INTERNATIONAL STANDARD

ISO 2811-4

First edition
1997-12-15

Paints and varnishes — Determination of density —

Part 4:
Pressure cup method

Peintures et vernis — Détermination de la masse volumique —

Partie 4: Méthode du cylindre sous pression

ISO 2811-4:1997(E)

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 2811-4 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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Printed in Switzerland

Paints and varnishes — Determination of density —

Part 4:
Pressure cup 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 pressure cup.

The method is suitable for products which are aerated. Emulsion paints, for example, often trap small air bubbles, and these may still be present when the density is measured.

It is not however suitable for textured paints which contain coarse particles.

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

The product under test is compressed in a cylindrical pressure cup to reduce any error due to air bubbles which have not been eliminated. The density is calculated from the mass of the product and the volume of the cylinder.

NOTE — Air is more soluble at higher pressures, and it is thought that the main mechanism of bubble removal is by dissolving. Any undissolved bubbles are compressed to a fraction of their original size.

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 ± 0,5) °C is specified in this part of ISO 2811. However, it may be more convenient to carry out comparative testing at some other agreed temperature, for example (20 ± 0,5) °C as specified by relevant weights and measures legislation (see also annex B, clause B.2).

The test sample and pressure cup 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:

6.1 Pressure cup, comprising a hollow cylinder closed by a screw-driven piston at its lower end and by a pressure-release cap at the top (see figure 1). A calibration collar on the screw is set to stop further movement of the piston when the volume contained in the cylinder is 100 ml. The pressure-release cap is designed to let liquid escape when the pressure in the cylinder rises above (1 ± 0,1) MPa (10 bar). The apparatus is made of a strong, inert material, for example stainless steel, and it is easily dismantled for cleaning.

6.2 Thermometer, accurate to 0,2 °C and graduated at intervals of 0,2 °C or finer.

6.3 Temperature-controlled chamber, capable of maintaining the pressure cup and sample at the specified or agreed temperature (see clause 5).

6.4 Balance, accurate to 10 mg.

7 Sample

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

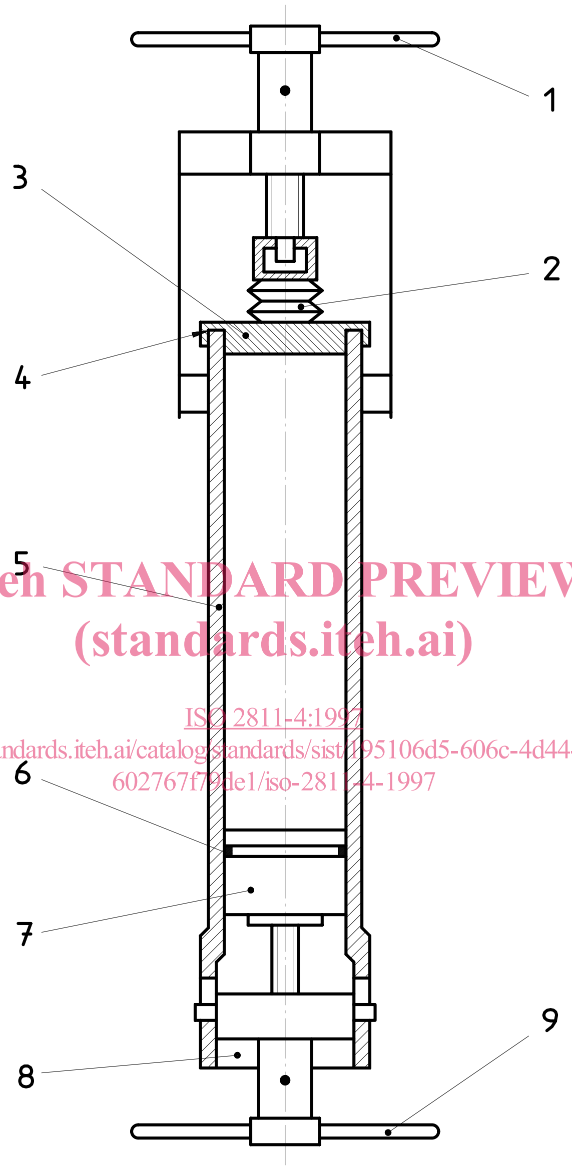
Carry out the determination in duplicate, each time on a fresh test sample.

8.2 Determination

Weigh the apparatus dismantled, ensure that it is clean and has a trace of grease on the moving parts. Fit the piston in its lowest position in the cylinder.

Weigh the complete apparatus to the nearest 10 mg (m_1). Bring the apparatus and the test sample to the specified or agreed temperature by placing it next to the balance for at least 30 min.

Pour the test sample into the cylinder until it is almost full, and allow sufficient time for the test sample and the cylinder to come to equilibrium at the test temperature. Confirm that the temperature is correct using the thermometer (6.2). Secure the pressure-release cap in position, in accordance with the manufacturer's instructions.



Key

1) Pressure-setting handle
2) Pressure-release device
3) End cap
4) End seal
5) Cylinder (closed volume 100 ml)
6) Piston seal
7) Piston
8) Calibration-collar stop
9) Pressure-application handle

Figure 1 — Pressure cup

Compress the test sample by turning the screw. As the pressure reaches 1 MPa, excess paint is forced out between the cylinder and the cap. Continue turning the screw until the calibration collar stops further movement.

NOTE — A rag held around the top of the cylinder will reduce the mess. It is essential that some excess paint is forced out, to confirm that full pressure is achieved.

Clean and dry the outside of the filled cup, and weigh it to the nearest 10 mg (m_2).

Unscrew the piston enough to reduce the pressure. Dismantle, empty and clean the apparatus.

For reference tests, and periodically during routine tests, check the calibration of the apparatus, using pure water as the test liquid (see annex A).

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 pressure cup;

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

V_t is the volume, in millilitres, of the pressure cup 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.

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 0,005 g/ml.

10.2 Reproducibility (R)

No data is currently available.

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 2811-4);

c) the supplier and serial number or other identification of the pressure cup;

d) the test temperature;

e) the results of each density measurement and the mean, rounded to the nearest 0,01 g/ml;

f) any deviation from the test method specified;

g) the date of the test.

Annex A
(normative)

Calibration of the pressure cup

A.1 Procedure

Put the pressure cup in a container next to the balance in order for it to reach room temperature (approximately 30 min). Then weigh using the balance and record this mass (m_1).

Almost fill the pressure cup with previously boiled distilled or deionized water of grade 2 purity as defined in ISO 3696, which has been brought to a temperature of not more than 1 °C below the test temperature. Secure the pressure-release cap in position, in accordance with the manufacuter's instructions.

Place the pressure cup in the temperature-controlled chamber and wait until the test temperature is reached then compress the water by turning the screw. Continue until the calibration collar stops further movement, and wipe off any excess water with an absorbent material (cloth or paper), leaving the pressure cup exactly filled. Take the pressure cup out of the chamber and dry it thoroughly on the outside. Avoid additional heating up. Weigh the filled pressure cup immediately (m_3).

NOTE — Handle the pressure cup as little as possible to minimize temperature increases.

A.2 Calculation of the volume of the pressure cup

Calculate the volume V_t, in millilitres, of the pressure cup at temperature t_T using the following equation:

$$V_t = \frac{m_3 - m_1}{\rho_W}$$

where

m_1 is the mass, in grams, of the empty pressure cup;

m_3 is the mass, in grams, of the pressure cup 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).

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
				27	0,996 5
20,1	0,998 2	23,1	0,997 5	28	0,996 2
20,2	0,998 2	23,2	0,997 5	29	0,995 9
20,3	0,998 1	23,3	0,997 5	30	0,995 7
20,4	0,998 1	23,4	0,997 4	31	0,995 3
20,5	0,998 1	23,5	0,997 4	32	0,995 0
20,6	0,998 1	23,6	0,997 4	33	0,994 7
20,7	0,998 1	23,7	0,997 4	34	0,994 4
20,8	0,998 0	23,8	0,997 3	35	0,994 0
20,9	0,998 0	23,9	0,997 3		
21	0,998 0	24	0,997 3	36	0,993 7
				37	0,993 3
21,1	0,998 0	24,1	0,997 3	38	0,993 0
21,2	0,998 0	24,2	0,997 2	39	0,992 6
21,3	0,997 9	24,3	0,997 2	40	0,992 2
21,4	0,997 9	24,4	0,997 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 pressure cup

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

Calculate, to five significant figures, the volume V_t, in millilitres, of the pressure cup at the test temperature using the following equation:

$$V_t = V_C\left[1 + \gamma_P\left(t_T - t_C\right)\right]$$

where

V_C is the volume, in millilitres, of the pressure cup at the calibration 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 pressure cup is made (see table B.1).

Table B.1 — Coefficient of thermal expansion γ_P of material used for pressure cups

Material	γ_P °C^{-1}
Austenitic stainless steel	48×10^{-6}