INTERNATIONAL STANDARD

ISO

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Coating powders —

Part 3:
Determination of density by liquid displacement pyknometer

Poudres pour revêtement —

Partie 3: Détermination de la masse volumique à l'aide d'un pycnomètre à déplacement de liquide

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 8130-3 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Sub-Committee SC 9, *General test methods for paints and varnishes*.

ISO 8130 consists of the following parts, under the general title *Coating powders*:

— *Part 1: Determination of particle size distribution by sieving*

— *Part 2: Determination of density by gas comparison pyknometer (referee method)*

— *Part 3: Determination of density by liquid displacement pyknometer*

— *Part 4: Calculation of lower explosion limit*

— *Part 5: Determination of flow properties of a powder/air mixture*

— *Part 6: Determination of gel time of thermosetting coating powders at a given temperature*

— *Part 7: Determination of loss of mass on stoving*

— *Part 8: Assessment of the storage stability of thermosetting powders*

— *Part 9: Sampling*

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Coating powders —

Part 3:
Determination of density by liquid displacement pyknometer

1 Scope

This part of ISO 8130 specifies a liquid displacement pyknometer method for the determination of the density of coating powders. The method is based on a determination of the mass and the volume of a test portion.

The apparatus specified is relatively inexpensive, but the liquid displacement pyknometer method is liable to give erroneous results, particularly if the powder swells in contact with the displacement liquid used or the displacement liquid does not totally displace the air between the powder particles. The liquid displacement method is much slower in execution and less accurate than the gas comparison pyknometer method specified in ISO 8130-2 and is only to be used if it can be shown that the same results will be obtained as for the gas comparison pyknometer method.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 8130. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 8130 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 787-10:1981, *General methods of test for pigments and extenders — Part 10: Determination of density — Pyknometer method.*

ISO 842:1984, *Raw materials for paints and varnishes — Sampling.*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

ISO 8130-2:1992, *Coating powders — Part 2: Determination of density by gas comparison pyknometer (referee method).*

3 Principle

The density of the coating powder is determined in a liquid displacement pyknometer in accordance with ISO 787-10, using a liquid that completely wets the product under test without swelling or dissolving it.

4 Materials

4.1 Water, of at least grade 3 purity as defined in ISO 3696, freshly boiled and cooled.

4.2 Displacement liquid: any suitable aliphatic hydrocarbon or mixture of aliphatic hydrocarbons, free from aromatic compounds, with a boiling point within the range 80 °C to 140 °C.

n-Heptane has been found to be suitable in many instances for the determination of the density of thermosetting coating powders. However, if there is an interaction between *n*-heptane and the product under test or its components, a suitable alternative liquid shall be used.

4.3 Acetone, analytical grade.

5 Apparatus

5.1 Pyknometer, complying with ISO 787-10.

5.2 Vacuum pump, with rubber pressure tube attachment.

5.3 Balance, capacity 200 g, capable of weighing to 1 mg or less.

6 Sampling

Take a representative sample of the product to be tested, as described in ISO 842.

7 Procedure

Carry out the determination in duplicate at $(23 \pm 0,5)$ °C. Weigh to the nearest 1 mg.

7.1 Determination of the density of the displacement liquid

Weigh the empty clean pyknometer (5.1), fill it to the mark with the displacement liquid (4.2) and reweigh. Empty the pyknometer, wash it with three separate portions of acetone (4.3) and dry it under vacuum. Refill the pyknometer with water (4.1) and reweigh.

Calculate the density ϱ_1, in grams per millilitre, of the displacement liquid at 23 °C, using the equation

$$\varrho_1 = \frac{m_1 - m_0}{m_2 - m_0} \times \varrho_0$$

where

m_0 is the mass, in grams, of the empty pyknometer;

m_1 is the mass, in grams, of the pyknometer containing the displacement liquid;

m_2 is the mass, in grams, of the pyknometer containing water;

ϱ_0 is the density, in grams per millilitre, of the water. (For the purposes of this part of ISO 8130, take the density of water at 23 °C as 0,998 g/ml.)

7.2 Determination of the density of the coating powder

Empty the pyknometer, wash it with three separate portions of acetone and dry it under vacuum. Introduce a test portion of the sample of between 3 g and 4 g into the pyknometer and reweigh. Add sufficient displacement liquid to wet the test portion and just

cover it. Attach the opening of the pyknometer to the pressure tube connected to the vacuum pump (5.2). Reduce the pressure in the pyknometer to a maximum of 1,2 kPa[1)] and shake until there is no further emission of air from the powder/liquid mixture. Carefully allow the pressure in the pyknometer to rise to atmospheric pressure. Then fill the pyknometer to the mark with displacement liquid, taking care not to disturb the sedimented powder. Take care also on filling in order to avoid powder remaining in the neck of the pyknometer. Reweigh the filled pyknometer.

8 Expression of results

8.1 Calculation

Calculate the density ϱ_p, in grams per millilitre, of the coating powder at 23 °C, using the equation:

$$\varrho_p = \frac{m_3 - m_0}{(m_1 - m_0) - (m_4 - m_3)} \times \varrho_1$$

where

m_0 is the mass, in grams, of the empty pyknometer;

m_1 is the mass, in grams, of the pyknometer containing the displacement liquid;

m_3 is the mass, in grams, of the pyknometer containing the test portion;

m_4 is the mass, in grams, of the pyknometer containing the test portion and the displacement liquid;

ϱ_1 is the density, in grams per millilitre, of the displacement liquid at 23 °C.

If the two determinations differ by more than 0,04 g/ml, repeat the determination (7.2).

Calculate the mean of two valid determinations and report the result to the nearest 0,01 g/ml.

8.2 Precision

Only limited precision data are currently available. As a rule, a reproducibility of 0,05 g/ml can be expected.

9 Test report

The test report shall contain at least the following information:

a) all details necessary to identify the product tested;

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1) 100 kPa = 1 bar