Method 7.1 – Raw sugar: pol

1. [Scope]

T[his is ap]plicable to all raw sugars and may be used to obtain data for factory co[ntrol purpose]s and for sugar payment. The method refers to Method 11.2 for the dete[rminati]on of the total and basic lead content of lead sub-acetate.

2. Principle

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 a specified temperature. For solutions containing only pure sucrose in water, pol is a measure of the concentration of sucrose present; 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 raw sugar, 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 recommended that the measurement be taken at 20.0°C.

3. Apparatus

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

3.2   **Polarising tube**: 200 mm

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

3.3   **Quartz control plate**: ± 100°Z, officially certified at 20.0 ± 0.5°C to two decimal places

3.4   **Analytical balance** readable to 0.001 g

3.5   **Pol dish**: stainless steel

3.6   **Pol flasks** (calibrated): 100 cm^3

3.7   **Filtration apparatus**

funnel: 100 mm φ stemless
beakers: 3 × 150 cm^3
watch/cover glass: 100 mm φ

3.8   **Water baths** (optional)

If the polarising tube is not equipped with a temperature sensor a thermostatically controlled circulating water bath connected to the tube and maintained at $20.0 \pm 0.1°C$ is absolutely necessary.

If the polarising tube is not equipped with a temperature sensor a sample water bath at $20.0 \pm 0.1°C$ is also needed.

3.9   **Water bath**: boiling

3.10   **Conical flask**: 2 litre

3.11   **Filter paper**: Whatman No. 91, S&S 3000 or equivalent (185 mm ϕ)

3.12   **Buchner funnel**: (185 mm ϕ)

3.13   **Buchner flask**: 2 litre

3.14   **Filter paper**: Whatman No. 540 or equivalent (185 mm ϕ)

3.15   **Container for lead solution**: airtight with tap

3.16   **Dropper or Pasteur pipette**

4. Reagents

4.1   **Acetic acid** (concentrated or glacial)

Acetic acid (CH₃COOH) is a corrosive acid and contact with the skin, eyes and through inhalation must be avoided. Work in a fume cupboard while wearing gloves and safety glasses.

4.2   **Nitrogen gas**

4.3   **Lead sub-acetate solution**

Lead sub-acetate trihydrate [Pb(OAc)₂· 3H₂O] 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. Wear a dust mask, safety glasses and gloves during use.

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 (NO₃)	< 0.003%
Copper (Cu)	< 0.002%
Substances not precipitated by H₂S (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.

Weigh 560 g of lead sub-acetate trihydrate powder into a 2 litre conical flask and add 1 litre of freshly boiled distilled water. Boil for 30 minutes in a fume cupboard, cover the beaker and stand overnight. Decant the supernatant liquor and filter through a Whatman No. 540 or equivalent filter paper using a Buchner funnel and flask. Use vacuum to aid filtration if necessary. Dilute the filtrate with recently boiled distilled water to a density of 1.24 ± 0.01 g/cm^3 or a total lead content of 24.4 ± 1.0 g PbO/100 cm^3. The basic lead content must be between 9.5 and 10.5 g PbO/100 cm^3 (refer to Method 11.2).

If the lead content is lower than the required range, boil the solution to evaporate some of the water. If the lead content is higher than the required range, reduce the concentration by adding glacial acetic acid. After adjustment determine both the total lead and basic lead contents again. Keep the solution in an airtight container with an automatic dispenser to avoid contact with carbon dioxide in the air. Flush the container with nitrogen gas before closing to avoid the presence of carbon dioxide.

4.4   Ether

Diethyl ether [(CH$_3$)$_2$O] is highly flammable, toxic and will cause fatigue and unconsciousness when the fumes are inhaled. Ether will also form explosive peroxides when exposed to air and sunlight for long periods. Inspect ether containers for leaks to avoid evaporation.

5. Procedure

5.1   Preparation of the sample solution

Weigh 26.000 ± 0.002 g of the sugar as rapidly as possible accurately in a pol dish. Transfer the sugar to a 100 cm^3 calibrated pol flask by washing with distilled water to a volume not exceeding 70 cm^3 and dissolve the sugar by swirling. Add distilled water until the bulb of the flask is full and mix by gentle swirling. Add basic lead acetate solution according to the expected polarisation of the raw sugar: use 1.0 cm^3 for an expected pol of 99.00°Z and below and use 0.5 cm^3 for an expected pol of more than 99.00°Z. Add water to just below the mark.

Add 1 drop of ether to clear the meniscus. (If the polarising tube is not equipped with a temperature sensor, adjust the temperature of the sugar solution to 20.0°C by standing the flask in a water bath for at least 30 minutes. Include a beaker with distilled water.) Dry the inside wall of the neck of the flask with rolled filter paper. Using a dropper or Pasteur pipette, make the solution to the mark with the 20.0°C distilled water against a well-lit background. If fine bagasse or fiber particles are present, flick the side of the neck so that the true position of the meniscus can be seen. Remove any further drops from the neck of the flask. Mix the flask contents thoroughly.

5.2   Sample Filtration

Filter the solution through the fluted filter paper supported in a funnel which rests directly on a 150 cm^3 beaker. Seal the funnel with a watch glass to minimise evaporation. Discard the first 15 cm^3 of the filtrate and collect about 60 cm^3 of the filtrate in another clean, dry beaker to avoid contamination. Do not allow the filtrate to touch the bottom of the funnel or filter paper. Do not replenish the solution in the filter funnel.

5.3   Preparation of the polarimeter

5.3.1   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** with a visible light source (589 nm) 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).

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

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 **polarimeter** must be calibrated to show the correct adjusted value for the quartz plate.

5.3.2 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.

5.4 Reading of the sample (filtrate)

Pour the filtrate into the polarising tube using three portions to ensure complete displacement of the previous solution in the tube. Record the reading once it stabilizes. If the polarising tube is not equipped with a temperature sensor, record the temperature of the filtrate.

6. Calculations

The pol of the sugar is the polarisation reading to the nearest $\pm 0.01°Z$ at $20.0 \pm 0.1°C$ corrected for the water blank and the quartz plate difference when using a saccharimeter. If the temperature of the sample is other than $20.0 \pm 0.1°C$ correct the reading for the effect of this temperature according to Equation 2 (applicable to 589 nm).

$$P_{20} = \frac{\text{pol reading}}{1 - 0.000455 \times (T - 20.0)} \qquad \text{(Equation 2)}$$

where
P_{20}	$\equiv$	polarisation reading at 20.0°C
T	$\equiv$	temperature of solution in °C
P_T	$\equiv$	corrected polarisation reading at temperature T

Finally adjust this value for the pol flask calibrated volume using Equation 3.

$$\text{Pol (°Z)} = P_{20} - (100 - \text{pol flask volume}) \text{ cm}^3 \qquad \text{(Equation 3)}$$

where
P_{20}	$\equiv$	polarisation reading at 20.0°C

Report results in °Z to two decimal places.

7. Example

When using a polarimeter:

Instrument reading on air	=	0.00°Z	
Quartz plate value at 20.0°C	=	99.97°Z	(certified)
Quartz plate reading	=	100.00°Z	
Quartz plate temperature	=	23.1°C	
Quartz plate at 20.0°C	=	99.96°Z	(Equation 1)
Quartz plate difference	=	-0.01°Z	

The instrument therefore needs to be calibrated to show the correct quartz plate reading.

Water blank	=	0.00°Z	
Polarimeter reading	=	98.21°Z	
Solution temperature	=	22.7°C	
Pol value at 20.0°C	=	98.33°Z	(Equation 2)
Calibrated pol flask volume	=	100.03 cm^3	
Final pol	=	98.36°Z	(Equation 3)

8. Precision

The tolerance associated with the analysis is ± 0.05°Z.

9. References

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

Mellet P (1987). Laboratory control procedures for South African sugar factories. *SMRI Technical Note,* No. 1473.

Mellet P, Lionnet GRE, Kimmerling ZJ and Bennett PJ (1982). Standards for analytical precision of sugar and molasses analyses. *Proc S Afr Sug Technol Ass*, **56**: 55-57.

SASTA (1985). *Laboratory Manual for South African Sugar Factories.* 3rd Edition: 317.

SMRI (2004). Determination of the polarisation (pol) of raw sugar. *SMRI Test Methods*, TM044.

van Staden LF, Naidu C and Simpson R (1999). Updated methods and recommendations for mill laboratories. *SMRI Technical Note,* No. 1817.