

MT 185.1 WET SIEVE TEST

SCOPE

The method is suitable for the determination of the amount of non-dispersible material in formulations, that are applied as dispersions in water.

REASONS FOR THE REVISION

The objective of revision was to combine MT 185 and MT 182 into a single method. The procedure was simplified, editorial changes have been made and obsolete references have been removed. Where the same conditions are used, results obtained with MT 185.1 are equivalent to those obtained with MT 185 or MT 182. MT 185.1 supersedes MT 185 and MT 182.

OUTLINE OF METHOD

A sample of the formulation is dispersed in water and the suspension formed is transferred to a sieve and washed. The amount of the material retained on the sieve is determined by drying and weighing.

APPARATUS

Balance, with an accuracy of at least ± 0.001 g

Beaker, 250 ml

Drying device, capable of maintaining a temperature of 70 °C

Glass dish, for drying of residues

Magnetic stir bar, length approximately 2 cm

Magnetic stirrer

Flexible tubing, of 5 – 10 mm internal diameter

Sieve, 75 μm mesh size, metal wire cloth, e.g., 20 cm diameter

PROCEDURE

All operations are performed at ambient temperature (25 ± 5 °C).

Tap water is used as it is.

(a) Wetting

Weigh 10 g of the test sample (m_s) to the nearest 0.1 g into the beaker and add tap water (100 ml). Allow to stand for 60 s. Then stir with the magnetic stirrer for 5 min, making no deliberate attempt to break up any lumps (Note 1).

(b) Wet sieving

Transfer the slurry to the sieve, rinse with tap water, remove the magnetic stir bar after washing any dispersed material from the stir bar into the sieve. Wash the material on the sieve with a jet of tap water using a flexible tubing delivering 1-5 l of water per min. Keep the end of the tubing at a distance of 2 – 5 cm from the surface of the sieve. Rinse any residue from the walls of the sieve. Move the residue across the surface of the sieve and push it together. Continue the

washing until the visible quantity of residue remains constant (max. 10 min).

Pull the jet of tap water away over the edge of the sieve (Note 2).

If there is no visible residue the process can be stopped. No weighing is necessary.

If there is a visible residue transfer the residue to a tared glass dish with a jet of deionised water from a wash bottle. Dry to constant weight (Note 3) and record the weight of the sample remained on the sieve (m_r) to the nearest 0.001 g.

CALCULATION

Calculate the weight of the residues as a percentage of the sample weight

Residue remaining on the sieve = $m_r / m_s * 100$ [%]

m_s mass of the sample [g]

m_r mass of the residue [g]

REPORTING

If no residues are visible, the result is < 0.1 %.

Report residue remaining on a sieve to the nearest 0.1 %.

Note 1 The speed of rotation of the magnetic stir bar should be chosen such that a vortex is formed on the surface of the liquid. Care should be taken that the dispersion does not become aerated by over-vigorous dispersion of the sample.

Note 2 Alternative washing procedure using a peristaltic pump and recycling the washing water

APPARATUS

Beakers, 250 ml and 2000 ml

Flexible tubing, of 5 - 10 mm internal diameter to be used with the pump

Peristaltic pump, with a pumping capacity of at least 1000 ml/min

PROCEDURE

Example: Fill a 2 l beaker to the 1000 ml mark with tap water. This beaker of water will serve as the re-circulation reservoir for the wet sieving procedure. Attach the tubing to the pump. Place the intake end of the tubing into the beaker and secure (tape or clamp) to the side of the beaker. The tip of the tubing should stay submerged 3 to 4 cm below the water line. Take care that the flow rate of the pump is 1 – 5 l/min.

Wet sieving: Holding the sieve over a waste receptacle, transfer the slurry to the sieve. Gently tap the side of the sieve until the liquid portion of the slurry has passed through the screen into the waste container. Position the sieve directly over the 2 l re-circulation

reservoir containing 1 l of water. Proceed as describe under (b) Wet sieving.

Note 3

A temperature of 70 °C is recommended. If necessary, the temperature must be lowered to avoid decomposition or volatilisation of formulation components at the drying temperature.