MT 2XX Density of Solids and Liquids with Automated Systems

SCOPE

This method is intended to determine the density of solids and liquids with automated systems. Two different procedures are described depending on the physical state of the test sample.

- Gas pycnometer for solids (section A)
- Oscillating density meter for liquids (section B)

The gas pycnometer method is suitable for measuring the density of solids. It is designed to measure volume of powders, granules or any other solid objects having low vapor pressures and to permit the computation of absolute density when weight information is supplied.
The oscillating density meter method is suitable for measuring the density of liquids.

A) GAS PYCNOMETER

OUTLINE OF METHOD

The density of a solid is measured at a defined temperature with the gas comparison pycnometer. The volume of the weighed sample is measured in air or in an inert gas in a cylinder of variable calibrated volume.

REAGENTS

Reference solids, with known density, e.g. stainless steel sphere standards, used for the calibration of the instrument

APPARATUS

Gas pycnometer
Analytical balance, with an accuracy of at least ± 0.001 g

PROCEDURE

Operate, equilibrate and calibrate the gas pycnometer according to the instructions for use.

(a) Sample preparation

Weigh the empty sample chamber used for calibration and record the weight to the nearest 0.001 g. Add the solid sample, re-weigh the sample chamber and record the weight to the nearest 0.001 g. Calculate the weight of the sample. Close and seal the sample chamber.

(b) Determination

Place the sample chamber in the gas pycnometer and start the measurement according to the instructions for use when the sample has reached a defined constant temperature (e.g. 20.0 °C). The density is determined automatically by the instrument.

B) OSCILLATING DENSITY METER

OUTLINE OF METHOD

A mechanical oscillator in form of a U-tube is agitated to vibrate at its resonance frequency. When introducing a liquid sample into the tube, the oscillation frequency changes depending on the mass of the liquid. The corresponding density of the sample at the measurement temperature is automatically calculated.

REAGENTS

Reference liquid(s), with known density

APPARATUS
Oscillating density meter
Single-use syringes, e.g. 5 ml or 20 ml
If required:
Automated Sampler
Magnetic Stirrer
Sieve, e.g. with a diameter of 5 cm and a mesh size of 250 µm, recommended for removing bubbles/micro-foam by sieving the sample
Ultrasonic bath, for degassing the sample to remove bubbles/micro-foam
Vacuum desiccator, Vacuum box, e.g. made of acrylic glass, connected to a vacuum pump
Vacuum pump

PROCEDURE
Operate, equilibrate and calibrate the oscillating density meter according to the instructions for use.

(a) Sample preparation
Ensure that the sample is homogeneous.
If required, remove any air bubbles or micro-foam from the sample, ensuring that the sample is not changed, e.g. due to evaporation. Following procedures can be applied (Note 1):

(a1) Degassing by vacuum
Place a homogeneous sample in a beaker with a magnetic stirring bar in a vacuum box on a stirrer plate. Then apply, while slowly stirring the sample, a slight vacuum.

(a2) Degassing by sieving
Pour a homogeneous sample through a sieve and collect in a beaker.

(a3) Degassing by ultra-sonication
Place a homogeneous sample in a beaker in an ultrasonic bath and apply sonication.

(b) Determination
Fill the measuring cell with homogenous sample free of air bubbles, e.g. using a syringe or an automated sampler (Note 2).
After the sample has reached a defined constant temperature (e.g. 20.0 °C), start the measurement.

REPORTING
The following information should be reported:
• Test temperature to the nearest 0.1 °C
• Density in g/cm^3 to the nearest 0.001 g/cm^3
or
• Relative density () to the nearest 0.001, being the ratio of the density of the sample at 20 °C and the density of water at 4 °C (0.999972 g/cm^3)

Note 1 For clear or opalescent liquids (e.g. EC, LS, ME, SL) a sample preparation is not necessary.

Note 2 In cases where the sample material tends to develop a phase separation, the automated sampler should not be used.