Method 7.7 – Raw sugar: gums

1. Rationale

This method is applicable to all raw sugars 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 are natural polysaccharides such as starch, deterioration polysaccharides such as dextran, other organic compounds such as waxes, some inorganic compounds (ash) and protein.

3. Apparatus

3.1 **Analytical balance** readable to 0.0001 g

3.2 **Magnetic stirrer** with stirrer bar

3.3 **Beakers**: 100, 250 cm^3

3.4 **Light duty balance** readable to 0.01 g

3.5 **Centrifuge** operating at 5000 ± 200 rpm and centrifuge tubes (50 cm^3 capacity)

3.6 **Screen**: 100 mm ϕ× 80 mm, pore opening 75 µm

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 and 200 cm^3

3.16 **Refractometer** operating at 20.0 ± 0.1°C

4. Reagents

4.1 Ethanol

Ethanol (CH_3CH_2OH, absolute alcohol) is a flammable liquid and is toxic when swallowed. Wear safety glasses to avoid contact with the eyes.

4.2 **Hydrochloric acid** (1:1)

Hydrochloric acid (HCl) 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 cm^3 of concentrated hydrochloric acid (HCl, 32%) to 100 cm^3 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 cm^3 of the diluted hydrochloric acid (1:1) to 1000 cm^3 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 in a 250 cm^3 beaker, add 250 cm^3 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 cm^3 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 × 5 cm^3 portions of acidified alcohol followed by 4 × 5 cm^3 portions of distilled water and dry at 105 ± 5°C for one hour. Ignite the crucible and content in the muffle furnace for 30 minutes at 650 ± 25°C and cool in the desiccator for 1 hour.

5.2 Sample preparation

Weigh 30.0 ± 0.1 g sugar in a 100 cm^3 beaker. Dissolve in 30 cm^3 warm water. Filter the sugar solution through the screen into a centrifuge tube. Centrifuge the solution for 5 minutes at 5 000 ± 200 rpm. Measure the refractometer Brix of the supernatant liquid at 20.0°C.

Weigh 30.0 ± 0.1 g of supernatant liquid into a 250 cm^3 beaker. While stirring the liquor, slowly add 150 cm^3 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 ± 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 five times with acidified ethanol. Wash the precipitate with absolute alcohol (2 × 5 cm^3). Wipe the outside of the crucible, dry in the oven for 3 hours and allow

cooling 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 ± 25°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 ≡ Mass of crucible before incineration (g)
M_2 ≡ Mass of crucible after incineration (g)

Report to the nearest 50 mg/kg.

7. Example

Refractometer reading of supernatant at 20.0°C = 51.1°Bx

Total solids in 30 g clear liquor = 0.3 × 51.1°Bx
= 15.33 g

Mass of crucible before ignition = 20.4475 g
Mass of crucible after ignition = 20.4380 g
Mass of gums = 0.0095 g

$$\text{Gums} = \frac{9.5 \text{ mg}}{0.01533 \text{ kg}}$$
= 620 mg/kg

Report as 600 mg/kg

8. Precision

The tolerance associated with the analysis is ± 150 mg/kg.

9. References

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: 329 - 330.

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