



Method 8.9 – Refined sugar: insoluble solids

1. Rationale

This method is applicable to all refined and white sugars and determines the mass of the total insoluble solids in a sample by filtering a solution of the sample through a 8.0 μm pore size membrane.

2. Principle

The sugar sample is dissolved in hot water and filtered through a membrane filter. The membrane filter and the retained insoluble matter are thoroughly washed, dried and weighed. The insoluble matter is calculated from the increase in mass of the membrane filter.

3. Apparatus

- 3.1 **Filtration apparatus:** a magnetic filtration unit fitted to a Buchner flask
- 3.2 **Erlenmeyer flask:** 2 000 cm^3
- 3.3 **Magnetic stirrer** and stirrer bar
- 3.4 **Tweezers**
- 3.5 **Petri dish** (plastic)
- 3.6 **Drying oven** maintained at $63 \pm 1^\circ\text{C}$
- 3.7 **Analytical balance** readable to 0.0001 g
- 3.8 **Top pan balance** readable to 1 g
- 3.9 **Desiccator** with self-indicating silica gel
- 3.10 **Stopwatch**
- 3.11 **Membrane filter** (cellulose acetate): 47 mm ϕ , 8.0 μm pore size

4. Procedure

4.1 Preparation of prefilter and membrane filter

Wash the membrane filter by immersion in boiling distilled water for 6 minutes, drain the excess water from the membrane and transfer to a petri dish using tweezers. Place the petri dish in the oven at $63 \pm 1^\circ\text{C}$ for 1 hour and allow cooling in a desiccator for half an hour.

4.2 Sample preparation

Riffle 1 000 g of well mixed sugar into the 2 000 cm³ Erlenmeyer flask, add hot distilled water (95°C) to a volume of about 1600 cm³ and dissolve. Add distilled water to a volume of about 1 800 cm³ and heat the solution to a temperature of approximately 90 to 95°C (just before it starts to boil).

4.3 Filtration

Weigh the membrane filter on an analytical balance and record the mass. Assemble the filtration apparatus by putting the membrane filter on the disc holder and wet with distilled water. Filter the hot sugar solution through the assembled filter and record the total time taken.

Once all the sugar solution has passed through the filter wash the membranes with 10 × 100 cm³ portions of hot (95°C) distilled water, allowing the water to drain completely between washings. Wash the membrane filter with a wash bottle containing hot distilled water by lifting the membrane with tweezers and washing the sides and underneath thoroughly. Take care not to wash away any of the insoluble solids. Allow the funnel containing the filters to drain completely under vacuum for 1 minute.

Transfer the membrane filter containing the suspended solids to the petri dish with tweezers and place the dish, with the lid removed, in the oven (63 ± 1°C) for 1½ hours. Place the lid on the dish, remove it from the oven and place it in a desiccator for 10 minutes. Weigh the membrane filter on the analytical balance and record the mass.

5. Calculations

$$\text{Insoluble matter (mg/kg)} = (M_3 - M_2) \times 1000 \times \frac{1000}{M_1}$$

where M_1	≡	mass of the sample (g)
M_2	≡	mass of the membrane filter (g)
M_3	≡	mass of the membrane and insoluble matter (g)

Report results in mg/kg to the nearest unit together with the time taken to filter the sample (minutes).

6. Example

mass of the sample	=	991.19 g
mass of the membrane filter	=	0.0805 g
mass of the membrane and insoluble matter	=	0.0846 g

$$\begin{aligned} \text{Insoluble matter (mg/kg or mg/kg)} &= (0.2140 - 0.2072) \text{ g} \times 1000 \times \frac{1000}{100.00 \text{ g}} \\ &= 4.14 \text{ mg/kg} \end{aligned}$$

Report as 4 mg/kg

7. Precision

The tolerance associated with the analysis is ± 3 mg/kg.

8. References

ICUMSA (1994). Insoluble matter in white sugar. *ICUMSA Methods Book*, GS2/3-19.

SMRI (2004). Determination of the insoluble matter of raw/golden brown sugar. *SMRI Test Methods*, TM028.