



Method 7.2 – Raw sugar: NIR pol

1. Rationale

This method is applicable to all raw sugars and may be used to obtain data for factory control purposes and for sugar payment. This method eliminates the use of lead subacetate clarification. Because the sample solution colour will be higher in the absence of lead clarification a polarimeter operating at a near infrared (NIR) wavelength must be used.

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 NIR light source (*e.g.* 882 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 cm³

3.7 Filtration apparatus

funnel: 100 mm ϕ stemless

beakers: 3 × 150 cm³

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 $20.0 \pm 0.1^\circ\text{C}$ is absolutely necessary.

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

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

4. Reagents

4.1 Celite Filtercel

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

4.2 Ether

Diethyl ether $[(\text{CH}_3)_2\text{O}]$ 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 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 the 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

Transfer 3.5 g Celite Filtercel filter aid to a fluted filter paper supported in the funnel which rests directly on a 150 cm^3 beaker and filter the solution. 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 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 be 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 $20.0 \pm 0.5^\circ\text{C}$, a temperature correction must be applied using Equation 1 (applicable to NIR wavelengths).

$$Q_R = Q_T - 0.000139 \times (T - 20) \times Q_{20} \quad (\text{Equation 1})$$

where	T	≡	temperature of the quartz plate in °C
	Q _T	≡	quartz plate reading at temperature T
	Q _R	≡	quartz plate reading corrected to 20.0°C
	Q ₂₀	≡	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 $20.0 \pm 0.1^\circ\text{C}$ 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
- adjust the side glass ends, or
- correct the final reading by subtracting the 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 $\pm 0.01^\circ\text{Z}$ at $20.0 \pm 0.1^\circ\text{C}$ corrected for the water blank and the quartz plate difference when using a saccharimeter. If the temperature of the sample is other than $20.0 \pm 0.1^\circ\text{C}$ correct the reading for the effect of this temperature according to Equation 2 (applicable to NIR wavelengths).

$$P_{20} = \frac{\text{pol reading}}{1 - 0.000478 \times (T - 20.0)} \quad (\text{Equation 2})$$

where	P ₂₀	≡	polarisation reading at 20.0°C
	T	≡	temperature of solution in °C
	P _T	≡	corrected polarisation reading at temperature T

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

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

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 reading	=	0.00°Z	
Polarimeter reading	=	98.21°Z	
Solution temperature	=	22.7°C	
Pol value at 20.0°C	=	98.34°Z	(Equation 2)
Calibrated pol flask volume	=	100.03 cm ³	
Final pol	=	98.37°Z	(Equation 3)

8. Precision

The tolerance associated with the analysis is $\pm 0.05^{\circ}\text{Z}$.

9. References

ICUMSA (2003). Polarisation of raw sugar without wet lead clarification. *ICUMSA Methods Book*, GS1/2/3-2.

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

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.