Method 5.1 – Syrup and remelt: Brix, pol and purity

1. Rationale

This method is applicable to factory syrups and remelt and may be used to obtain data for factory control purposes. The method refers to Method 11.2 for the analysis of lead sub-acetate. All measurements must be made at 20.0 ± 0.1°C.

2. Principle

The well-mixed sample is diluted (1+3 or 1:4) 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 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 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 ± 0.5°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 baths** (optional)

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**: 50 cm^3

4.12 **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 [$Pb(OAc)_2 \cdot 3H_2O$], also called basic lead acetate, is poisonous and will accumulate in the human body. Direct contact through the skin, inhalation (powder dust) or swallowing must 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 100.00 ± 0.05 g of the sample into a 500 cm^3 conical flask. Weigh 300.00 ± 0.05 g distilled water into the same flask to bring the total mass to 400.00 ± 0.10 g. Record these masses to calculate the dilution factor. Stopper the flask and mix thoroughly on the sample shaker.

6.2 Brix determination

Pipette 50 cm^3 of the well-mixed stock solution and transfer to the 250 cm^3 Schott bottle. Weigh 1 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 cm^3 of filtrate and collect about 20 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.

6.2.1 Reading of the sample (filtrate)

Zero the refractometer using distilled water. If the reading is not 0.00°Bx at 20.0°C, record the value as the water blank.

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

Pipette 150 cm^3 of the stock solution and transfer to a 250 cm^3 Schott bottle. Weigh 1.5 g of the dry lead sub-acetate powder while wearing a dust mask and gloves and add to the Schott bottle. Shake vigorously and allow the solution to stand for at least 5 minutes to permit reaction and 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 15 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**. 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 ± 0.5°C, a temperature correction must be applied using Equation 1 (applicable to 589 nm).

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

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 polarising tube using three portions to ensure complete displacement of the previous solution. Record the reading once it stabilizes. If the polarising tube is not equipped with a temperature sensor the reading must be made 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 for the quartz plate difference when using a saccharimeter. Adjust the resulting polarisation value to the Brix of the diluted solution at 20.0°C to obtain the pol according to the Schmitz formula indicated in Equation 2.

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

Calculate the final pol of the sample by multiplying with the dilution factor.

7.4 Apparent purity

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

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. Example

When using a polarimeter:

Instrument 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 syrup in stock solution	=	100.89 g	
Mass of total solution	=	400.02 g	
Dilution factor	=	3.97	

Brix at 20.0°C:

Water blank	=	0.00°Bx	
Refractometer reading at 20.0°C	=	17.66°	
Brix of the solution at 20.0°C	=	17.66°Bx	
Multiply by the dilution factor	=	17.66° × 3.97	
Brix of the syrup at 20.0°C	=	70.11°Bx	

Pol at 20.0°C:

Polarimeter reading at 20.0°C	=	63.40°	
Brix of the solution at 20.0°C	=	17.66°Bx	
Pol of the solution at 20.0°C	=	15.41°Z	(Equation 2)
Multiply by the dilution factor	=	15.41°Z × 3.97	
Pol of the syrup at 20.0°C	=	61.18°Z	

Apparent purity:

Apparent purity	=	$\frac{61.18°Z}{70.66°Bx} \times 100$	
	=	86.6%	

10. 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: 278 - 279.

SMRI (1997). Determination of the polarisation (pol) of syrup. *SMRI Test Methods*, TM045.

SMRI (1997). Determination of the refractometer Brix in syrup. *SMRI Test Methods*, TM008.