Method 6.1 – Boiling house products: Brix, pol and purity

1. [Scope]

T[his method is ap]plicable to A-, B- and C- (or final) molasses, A-, B- and C-nutsch, A-, B- an[d C-mass]e[cuit]e, seed and magma and may be used to obtain data for factory control pur[poses and] for purchasing contracts (final molasses). All measurements must be made at 20.0 ± 0.1°C. The method refers to Method 11.2 for the analysis of lead.

2. Principle

The well-mixed sample is diluted (1+4 or 1:5) and divided into two portions. The first portion is filtered with the help of a filter aid and used to determine the Brix of the solution. The second portion is further diluted and reacted with lead powder for clarification and used to determine the pol of the solution. When calculating the final pol of the sample the Brix of the solution is always needed.

3. Definitions

3.1   Brix

The refractometer Brix of a solution is defined as the concentration of the total dissolved solids in solution (in grams of solute per 100 g of solution). For solutions containing only pure sucrose in water, Brix is a measure of the concentration of the sucrose. The measurement is affected by the presence of suspended matter which must therefore be removed by filtration. It is essential that the measurement be carried out at 20.0°C.

3.2   Pol

The pol (polarisation) of a solution is defined as the concentration (in grams of solute per 100 g of solution) of a solution of pure sucrose in water having the same optical rotation as the sample at the same temperature. For solutions containing only pure sucrose in water, pol is a measure of the concentration of the sucrose; for solutions containing sucrose and other optically active substances, pol represents the sum of the rotations of the constituents present and is therefore referred to as "apparent sucrose". In cane sugar factory streams, the contribution of sucrose to this sum far exceeds that of other constituents. Pol is expressed in °Z according to the International Sugar Scale. It is essential that the measurement be carried out at 20.0°C.

3.3   Refractive apparent purity

The refractive apparent purity of a solution is defined as the percentage ratio of pol to Brix in the sample.

4. Apparatus

4.1   **Refractometer** readable to 0.01°Bx

The refractometer should either be equipped with a temperature sensor or be jacketed and connected to a thermostatically-controlled water bath to maintain a solution temperature of 20.0 ± 0.1°C during measurement.

4.2   Polarimeter/saccharimeter calibrated in sugar degrees (°Z) with a visible light source at 589 nm

4.3   Polarising tube: length 200 mm

The tube should either be connected electronically to the polarimeter *via* a temperature sensor or be jacketed and connected to a thermostatically-controlled water bath to maintain a solution temperature of 20.0 ± 0.1°C during measurement.

4.4   Quartz control plate: ± 50°Z, officially certified at 20.0 ± 0.05°C to two decimal places

4.5   Top pan balance readable to 0.01 g

4.6   Schott bottles: 2×250 cm^3

4.7   Filtration apparatus

funnels: 2×100 mm ϕ stemless
beakers: 4×150 cm^3
watch/cover glass: 2×100 mm ϕ

4.8   Water bath thermostatically controlled at 20.0 ± 0.1°C

If the polarising tube and refractometer are not equipped with temperature sensors a thermostatically controlled circulating water bath connected to the tube and refractometer and maintained at 20.0 ± 0.1°C is absolutely necessary. A water bath to bring the sample temperatures to 20.0 ± 0.1°C is then also needed.

4.9   Conical flask and stopper: 500 cm^3

4.10   Sample shaker

4.11   Pipette: 100 cm^3

4.12   Volumetric flask: 200 cm^3

4.13   Filter paper

Whatman No. 6, Postslip medium white w/s or equivalent (for Brix): 185 mm ϕ
Whatman No. 91, S&S 3000 or equivalent (for pol): 185 mm ϕ

5. Reagents

5.1   Celite 577

Celite is an inert powder and inhalation may cause asbestosis of the lungs. Wear a dust mask during use.

5.2   Lead sub-acetate powder

Lead sub-acetate trihydrate [Pb(OAc)$_2$· 3H$_2$O], also called basic lead acetate, is poisonous and will accumulate in the human body. Direct contact through the skin, inhalation (powder dust) or swallowing should be avoided.

The lead sub-acetate should conform to the following specifications:

Basic lead (as PbO)	> 33%
Moisture at 105°C	< 1.5%
Insoluble in dilute acetic acid	< 0.02%
Insoluble in water	< 1.0%
Chloride (Cl)	< 0.003%
Nitrate and nitrite (NO_3)	< 0.003%
Copper (Cu)	< 0.002%
Substances not precipitated by H_2S (as sulphates)	< 0.30%
Iron	< 0.002%

Refer to Method 11.2 for the determination of the total and basic lead content of lead sub-acetate.

6. Procedure

6.1   Sample stock solution

Weigh 50.00 ± 0.05 g of the well-mixed sample into a 500 cm³ conical flask. Weigh 200.00 ± 0.05 g of water into the same flask to bring the total mass to 250.00 ± 0.10 g. Record these masses to calculated the dilution factor. Stopper the flask and mix thoroughly on the sample shaker.

6.2   Brix determination

Pipette 100 cm³ of the well-mixed stock solution and transfer to the 250 cm³ Schott bottle. Weigh 2 g Celite 577 powder while wearing a dust mask and add to the Schott bottle. Mix and filter the solution through fluted filter paper (Whatman No. 6, Postslip medium white w/s or equivalent) supported in a funnel which rests directly on a beaker. Seal the funnel with a watch glass to minimise evaporation. Discard the first 10 cm³ of filtrate and collect about 20 cm³ of the filtrate in another clean, dry beaker. Do not allow the filtrate to touch the bottom of the funnel or filter paper. Do not replenish the solution in the filter funnel.

6.2.1   Reading of the sample (filtrate)

Zero the refractometer using distilled water. Pour the filtrate into the refractometer cell compartment using three portions to ensure complete displacement of the previous solution. Record the reading once it stabilizes at 20.0°C.

6.3   Pol determination

Weigh the following amounts of dry lead sub-acetate powder while wearing a dust mask and gloves in a 250 cm³ Schott bottle:

A-massecuite, seed and magma	4 g
A-molasses and A-nutsch	5 g
C-massecuite	6 g
B-massecuite, B- and C-molasses and B-nutsch	10 g

For *C-molasses, C-massecuite* and *C-magma* pipette 100 cm³ of the stock solution and transfer to a 200 cm³ volumetric flask. Add about 50 cm³ water and swirl gently to mix. Make to the mark, stopper and mix. To allow for this dilution the first polarimeter reading must be multiplied by 2. Transfer the dilute mixture to the Schott bottle while wearing a dust mask.

For *all the other products* pipette 150 cm^3 of the stock solution and transfer to the Schott bottle while wearing a dust mask.

Shake vigorously and allow the solution to stand for at least 10 minutes to permit settling of the precipitate. Filter the solution through fluted filter paper (Whatman No. 91, S&S 3000 or equivalent) supported in a funnel which rests directly on a beaker. Seal the funnel with a watch glass to minimise evaporation. Discard the first 25 cm^3 of filtrate and collect about 60 cm^3 of the filtrate in another clean, dry beaker. Do not allow the filtrate to touch the bottom of the funnel or filter paper. Do not replenish the solution in the filter funnel.

6.3.1   Preparation of the polarimeter

6.3.1.1   Quartz plate

Zero the polarimeter on air with the cell compartment empty. Record the reading of the quartz plate.

No temperature measurement is needed when using a **saccharimeter**. However, the difference between the quartz plate reading and the certified quartz plate value must subtracted from any subsequent sample readings.

When using a **polarimeter** and if the quartz plate is not equipped with a temperature sensor and the temperature of the quartz plate is other than $20.0 \pm 0.5°C$, a temperature correction must be applied using Equation 1 (applicable to 589 nm).

$$Q_R = Q_T - 0.000144 \times (T\text{-}20) \times Q_{20} \qquad \text{(Equation 1)}$$

where T $\equiv$ temperature of the quartz plate in °C
Q_T $\equiv$ quartz plate reading at temperature T
Q_R $\equiv$ quartz plate reading corrected to 20.0°C
Q_{20} $\equiv$ certified quartz plate value at 20.0°C

The instrument must be calibrated to show the correct adjusted value for the quartz plate

6.3.1.2   Polarising tube

Determine the optical rotation of the polarising tube by filling it with water (at 20.0°C if the polarising tube is not equipped with a temperature sensor). If the reading is not 00.00°Z do the following:

- clean the polarising tube thoroughly
- adjust the side glass ends, or
- correct the final reading by subtracting this water blank reading.

6.3.2   Reading of the sample (filtrate)

Pour the filtrate into the pol tube using three portions to ensure complete displacement of the previous solution. Record the reading once it stabilizes. If the pol tube is not equipped with a temperature sensor the reading must be taken at 20.0°C.

7. Calculations

7.1   Dilution factor

$$\text{Dilution factor} = \frac{\text{mass final solution (g)}}{\text{mass sample (g)}}$$

7.2   Brix

Correct the refractometer reading for the water blank.

7.3   Pol

Correct the polarisation reading for the water blank and the quartz plate difference when using a saccharimeter. For *C-molasses, C-massecuite* and *C-magma* multiplying the polarimeter reading by 2 to allow for the second dilution.

Adjust the polarisation value to the Brix of the diluted solution at 20.0°C to obtain the pol of the sample according to the Schmitz formula indicated in Equation 2.

$$\text{pol} = \frac{\text{polarimeter reading}}{\left(0.0000576 \times \text{Brix}^2 + 0.014752 \times \text{Brix} + 3.83545\right)} \qquad \text{(Equation 2)}$$

7.4   Apparent purity

$$\text{Apparent purity (\%)} \quad = \frac{\text{Pol}}{\text{Brix}} \times 100$$

8. Expression of results

Report Brix in °Bx to two decimal places.
Report pol in °Z to two decimal places.
Report the apparent purity as a percentage to one decimal place.

9. Precision

The tolerance for C-molasses associated with the Brix analysis is ± 0.45°Bx and with the pol analysis is ± 0.2°Z.

10. Example

When using a polarimeter:

Instrument reading on air	=	0.00°Z
Quartz plate value at 20.0°C	=	50.00°Z
Quartz plate reading	=	50.03°Z
Quartz plate temperature	=	23.6°C
Quartz plate at 20.0°C	=	50.00°Z (Equation 1)
Water blank	=	0.00°Z

Dilution factor:

Mass of molasses in stock solution	=	49.89 g
Mass of total solution	=	250.02 g
Dilution factor	=	5.01

Brix at 20.0°C:

Water blank	=	0.00°Bx
Refractometer reading at 20.0°C	=	16.45°
Brix of the solution at 20.0°C	=	16.45°Bx
Multiply by the dilution factor	=	16.45° × 5.01
Brix of the molasses at 20.0°C	=	82.42°Bx

Pol at 20.0°C:

Polarimeter reading at 20.0°C	=	13.05°
Polarimeter reading × 2	=	26.10°
Brix or the solution at 20.0°C	=	16.45°Bx
Pol of the solution at 20.0°C	=	6.40°Z	(Equation 2)

Multiply by the dilution factor	=	6.40°Z × 5.01
Pol of the molasses at 20.0°C	=	32.06°Z

Apparent purity:

Apparent purity   = $\dfrac{32.06°Z}{82.42°Bx} \times 100$

          =   38.9%

11. References

ICUMSA (1994). Determination of the polarisation of raw sugar by polarimetry. *ICUMSA Methods Book*, GS1/2/3-1.

SASTA (1985). *Laboratory Manual for South African Sugar Factories.* 3rd Edition: 297 - 299, 300 - 303.

SMRI (1997). Determination of the polarisation (pol) of molasses. *SMRI Test Methods*, TM043.

SMRI (1997). Determination of the refractometer Brix in molasses. *SMRI Test Methods*, TM007.