardised at two pH values, e.g. by using the reference buffer solutions at pH 4,00 and pH 9,00.

If the pH solutions are to be adjusted, e.g. for colour measurements, the pH meter should be set at pH 7,00 using only the standard buffer of pH 7,00.

Standardisation should be performed once per day. If a slow response or poor span between two buffer values is observed rejuvenation of the electrode may be necessary and can be done as described in the Laboratory Manual for South African Sugar Factories (1985 Ed.), paragraph (e), page 155.

#### 3.6. Ovens

It is strongly recommended that each laboratory should have a thermo-couple with a digital display meter so

that temperatures can be checked easily. Before any temperature is checked with the thermo-couple, check the correctness of its reading by immersing it in boiling distilled water. Temperature corrections for the effect of altitude can be obtained from Table 1 below.

Table 1

Effect of Altitude on the Boiling Point of Distilled Water

Altitude, m	Boiling point, °C
0	100
200	99,3
400	98,6
600	97,8
800	96,3
1 000	96,8

When checking the correctness of the individual compartments insert the thermo-couple in the centre of the sample and follow the rise of the sample temperature visually. The temperature should stabilise around 105°C within 10 minutes after starting the heating cycle. If this is not achieved the oven should be serviced by a suitably qualified technician. The maximum/minimum heating cycle of the ovens should be monitored as the control switches in and out. This should not exceed  $105 \pm 3^\circ\text{C}$ . These checks should be performed once per week.

If this is not achieved the oven should be serviced by a suitably qualified technician. It is recommended that all oven doors be fitted with suitable external catches to ensure proper closing. Inspect all sample tray gauzes to ensure that they are neither broken nor blocked.

Ovens used for drying of sugar, filter cake, mud, etc. should also be checked to ensure that they operate at the correct temperatures.

### 3.7. Water Dispenser

Observe that the dispenser functions correctly by checking that the water supply is shut off promptly, thus preventing any further dripping of water. Place a tared container of suitable size under the discharge of the dispenser and weigh the discharged water. For Autolab a tolerance of  $\pm 10$  g is permitted but otherwise the dispenser should discharge the requisite quantity of water to within 1 g determined on five replicates. This checking procedure should be performed once per week.

### 3.8. Cold\_Digester

Check that the shaft has three pairs of cross-mounted spring steel blades and that the outer edges are sharp and not worn. Make sure the automatic timers on all digesters are set between 19,5 and 20,5 minutes. Check that the cutter blades are rotating at 7 200 rpm and that the bowls, blades, etc. are clean. This checking procedure should be performed once per week.

### 3.9. Decanter

Check for cleanliness, adequate cooling (i.e. the cooled sample temperature should be within 5°C of that of the cooling water) and that no cooling water leaks into the sample. The checking procedure should be performed once per week.

### 3.10. Juice Riffle

Check for cleanliness and that the alignment of the stage funnels is correct. Observe the operator when he/she is decanting the sample into the riffle for sub-sampling.

This should be performed after the sample has been well mixed and the decanting should be done in one rapid constant, continuous discharge. The checking procedure should be performed once per week.

### 3.11. Conductivity Meter

Prepare a 0,01M potassium chloride solution as described in the Laboratory Manual for South African Sugar Factories (1985 Ed.), pp. 156 - 157. Check the cell constant against this solution adhering strictly to the method described in the manual and compare it with the cell constant in use in the laboratory. If the cell constant as determined differs by more than 0,01 compared with the one in use the cell must be cleaned by soaking it in sodium hypochlorite solution for 5 minutes and washing well afterwards with distilled water. Re-establish the cell constant.

Confirm the validity of the cell constant by analysing four VHP sugars of which the conductivity/sulphated ash contents are known (obtainable from SMRI). The checking procedure should be performed once per month.

### 3.12. Spectrophotometer

Check the instrument and cuvettes for cleanliness. Analyse at least four sugar samples, two refined, two raw, with known colour contents. If the colour values for the refined sugars differ by more than 3 units from the established values or those of the raws by more than 120 units from those of the reference samples, check the accuracy of the pH settings using new standard buffer solutions to set the pH meter. Also ensure

that the chemicals used in the preparation of the samples are in accordance with those specified in the Laboratory Manual for South African Sugar Factories (1985 Ed.).

If the inspection of pH, chemicals, etc. reveals no error, check the correctness of the wavelength settings of the instrument by using one, or both of the following procedures:

(a) Potassium permanganate

Accurately weigh 0,7900 g Analar potassium permanganate, dissolve in distilled water and transfer to a 1 litre volumetric flask. Make to the mark and pipette 10 cm<sup>3</sup> of this solution into a 100 cm<sup>3</sup> volumetric flask, add 1 cm<sup>3</sup> of concentrated sulphuric acid, and 1 cm<sup>3</sup> of orthophosphoric acid, mix, cool and make to the mark. Store in a dark bottle. Read in a 1 cm cell against water at 310 nm and 545 nm.

$$\begin{aligned} \text{OD at 310 nm} &= 0,870 \pm 0,025 \\ \text{OD at 545 nm} &= 1,108 \pm 0,035 \end{aligned}$$

Note that the stock solution must be made up freshly each time the test is done.

(b) Potassium dichromate

Accurately weigh 1,4700 g Analar potassium dichromate, dissolve in distilled water and transfer to a 1 litre volumetric flask. Make to the mark and pipette 10 cm<sup>3</sup> of this solution into a 100 cm<sup>3</sup> volumetric flask, add 1 cm<sup>3</sup> of concentrated sulphuric acid and 1 cm<sup>3</sup> of orthophosphoric acid, mix, cool and make to the mark. Read in a 1 cm cell against water at 350 nm

$$\text{OD at 350 nm} = 1,413 \pm 0,045$$

These tests should be performed once per month and if the absorbances measured fall outside the stated values the instrument must be sent for repairs. The stock solution must be made up freshly each time the test is done.

### 3.13. Muffle Furnace

Check the correctness of the displayed temperature by means of a suitable thermo-couple once per month.

## 4. ANALYSES

### 4.1. General

It is not usually necessary to check that the analyses

performed on all products are being done correctly, e.g. pol and brix analyses needn't be performed on first expressed juice, mixed juice, clear juice, filter feed, filtrate, syrup, all massecuites, molasses etc. By selecting a number of products where reference samples can be introduced for check analyses a reasonably sound evaluation can be made of whether analyses are being done correctly.

#### 4.2. Reference Samples

Each laboratory should at all times have adequate reference samples available. These samples can be prepared during the off-crop and stored in well sealed containers and, depending on the type of sample, be kept in the refrigerator, deep freeze, or in cupboards.

Reference samples should be prepared by thoroughly pre-mixing bulk samples and sub-dividing each into 30 smaller samples weighing about 200 g. Raw sugar sub-samples should be prepared from VHP sugar and kept in heat sealed thick wall plastic sachets. The samples must be stored in a cupboard to avoid unnecessary exposure to daylight. Samples of final molasses should be kept in well sealed plastic bottles with screw type lids. After the lids of the plastic bottles have been fitted the lids must be further secured by wrapping several turns of masking tape round the junction between lid and bottle. All final molasses reference samples should be stored in a deep freeze, otherwise in a refrigerator. Prior to opening final molasses references samples for use they should be left overnight at ambient temperature otherwise condensate may form on the surface of the cold sample when the bottle is opened leading to dilution of the sample.

It is necessary that sugar reference samples be prepared for pol, moisture, colour, grain size and conductivity ash using VHP sugars, and reference samples of refined sugars for colour, grain size and conductivity ash. Final molasses reference samples should be prepared for pol, brix, dry solids, sulphated ash, reducing sugars and sucrose.

Once the bulk samples have been adequately sub-sampled, one of each of the sugar and final molasses sub-samples should be forwarded to the SMRI for analysis. The values obtained by the SMRI should be considered to be accurate and when the reference samples are used in future for control purposes the following tolerances must be accepted. If the answers obtained by the mill laboratory staff fall outside these tolerances a thorough investigation to establish the reason(s) must be conducted. If necessary the SMRI can be contacted to assist with this.

#### Tolerances

Raw Sugar (VHP)

Pol  $\pm$  0,05°S  
 Moisture  $\pm$  0,02%  
 ICUMSA colour  $\pm$  120 units  
 % Fines  $\pm$  3% absolute  
 Conductivity ash  $\pm$  0,02% absolute

Refined Sugar

Colour  $\pm$  3 ICUMSA units  
 Conductivity ash  $\pm$  0,003% absolute  
 % Through 0,600 mm  $\pm$  3% absolute

Final Molasses

Pol  $\pm$  0,10°S absolute  
 Brix  $\pm$  0,5°Bx absolute  
 Dry solids  $\pm$  0,3% absolute  
 Sulphated ash  $\pm$  0,2% absolute  
 Reducing sugars  $\pm$  0,3% absolute (Lane and Eynon)  
 Sucrose  $\pm$  0,5% absolute (Lane and Eynon)

4.3. The Use of Reference Samples

Once per month a VHP reference sample should be introduced for all shift analysts to determine pol, moisture, colour and where applicable, grain size and conductivity ash. Final molasses reference samples should similarly be introduced for pol, moisture, brix reducing sugars and sucrose and where applicable for dry solids and sulphated ash. Refined sugar reference samples should be analysed monthly by all shift analysts for colour and ash, and if required, for grain size distribution.

A record should be kept of all results obtained.

4.4. General Laboratory Control4.4.1. The frequency of checking instruments

The procedures outlined in Table 2 are proposed.

5. ANNUAL LABORATORY AUDIT

For the proper functioning of a laboratory it is essential that a complete audit of all its functions be performed once a year. It is suggested that this audit be performed during the off-crop.

First it is necessary to check all instrumentation used by the laboratory referring to Section 3 of this report and Chapters 4, 5 and 6 in the Laboratory Manual for South African Sugar Factories (1985 Ed.). All instruments used must function correctly before any attempts are made to check testing procedures, and all standard graphs must be re-established.

Table 2

Frequency of Testing Various Instruments in the Laboratory

Frequency	Action
<u>Per Shift</u>	Polarimeter/saccharimeter readings: 3.3.1., 3,3,2., 3.3.3., 3.3.4.  (Keep strict records of all results)
<u>Per Day</u>	Refractometer span: 3.4.1. pH meters: 3.5.  (Keep strict record of all results)
<u>Per Week</u>	Ovens: 3.6. Water dispenser: 3.7. Cold digester: 3.8. Decanter: 3.9. Juice riffle: 3.10.  (Keep strict record of all results)
<u>Per Month</u>	Sampling: 2. Balances: 3.2. Conductivity meter: 3.11. Spectrophotometer: 3.12. Muffle furnace: 3.13. Reference samples for all shift analysts: 4.3.  (Keep strict record of all results)
<u>Six Monthly</u>	Linearity of polarimeter/saccharimeters: 3.3.5. Linearity of refractometers: 3.4.1.  (Keep strict record of all results)



While the instruments are being checked, all the reference samples should have been prepared and the SMRI analysis results should be available to allow all analysts that would normally be involved in analyses to do the necessary analyses in duplicate. It is however imperative for the Chief Chemist to do these analyses himself to serve as a major control. If no agreement can be obtained between the Chief Chemist's results and those of the SMRI, then please call on the SMRI for assistance. If the Chief Chemist's results are however in accordance with those of the SMRI he must personally assist those analysts where problems exist. Only as a last resort should SMRI be called upon to assist in these cases.