BS EN ISO 2811-4:2011

BSI Standards Publication

Paints and varnishes — Determination of density

Part 4: Pressure cup method (ISO 2811-4:2011)

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National foreword

This British Standard is the UK implementation of EN ISO 2811-4:2011. It supersedes BS EN ISO 2811-4:2001 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee STI/10, Test methods for paints.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Peintures et vernis - Détermination de la masse volumique - Partie 4: Méthode du cylindre sous pression (ISO 2811-4:2011)

Beschichtungsstoffe - Bestimmung der Dichte - Teil 4: Druckzylinder-Verfahren (ISO 2811-4:2011)

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Foreword

This document (EN ISO 2811-4:2011) has been prepared by Technical Committee ISO/TC 35 "Paints and varnishes" in collaboration with Technical Committee CEN/TC 139 “Paints and varnishes” the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by September 2011, and conflicting national standards shall be withdrawn at the latest by September 2011.

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Foreword

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ISO 2811-4 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 9, *General test methods for paints and varnishes*.

This second edition cancels and replaces the first edition (ISO 2811-4:1997), which has been technically revised.

The main changes are:

a) The unit for the density has been changed from grams per millilitre to grams per cubic centimetre, because this is the more common SI unit.

b) The determination in duplicate has been changed to a single determination.

c) The normative references have been updated.

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*

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INTERNATIONAL STANDARD **ISO 2811-4:2011(E)**

Paints and varnishes — Determination of density —

Part 4: Pressure cup method

1 Scope

This part of ISO 2811 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 might 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 referenced documents are indispensable for the application 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.

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

NOTE This is expressed in grams per cubic centimetre.

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.

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5 Temperature

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

Carry out the test at (23,0 ± 0,5) °C.

NOTE For some purposes, a different temperature, for example (20,0 ± 0,5) °C, might be needed.

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 cm^3. The pressure-release cap is designed to let liquid escape when the pressure in the cylinder rises above (1,0 ± 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 Sampling

Take a representative sample of the product under test, as described in ISO 15528. Examine and prepare the sample as described in ISO 1513.

8 Procedure

8.1 General

Carry out a single determination on a fresh test sample.

8.2 Determination

Weigh the dismantled apparatus, 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.

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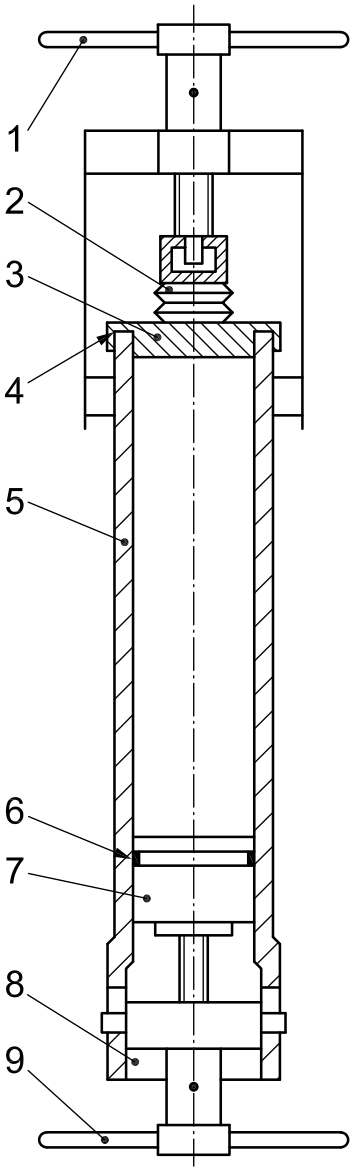

Key

1 pressure-setting handle
2 pressure-release device
3 end cap
4 end seal
5 cylinder (closed volume 100 cm^3)
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 about 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 reduces 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.

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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 cubic centimetre, at the test temperature, t_T, using Equation (1):

$$\rho = \frac{m_2 - m_1}{V_t} \quad (1)$$

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 cubic centimetres, 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/cm^3) is negligible in relation to the precision of the method.

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

10 Precision

10.1 General

The precision of the method depends on the characteristics of the product under test. For materials which contain no entrapped air, the values in 10.2 and 10.3 are valid.

10.2 Repeatability limit, r

The value below which the absolute difference between two single test 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, may be expected to lie, with a 95 % probability, is 0,005 g/cm^3.

10.3 Reproducibility limit, R

No data are 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 2811-4:2011;

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

d) the test temperature;

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e) the result of the density measurement, in grams per cubic centimetre, rounded to the nearest 0,001 g/cm^3;

f) any deviation from the test method specified;

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

h) the date of the test.

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Annex A
(normative)

Calibration of pressure cup

A.1 General

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 distilled or deionized water of grade 2 purity, as defined in ISO 3696, which has been previously boiled and then 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 manufacturer'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).

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 of the pressure cup, V_t, in cubic centimetres, at temperature, t_T, using Equation (A.1):

$$V_t = \frac{m_3 - m_1}{\rho_W} \tag{A.1}$$

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 cubic centimetre, of pure water at the test temperature, t_T (see Table A.1).

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

Temperature t_T °C	Density ρ_W g/cm³	Temperature t_T °C	Density ρ_W g/cm³	Temperature t_T °C	Density ρ_W g/cm³
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		

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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, the density should preferably be corrected for the change in volume of the pressure cup.

Calculate, to five significant figures, the volume, V_t, in cubic centimetres, of the pressure cup at the test temperature using Equation (B.1):

$$V_t = V_C\left[1+\gamma_P(t_T - t_C)\right] \tag{B.1}$$

where

V_C is the volume, in cubic centimetres, of the pressure cup at the calibration temperature, t_C;

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);

t_T is the test temperature, in degrees Celsius;

t_C is the calibration temperature, in degrees Celsius.

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

Material	γ_P $°C^{-1}$
Austenitic stainless steel	48×10^{-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 cubic centimetre, at the reference temperature can be calculated as follows:

$$\rho_C = \frac{\rho_t}{\left[1+\gamma_m(t_C - t_T)\right]} = \rho_t\left[1-\gamma_m(t_C - t_T)\right] \tag{B.2}$$

where

ρ_t is the density, in grams per cubic centimetre, of the product at the test temperature;

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

t_C is the reference temperature, in degrees Celsius;

t_T is the test temperature, in degrees Celsius.

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