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Paints and varnishes — Determination of density —

Part 1: Pyknometer method

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

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Foreword

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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 2811-1 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 9, *General test methods for paints and varnishes*.

Together with the other parts, this part of ISO 2811 cancels and replaces ISO 2811:1974, which has been technically revised.

ISO 2811 consists of the following parts, under the general title *Paints and varnishes — Determination of density*:

- *Part 1: Pyknometer method*
- *Part 2: Immersed body (plummet) method*
- *Part 3: Oscillation method*
- *Part 4: Pressure cup method*

Annex A forms an integral part of this part of ISO 2811. Annex B is for information only.

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Paints and varnishes — Determination of density —

Part 1:

Pyknometer method

1 Scope

This part of ISO 2811 is one of a series of standards dealing with the sampling and testing of paints, varnishes and related products.

It specifies a method for determining the density of paints, varnishes and related products using a pyknometer.

The method is limited to materials of low or medium viscosity at the temperature of test. The Hubbard pyknometer can be used for highly viscous materials.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 2811. 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 2811 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 1512:1991, *Paints and varnishes — Sampling of products in liquid or paste form.*

ISO 1513:1992, *Paints and varnishes — Examination and preparation of samples for testing.*

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

3 Definition

For the purposes of this part of ISO 2811, the following definition applies.

3.1 density, ρ : The mass divided by the volume of a portion of a material, expressed in grams per millilitre (g/ml).

4 Principle

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

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 $(23 \pm 0,5) ^\circ\text{C}$ is specified in this part of ISO 2811. It may be more convenient, however, to carry out comparative testing at some other agreed temperature, for example $(20 \pm 0,5) ^\circ\text{C}$ as specified by relevant weights and measures legislation (see also annex B, clause 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 $0,5 ^\circ\text{C}$ during test.

6 Apparatus

Ordinary laboratory apparatus and glassware, together with the following:

6.1 Pycnometer

6.1.1 Metal pycnometer, with a volume of either 50 ml or 100 ml, 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).

or

6.1.2 Glass pycnometer, with a volume in the range 10 ml to 100 ml (either Gay-Lussac or Hubbard type) (see figures 2a and 2b).

6.2 Analytical balance, accurate to 1 mg for pycnometers less than 50 ml in volume or accurate to 10 mg for 50 ml to 100 ml pycnometers.

6.3 Thermometer, accurate to $0,2 ^\circ\text{C}$ and graduated at intervals of $0,2 ^\circ\text{C}$ or finer.

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

6.5 Dust-proof container.

7 Sampling

Take a representative sample of the product to be tested, as described in ISO 1512. Examine and prepare the sample as described in ISO 1513.

8 Procedure

8.1 General

Carry out the determination in duplicate, each time on a fresh test sample.

The pycnometer shall be recalibrated at intervals, e.g. after about 100 measurements or if any changes are noted in the pycnometer (see annex A).

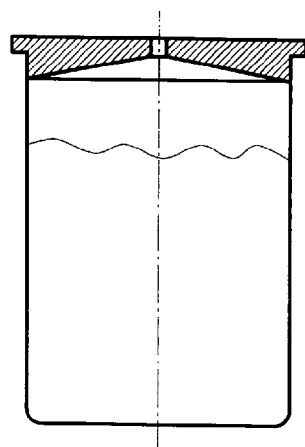


Figure 1 — Metal pyknometer

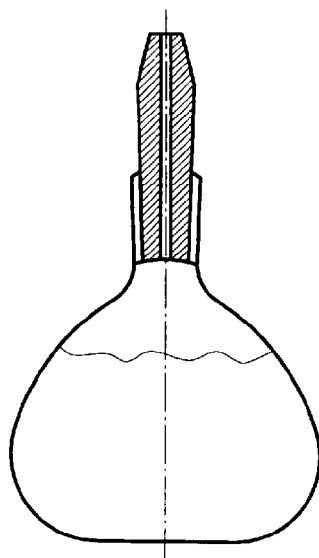


Figure 2a) — Gay-Lussac pyknometer

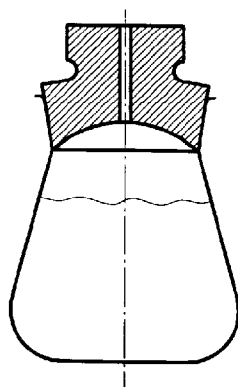


Figure 2b) — Hubbard pyknometer

8.2 Determination

If working with a temperature-controlled chamber (see 6.4), put the pycnometer (6.1) in its dust-proof container (6.5), 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) rather than a temperature-controlled chamber, put the pycnometer in its dust-proof container, 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 50 ml to 100 ml pycnometers and to the nearest 1 mg for pycnometers less than 50 ml in volume.

Fill the pycnometer with the product under test, 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. Then wipe carefully with cotton wool.

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

NOTE — 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 will cause too high a balance reading. It is advisable to minimize this source of error, and joints should be firmly seated, and air bubbles avoided.

9 Calculation

Calculate the density ρ of the product, in grams per millilitre, at the test temperature t_T using the following equation:

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

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 millilitres, of the pycnometer at the test temperature t_T , determined in accordance with annex A.

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/ml) is negligible in relation to the precision of the method.

If the test temperature used is not the reference temperature, then the density can be calculated using the equation in annex B, clause B.2.

10 Precision

10.1 repeatability (r)

The value below which the absolute difference between two single test results, each the mean of duplicates, obtained on identical material by one operator in one laboratory within a short interval of time using the standardized test method may be expected to lie with a 95 % probability is 0,001 g/ml.

10.2 Reproducibility (*R*)

The value below which the absolute difference between two test results, each the mean of duplicates, obtained on identical material by operators in different laboratories using the standardized test method may be expected to lie with a 95 % probability is 0,002 g/ml.

NOTE — These figures are taken from DIN 53217-2:1991, *Determination of density of paints, varnishes and similar coating materials by the pyknometer 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 part of ISO 2811 (i.e. ISO 2811-1);
- c) the type of pyknometer used;
- d) the test temperature;
- e) the density, in grams per millilitre, rounded to the nearest 0,001 g/ml;
- f) any deviation from the test method specified;
- g) the date of the test.

Annex A (normative)

Calibration of pyknometer

If a metal pyknometer is used, clean it carefully inside and outside using a solvent which leaves no residue on evaporation and thoroughly dry it. Avoid leaving fingerprints on the pyknometer as they will falsify the balance reading.

Allow the pyknometer to stand next to the balance in a dust-proof container for 30 min to reach ambient temperature, then weigh it (m_1).

Fill the pyknometer with previously boiled distilled or deionized water, of grade 2 purity as defined in ISO 3696, which has been 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 pyknometer.

Place the pyknometer 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 pyknometer from the water bath or chamber and thoroughly dry its outer surface. Prevent any further heating of the pyknometer and ensure that there is no further overflow of water. Immediately weigh the filled pyknometer (m_3).

NOTES

1 Since handling the pyknometer with bare hands will increase its temperature and cause more overflow, as well as leaving fingerprints, the use of tongs or cellulose wadding for handling is recommended.

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

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

A.1 Calculation of the volume of the pyknometer

Calculate the volume of the pyknometer V_t , in millilitres, at temperature t_T using one of the following equations:

$$V_t = \frac{m_3 - m_1}{\rho_w - \rho_A} \times \left(1 - \frac{\rho_A}{\rho_G} \right)$$

or

$$V_t = \frac{m_3 - m_1}{\rho_w - 0,0012} \times 0,999\,85$$

where

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

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

ρ_w is the density, in grams per millilitre, of pure water at the test temperature t_T (see table A.1);

ρ_A is the density of air (= 0,001 2 g/ml);

ρ_G is the density of the balance weights used (for steel, $\rho_G = 8 \text{ g/cm}^3$).

Table A.1 — Density of pure, air-free water

Temperature t_T °C	Density ρ_w g/ml	Temperature t_T °C	Density ρ_w g/ml	Temperature t_T °C	Density ρ_w g/ml
10	0,999 7	22	0,997 8	25	0,997 0
11	0,999 6				
12	0,999 5	22,1	0,997 8	25,1	0,997 0
13	0,999 4	22,2	0,997 7	25,2	0,997 0
14	0,999 2	22,3	0,997 7	25,3	0,997 0
15	0,999 1	22,4	0,997 7	25,4	0,996 9
16	0,998 9	22,5	0,997 7	25,5	0,996 9
17	0,998 8	22,6	0,997 6	25,6	0,996 9
18	0,998 6	22,7	0,997 6	25,7	0,996 9
19	0,998 4	22,8	0,997 6	25,8	0,996 8
		22,9	0,997 6	25,9	0,996 8
20	0,998 2	23	0,997 5	26	0,996 8
20,1	0,998 2	23,1	0,997 5	27	0,996 5
20,2	0,998 2	23,2	0,997 5	28	0,996 2
20,3	0,998 1	23,3	0,997 5	29	0,995 9
20,4	0,998 1	23,4	0,997 4	30	0,995 7
20,5	0,998 1	23,5	0,997 4	31	0,995 3
20,6	0,998 1	23,6	0,997 4	32	0,995 0
20,7	0,998 1	23,7	0,997 4	33	0,994 7
20,8	0,998 0	23,8	0,997 3	34	0,994 4
20,9	0,998 0	23,9	0,997 3	35	0,994 0
21	0,998 0	24	0,997 3	36	0,993 7
21,1	0,998 0	24,1	0,997 3	37	0,993 3
21,2	0,998 0	24,2	0,997 2	38	0,993 0
21,3	0,997 9	24,3	0,997 2	39	0,992 6
21,4	0,997 9	24,4	0,997 2	40	0,992 2
21,5	0,997 9	24,5	0,997 2		
21,6	0,997 9	24,6	0,997 1		
21,7	0,997 8	24,7	0,997 1		
21,8	0,997 8	24,8	0,997 1		
21,9	0,997 8	24,9	0,997 1		

Annex B (informative)

Temperature variation

B.1 Correction for thermal expansion of the pyknometer

If the test temperature t_T differs by more than 5 °C from the temperature at which the volume of the pyknometer is known, then the density should preferably be corrected for the change in volume of the pyknometer.

Calculate, to five significant figures, the volume V_T , in millilitres, of the pyknometer at the test temperature using the following equation:

$$V_T = V_C[1 + \gamma_P (t_T - t_C)]$$

where

V_C is the volume, in millilitres, of the pyknometer at the calibration temperature t_C ;

t_T is the test temperature, in degrees Celsius;

t_C is the calibration temperature, in degrees Celsius;

γ_P is the volume coefficient of thermal expansion, in reciprocal degrees Celsius (°C⁻¹), of the material from which the pyknometer is made (see table B.1).

Table B.1 — Coefficient of thermal expansion γ_P of materials used for pyknometers

Material	γ_P °C ⁻¹
Borosilicate glass	10×10^{-6}
Soda-lime glass	25×10^{-6}
Austenitic stainless steel	48×10^{-6}
Copper-zinc alloy (brass)	54×10^{-6} [Value for CuZn37 (Ms63)]
Aluminium	69×10^{-6}

B.2 Calculation of density at the reference temperature from measurements at other temperatures

If the density of the product under test is determined at a temperature different from the reference temperature the density ρ_C , in grams per millilitre, at the reference temperature can be calculated as follows:

$$\rho_C = \frac{\rho_t}{[1 + \gamma_m(t_C - t_T)]} = \rho_t[1 - \gamma_m(t_C - t_T)]$$

where

t_C is the reference temperature, in degrees Celsius;

t_T is the test temperature, in degrees Celsius;

γ_m is the volume coefficient of thermal expansion of the product under test, the approximate value of γ_m being $2 \times 10^{-4} \text{ } ^\circ\text{C}^{-1}$ for waterborne paints and $7 \times 10^{-4} \text{ } ^\circ\text{C}^{-1}$ for other paints;

ρ_t is the density, in grams per millilitre, of the product at the test temperature.

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Descriptors: paints, varnishes, tests, determination, density (mass/volume).

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