



Method 5.6 – Syrup: gums

1. Rationale

This method is applicable to factory syrups and is an empirical method based on the gravimetric determination of gums (total polysaccharides).

2. Principle

The mass of gums is measured gravimetrically after precipitation of all the polysaccharides (gums) with acidified alcohol. Included in the precipitate is natural polysaccharides such as starch, deterioration polysaccharides such as dextran, other organics such as waxes, some inorganics (ash) and protein.

3. Apparatus

- 3.1 **Analytical balance** readable to 0.0001 g
- 3.2 **Magnetic stirrer** with follower
- 3.3 **Beakers:** 250 cm³, two per sample
- 3.4 **Light duty balance** readable to 0.01 g
- 3.5 **Centrifuge** operating at 5000 ± 200 rpm and centrifuge tubes (50 cm³ capacity)
- 3.6 **Screen:** 100 mm ϕ × 80 mm, pore opening 75 μ m
- 3.7 **Gooch crucibles:** 36-40 mm, top ϕ
- 3.8 **Buchner flasks** to accommodate the crucibles
- 3.9 **Rubber sleeves** for use with the Buchner flasks and crucibles
- 3.10 **Drying oven** operating at 105 ± 5°C
- 3.11 **Muffle furnace** operating at 650 ± 25°C
- 3.12 **Desiccator** with self-indicating silica gel
- 3.13 **Watch glass:** 100 mm ϕ
- 3.14 **Glass rod**
- 3.15 **Measuring cylinders:** 25 cm³, 200 cm³
- 3.16 **Refractometer** operating at 20.0 ± 0.1°C

4. Reagents

4.1 Ethanol

Ethanol ($\text{CH}_3\text{CH}_2\text{OH}$, absolute alcohol) is a flammable liquid and is toxic when swallowed.

4.2 Hydrochloric acid (1:1)

Hydrochloric acid (HCl , 32%) is a corrosive acid and contact with the skin, eyes and through inhalation must be avoided. Use in a fume cupboard while wearing gloves and safety glasses.

Carefully add 100 cm³ of concentrated hydrochloric acid to 100 cm³ of distilled water. Always add the acid to the water, never the other way around. The dilution is exothermic and the solution will heat.

4.3 Acidified ethanol

Add 200 cm³ of the diluted hydrochloric acid (1:1) to 1 000 cm³ of ethanol.

4.4 Fibroxcel 10 filter aid, AEB Africa

4.5 Glass fiber pre-filter: 25 - 30 mm ϕ

5. Procedure

5.1 Gooch crucible preparation

Weigh about 6.2 g of Fibroxcel 10, add 250 cm³ distilled water and mix thoroughly for 45 minutes. This quantity of slurry is enough to prepare 10 Gooch crucibles; in practice prepare just enough slurry for immediate use. Place a crucible with a glass fiber pre-filter in a Buchner flask and add about 25 cm³ of the well-mixed slurry. Stand for about 10 seconds then apply vacuum. With the aid of the glass rod, gently press the filter pad down to form a smooth mat. Rinse the filter pad with 4 \times 5 cm³ portions of acidified alcohol followed by 4 \times 5 cm³ portions of distilled water and dry at 105 \pm 5°C for one hour. Ignite the crucible and content in the muffle furnace for 30 minutes at 650 \pm 25°C and cool in the desiccator for 1 hour.

5.2 Sample preparation

Weigh 25.00 \pm 0.01 g of the well mixed sample into a conical flask. Add water to 100.00 \pm 0.01 g and mix well. Measure the Brix of the solution in a refractometer at 20.0 \pm 0.1°C.

Weigh 25.00 \pm 0.01 g of the well mixed sample into a conical flask. Add water to 100.00 \pm 0.01 g and mix well. Transfer the solution to a centrifuge tube and centrifuge for 5 minutes at 5 000 \pm 200 rpm.

Weigh 30.0 \pm 0.1 g of supernatant liquid into a beaker. While stirring the liquor, slowly add 150 cm³ acidified alcohol. Remove the magnetic stirrer bar from the liquor and wash well with a small portion of the acidified alcohol to return any precipitate adhering to it to the beaker. Cover with a watch glass and allow to stand for 16 \pm 2 hours.

5.3 Sample filtration

Decant the supernatant liquor from the sample through the prepared crucible, ensuring that the filter pad remains firm. Transfer the precipitate quantitatively to the crucible, washing the beaker 5 times with acidified ethanol. Wash the precipitate with absolute

alcohol ($2 \times 5 \text{ cm}^3$). Wipe the outside of the crucible, dry in the oven for 3 hours and allow to cool in the desiccator for 1 hour. Weigh the crucible on the analytical balance and record the mass. Ignite the crucible and content in the muffle furnace at $650 \pm 25^\circ\text{C}$ for 30 minutes to incinerate the gums. Return the crucible to the desiccator and cool for 1 hour. Reweigh the crucible on the analytical balance and record the mass.

6. Calculations

$$\text{Total solids in supernatant liquid (g)} = \text{mass of liquid (g)} \times \frac{\text{Brix (}^\circ\text{Bx)}}{100}$$

$$\text{Gums (mg/kg)} = \frac{(M_1 - M_2) \times 1000}{\text{Total solids (g)} \div 1000}$$

where $M_1 \equiv$ Mass of crucible before incineration (g)

$M_2 \equiv$ Mass of crucible after incineration (g)

Report to the nearest 50 ppm.

7. Example

Brix of the solution at 20.0°C	=	17.84°Bx
Total solids in 30 g clear liquor	=	$0.3 \times 17.84^\circ\text{Bx}$
	=	5.35 g
Mass of crucible after drying	=	21.5875 g
Mass of crucible after ignition	=	21.5710 g
Mass of gums	=	0.0165 g
Gums	=	$\frac{0.0165 \text{ g} \times 1000}{5.35 \text{ g} \div 1000}$
	=	3 088 ppm

Report as 3 050 ppm

8. References

SASTA (1985). *Laboratory Manual for South African Sugar Factories*. 3rd Edition: 285 - 287.

SMRI (2004). Determination of the gums (total polysaccharides) in raw sugar without using asbestos. *SMRI Test Methods*, TM018.

SMRI (2004). Determination of the gums (total polysaccharides) in syrup. *SMRI Test Methods*, TM019.