



Method 6.2 – C-molasses: moisture by vacuum oven

1. Rationale

This method is applicable to C-molasses and is used to determine the dry solids or moisture content of a molasses sample as an alternative to the Karl Fisher titration method (Method 6.3).

2. Principle

A sample of molasses is mixed with sand and dried in a vacuum oven at 65°C. Under these conditions the maximum surface area of molasses is presented for evaporation whilst degradative reactions are minimised. The mass difference in the sample before and after drying is the moisture expressed as percentage on sample.

3. Definitions

3.1 Moisture

Water represents the primary heat-volatile liquid in molasses and is referred to as moisture or water content.

3.2 Dry solids

Dry solids are the material remaining after drying a product to constant mass or for a specific period. The mass of dry solids can also be found by deducting the mass of the moisture as determined in a specific manner from the mass of the product.

4. Apparatus

- 4.1 **Vacuum drying oven** thermostatically controlled at $65 \pm 2^\circ\text{C}$
- 4.2 **Analytical balance** readable to 0.1 mg
- 4.3 **Moisture dishes:** aluminium with lids, 80 mm ϕ \times 10 mm depth
- 4.4 **Stirring rods** with flattened ends
- 4.5 **Desiccator** containing self indicating silica gel
- 4.6 **Oven** controlled at $105 \pm 1^\circ\text{C}$
- 4.7 **Balance** readable to 0.01 g
- 4.8 **Conical flasks:** 500 cm³ wide neck with rubber stoppers to fit
- 4.9 **Mechanical shaker**

5. Reagents

5.1 Sand: particle size between 150 and 360 μm and purified with acid

6. Procedure

6.1 Preparation of a stock solution

Weigh approximately 50 g of the well mixed sample in a 500 cm^3 conical flask. Add distilled water to a total mass of 250 g. Mix on the mechanical shaker until a homogenous solution is obtained.

6.2 Determination of the dry solids

Weigh 40 g sand into a moisture dish. Place the open dish, lid and a stirring rod in the oven at $105 \pm 1^\circ\text{C}$ and dry for 2 hours. Remove and cool for 1 hour in the desiccator. Weigh the moisture dish containing sand together with the stirring rod and record the mass. Pipette 5 cm^3 of the well-mixed stock solution into the dish and close the dish firmly without delay. Reweigh the dish plus lid plus rod and record the mass.

Open the dish and using the rod, mix the molasses and sand very carefully until each grain of sand is well coated with molasses. Place the rod inside the dish. Place the open dish and its lid in the vacuum oven. Close the oven and apply vacuum and heat. Open the dry air bleed to give a flow of approximately 10 cm^3 per minute (at atmospheric pressure). Dry the sample in the oven at approximately $65 \pm 2^\circ\text{C}$ and approximately 13 kPa absolute pressure to constant mass (approximately 24 hours). Slowly release the vacuum, replace the lid on the dish, remove from the oven and place in a desiccator to cool to ambient temperature. Weigh and record the mass.

7. Calculations

$$\text{Dry solids \% molasses} = \frac{(M_3 - M_1)}{(M_2 - M_1)} \times 5 \times 100$$

where M_1	\equiv	dish, sand and rod (g)
M_2	\equiv	dish, sand, rod and molasses before drying (g)
M_3	\equiv	dish, sand, rod and molasses after drying (g)
5	\equiv	dilution factor (total mass/mass molasses)

Express results as percentage to 2 decimal places.

8. Example

mass of dish, sand and rod	=	107.6352 g
mass before drying	=	112.8598 g
mass of sample	=	112.8598 g - 107.6352 g
	=	5.2246 g
mass after drying	=	108.4852 g
mass of dry solids	=	0.8500 g

$$\begin{aligned} \text{dilution factor} &= \frac{252.59 \text{ g}}{50.48 \text{ g}} \\ &= 5.00 \\ \\ \text{Dry solids \% molasses} &= \frac{(108.4852 \text{ g} - 107.6352 \text{ g})}{(112.8598 \text{ g} - 107.6352 \text{ g})} \times 5 \times 100 \\ &= 81.35\% \end{aligned}$$

9. Precision

The tolerance associated with the analysis is ± 0.2 units.

10. References

ICUMSA (1994). Moisture in speciality sugars, molasses, cane raw sugars and syrups by the Karl Fischer procedure. *ICUMSA Methods Book*, GS4/7/3-12.

SASTA (1985). *Laboratory Manual for South African Sugar Factories*. 3rd Edition: 301 - 302.

SMRI (2000). Determination of the dry substance and moisture in molasses by vacuum oven drying on sand. *SMRI Test Methods*, TM035.