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Paints and varnishes — Determination of density —

Part 1: Pycnometer method

Peintures et vernis — Détermination de la masse volumique —
Partie 1: Méthode pycnométrique

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 9, *General test methods for paints and varnishes*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 139, *Paints and varnishes*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This fourth edition cancels and replaces the third edition (ISO 2811-1:2016), which has been technically revised.

The main changes are as follows:

— a requirement to de-aerate the sample prior to the determination in order to achieve reproducible results for the density has been added to 8.2;

— the text has been editorially revised and the normative references have been updated.

A list of all parts in the ISO 2811 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Paints and varnishes — Determination of density —

Part 1: Pycnometer method

1 Scope

This document specifies a method for determining the density of paints, varnishes and related products using a metal or Gay-Lussac pycnometer.

The method is limited to materials of low or medium viscosity at the temperature of test. The Hubbard pycnometer (see ISO 3507) can be used for highly viscous materials.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1513, *Paints and varnishes — Examination and preparation of test samples*

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

ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
density
ρ
mass divided by the volume of a portion of a material

Note 1 to entry: It is expressed in grams per cubic centimetre.

4 Principle

A pycnometer is filled with the product under test. The density is calculated from the mass of the product in the pycnometer and the known volume of the pycnometer.

5 Temperature

The effect of temperature on density is highly significant with respect to filling properties, and varies with the type of product.

For international reference purposes, it is essential to standardize one test temperature, and (23,0 ± 0,5) °C is specified in this document. It can be more convenient, however, to carry out comparative testing at another agreed temperature, for example (20,0 ± 0,5) °C, as specified by relevant weights and measures regulation (see B.2).

The test sample and pycnometer shall be conditioned to the specified or agreed temperature, and it shall be ensured that the temperature variation does not exceed 0,5 °C during testing.

6 Apparatus

Ordinary laboratory apparatus and glassware, together with the following shall be used.

6.1 Pycnometer

Either a) or b) below can be used.

a) Metal pycnometer, with a volume of either 50 cm^3 or 100 cm^3, a circular cross-section and a cylindrical form, made of a smoothly finished corrosion-resistant material with a snugly fitting lid having a hole in its centre.

The inside of the lid shall be concave (see Figure 1).

b) Glass pycnometer, with a volume in the range 10 cm^3 to 100 cm^3 (Gay-Lussac type) [see Figure 2].

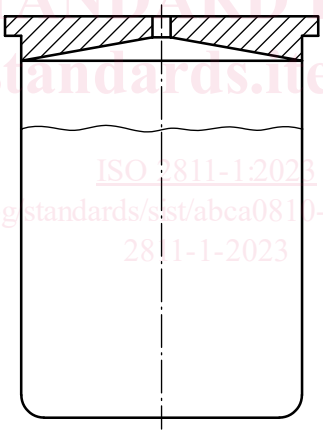

Figure 1 — Metal pycnometer

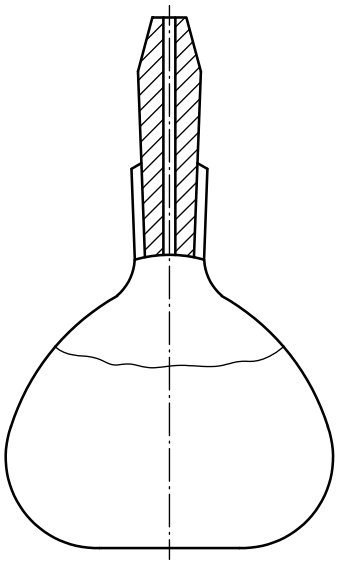

Figure 2 — Gay-Lussac pycnometer

6.2 Analytical balance, with a maximum permissible error of 1 mg for pycnometers for less than 50 ml or a maximum permissible error of 10 mg for 50 ml to 100 ml pycnometers.

The maximum permissible error of the balance required depends on the size of the pycnometer used (see also 8.2).

6.3 Thermometer, with a maximum permissible error of 0,2 °C.

NOTE Typically, a thermometer with a maximum permissible error of 0,2 °C has a resolution of 0,05 °C.

6.4 Temperature control unit

Either a) or b) below can be used.

a) Temperature-controlled chamber, capable of accommodating the balance, pycnometer and test sample and maintaining them at the specified or agreed temperature (see Clause 5).

b) Water bath, capable of maintaining the pycnometer and test sample at the specified or agreed temperature.

7 Sampling

Take a representative sample of the product under test as specified in ISO 15528.

Examine and prepare the sample as specified in ISO 1513. The sample shall be free from any air bubbles.

8 Procedure

8.1 General

Carry out a single determination on a fresh test sample.

The pycnometer shall be calibrated. An example of a calibration method is given in Annex A.

8.2 Determination

If working with a temperature-controlled chamber [see 6.4 a)], put the pycnometer (6.1) and the test sample next to the balance (6.2) in the chamber maintained at the specified or agreed temperature.

If working with a water bath [see 6.4 b)] rather than a temperature-controlled chamber, put the pycnometer and the test sample in the water bath, maintained at the specified or agreed temperature.

Allow approximately 30 min for temperature equilibrium to be reached.

Using the thermometer (6.3), measure the temperature, t_T, of the test sample.

Check throughout the determination that the temperature of the chamber or water bath remains within the specified limits.

Weigh the pycnometer and record the mass, m_1, to the nearest 10 mg for pycnometers of 50 cm^3 to 100 cm^3, and to the nearest 1 mg for pycnometers less than 50 cm^3 in volume.

Depending on the matrix, the sample shall be de-aerated prior to the determination in order to achieve reproducible results for the density.

NOTE 1 For waterborne coating matrices, de-aeration with a suitable mixing machine for about 30 s at 2 000 min^{-1} was found to be suitable.

Fill the pycnometer with the product under test, if necessary, after de-aeration, taking care to avoid the formation of air bubbles.

Place the lid or stopper of the pycnometer firmly in position and wipe off any excess liquid from the outside of the pycnometer with an absorbent material wetted with solvent; wipe carefully with cotton wool.

Record the mass of the pycnometer filled with the product under test, m_2.

NOTE 2 Liquid adhering to the ground-glass surfaces of a glass pycnometer or to the areas of contact between the lid and body of a metal pycnometer causes too high a balance reading. This source of error can be minimized by ensuring that the joints are firmly seated and by limiting air bubbles.

9 Calculation

Calculate the density, ρ, of the product, in grams per cubic centimetre, at the test temperature, t_T, using Formula (1):

$$\rho = \frac{m_2 - m_1}{V_t} \tag{1}$$

where

m_1 is the mass, in grams, of the empty pycnometer;

m_2 is the mass, in grams, of the pycnometer filled with the product at the test temperature, t_T;

V_t is the volume, in cubic centimetres, of the pycnometer at the test temperature, t_T, determined in accordance with Annex B.

NOTE The result is not corrected for air buoyancy because the uncorrected value is required by most filling-machine control procedures and the correction (0,001 2 g/cm^3) is negligible in relation to the precision of the method.

If the test temperature used is not the reference temperature, the density may be calculated using Formula (B.2).

10 Precision

10.1 Repeatability limit, *r*

The value below which the absolute difference between two single test results may be expected to lie, with a 95 % probability, is:

— 0,001 g/cm^3 for solvents, and

— 0,005 g/cm^3 for coating materials.

This applies to two single tests results obtained on identical material by one operator in one laboratory using the same equipment within a short interval of time using the standardized test method.

10.2 Reproducibility limit, *R*

The value below which the absolute difference between two test results may be expected to lie, with a 95 % probability, is:

— 0,002 g/cm^3 for solvents, and

— 0,007 g/cm^3 for coating materials.

This applies to two results obtained on identical material by operators in different laboratories using the standardized test method.

11 Test report

The test report shall include at least the following information:

a) all details necessary to identify the product tested;

b) a reference to this document, i.e. ISO 2811-1:2023;

c) the type of pycnometer used;

d) the test temperature;

e) the result of the density measurement, in grams per cubic centimetre, rounded to the nearest 0,001 g/cm^3 for pycnometers less than 50 cm^3 in volume and to the nearest 0,01 g/cm^3 for 50 cm^3 to 100 cm^3 pycnometers;

f) any deviation from the test method specified;

g) any unusual features (anomalies) observed during the test;

h) the date of the test.

Annex A
(informative)

Example of a calibration method

A.1 Procedure

Clean the pycnometer carefully inside and outside using a solvent which leaves no residue on evaporation and thoroughly dry it. Avoid leaving fingerprints on the pycnometer as they can falsify the balance reading.

Allow the pycnometer to stand next to the balance for 30 min to reach ambient temperature, then weigh it (m_1).

Fill the pycnometer with distilled or deionized water, of grade 2, as specified in ISO 3696, which has been previously boiled and then brought to a temperature not more than 1 °C below the test temperature and close it with the lid or stopper. Take care to prevent the formation of bubbles in the pycnometer.

Place the pycnometer on the water bath or in the temperature-controlled chamber and allow it to reach the test temperature. Remove any overflow by wiping with absorbent material (cloth or paper). Remove the pycnometer from the water bath or chamber and thoroughly dry its outer surface. Prevent any further heating of the pycnometer and ensure that there is no further overflow of water. Immediately weigh the filled pycnometer (m_3).

Since handling the pycnometer with bare hands increases its temperature and causes more overflow, as well as leaving fingerprints, the use of tongs or cellulose wadding for handling is recommended.

Immediate and rapid weighing of the filled pycnometer is necessary in order to minimize loss in mass due to evaporation of water through the overflow orifice.

It is essential that the pycnometer be calibrated at the same temperature as the density of the product under test is determined, since the volume of the pycnometer varies with the temperature. Otherwise, a correction should be made, as specified in Annex B.

A.2 Calculation of the volume of the pycnometer

Calculate the volume of the pycnometer, V_t, in cubic centimetres, at temperature, t_T, using Formula (A.1) or Formula (A.2):

$$V_t = \frac{m_3 - m_1}{\rho_W - \rho_A} \times \left(1 - \frac{\rho_A}{\rho_G}\right) \tag{A.1}$$

$$V_t = \frac{m_3 - m_1}{\rho_W - 0{,}001\,2} \times 0{,}999\,85 \tag{A.2}$$

where

m_1 is the mass, in grams, of the empty pycnometer;

m_3 is the mass, in grams, of the pycnometer filled with distilled water at the test temperature, t_T;

ρ_W is the density, in grams per cubic centimetre, of pure water at the test temperature, t_T (see Table A.1);