Method 3.2 – Juices: Brix, NIR pol and purity

1. Rationale

This method is applicable to factory juices and may be used to obtain data for factory control purposes. This method eliminates the use of lead sub-acetate 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. All measurements must be made at 20.0 ± 0.1°C.

2. Principle

The well-mixed juice is filtered with the help of a filter aid and used to determine the pol and Brix 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 a specified 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 juices, 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 NIR light source (*e.g.* 882 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 **Schott bottle**: 250 cm^3

4.6 **Filtration apparatus**

Either one of the following:

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

OR

pressurized filtration unit (*ca.* 1 bar pressure)
glass beakers: 3 × 150 cm^3

4.7 **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 temperature to 20.0 ± 0.1°C is then also needed.

4.8 **Pipette**: 50 cm^3

4.9 **Filter paper**

Whatman No. 6, Postslip medium white w/s or equivalent: 185 mm ϕ (gravity filtration) or 155 mm ϕ (pressure filtration)
S&S GF50 glass fibre prefilter: 155 mm ϕ (pressure filtration only)

5. Reagents

5.1 **Celite Filtercel**

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

6. Procedure

6.1 Sample Filtration

6.1.1 Pressurized filtration

Add 6 g Celite Filtercel filter aid to 200 cm^3 of the sample in a 250 cm^3 Schott bottle. Shake until the filter aid is completely wet. Pressure-filter through the filter paper, discarding the first 25 cm^3 and collecting about 100 cm^3 of the rest of the filtrate that is clear of foam.

Repeat this filtration with fresh sample and fresh Celite using a new filter paper to prevent contamination from the previous sample in the filtration unit. Only use the second filtrate for the analysis. If the sample filtration takes longer than 30 seconds due to the nature of the sample, use a glass fibre prefilter on top of the filter paper to aid filtration.

6.1.2 Gravity filtration

Add 3 g Celite Filtercel filter aid to 100 cm^3 of the sample in a 150 cm^3 beaker, mix until the filter aid is thoroughly wet and filter the mixture through the fluted filter paper supported in a funnel resting directly on a beaker. Seal the funnel with a watch glass to minimise evaporation. Discard the first 25 cm^3 of filtrate and collect the remainder 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 Brix determination

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

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 be subtracted from any subsequent sample readings.

When using a **polarimeter** where 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 NIR wavelengths).

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

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.

6.3.1.2 Polarising tube

Determine the optical rotation of the polarising tube by filling it with distilled water (at 20.0 ± 0.1°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.

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 ± 0.1°C.

7. Calculations

7.1 Brix

Correct the refractometer reading for the water blank.

7.2 Pol

Correct the polarisation reading for the water blank and the quartz plate difference when using a saccharimeter. Adjust the resulting polarisation value to the Brix of the sample at 20.0°C to obtain the pol according to the Schmitz formula indicated in Equation 2.

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

7.3 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. Precision

The tolerance associated with the Brix analysis is ± 0.05°Bx and with the pol analysis is ± 0.05°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	

Brix at 20.0°C:

Instrument reading on water	=	0.00°	
Refractometer reading at 20.0°C	=	10.17°	
Brix of the juice at 20.0°C	=	10.17°Bx	

Pol at 20.0°C:

Polarimeter reading at 20.0°C	=	35.59°	
Pol of the juice at 20.0°C	=	8.92°Z	(Equation 2)

Apparent purity:

$$\text{Apparent purity} = \frac{8.92°\text{Z}}{10.17°\text{Bx}} \times 100$$

$$= 87.7\%$$

11. References

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SMRI (1997). Determination of the refractometer Brix in juice. *SMRI Test Methods*, TM005.