



## Method 7.13 – Raw sugar: insoluble solids

### 1. Rationale

This method is applicable to all raw and brown sugars and determines the mass of the total insoluble solids in a sample solution that does not filter through a 8.0 µm pore size membrane.

### 2. Principle

The sugar sample is dissolved in hot water and filtered through a prefilter and a membrane filter. The prefilter, 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 prefilter and the membrane filter.

### 3. Apparatus

- 3.1 **Prefilter** (glass fibre): 47 mm  $\phi$
- 3.2 **Membrane filter** (cellulose acetate): 47 mm  $\phi$ , 8.0 µm pore size
- 3.3 **Filtration apparatus**: a magnetic filtration unit fitted to a Buchner flask
- 3.4 **Conical flask**: 1 000 cm<sup>3</sup>
- 3.5 **Magnetic stirrer** and stirrer bar
- 3.6 **Tweezers**
- 3.7 **Petri dish** (plastic)
- 3.8 **Drying oven** maintained at 63 ± 1°C
- 3.9 **Analytical balance** readable to 0.0001 g
- 3.10 **Top pan balance** readable to 1 g
- 3.11 **Desiccator** with self-indicating silica gel
- 3.12 **Stopwatch**

### 4. Procedure

#### 4.1 Preparation of prefilter and membrane filter

Wash the prefilter by filtering approximately 500 cm<sup>3</sup> hot (95°C) distilled water in the filtration apparatus. Allow the filter to dry on the filtration apparatus and transfer to a clean, dry petri dish.

Wash the membrane filter by immersion in boiling distilled water for 6 minutes, drain the excess water from the membrane and transfer to the same petri dish using a tweezer. Place the petri dish in the oven at 63°C for 1 hour and allow cooling in a desiccator for half an hour.

#### 4.2 Sample preparation

Weigh  $100 \pm 10$  g of well mixed riffled sugar into the 1 000 cm<sup>3</sup> conical flask, add 400 cm<sup>3</sup> hot distilled water (95°C) and dissolve. Heat the solution to a temperature of approximately 90 to 95°C (just before it starts to boil).

#### 4.3 Filtration

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

Once all the sugar solution has passed through the filters wash the membranes with  $10 \times 100$  cm<sup>3</sup> portions of hot (95°C) distilled water, allowing the water to drain completely between washings. Wash the prefilter and membrane filter with a wash bottle containing hot distilled water by lifting the prefilter and 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 prefilter and membrane filter containing the suspended solids to the petri dish with tweezers and place the dish, with the lid removed, in the oven (63°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 prefilter and 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 prefilter and membrane filter (g)  
 $M_3$  ≡ mass of the prefilter, membrane and insoluble matter (g)

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

### 6. Example

mass of sample	=	100.00 g
mass of the prefilter and membrane	=	0.2072 g
mass of the prefilter, membrane and insoluble matter	=	0.2140 g

$$\begin{aligned} \text{insoluble solids} &= (0.2140 - 0.2072) \text{ g} \times 1000 \times \frac{1000}{100.00 \text{ g}} \\ &= 68 \text{ mg/kg} \end{aligned}$$

## **7. Precision**

The tolerance associated with the analysis is  $\pm 15$  mg/kg between 0 - 200 mg/kg and  $\pm 20$  mg/kg at higher levels.

## **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.