BS EN ISO 3233-1:2013

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Paints and varnishes — Determination of the percentage volume of non-volatile matter

Part 1: Method using a coated test panel to determine non-volatile matter and to determine dry film density by the Archimedes principle

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This British Standard is the UK implementation of EN ISO 3233-1:2013. It supersedes BS 3900-A10:1998 which is withdrawn.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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Paints and varnishes - Determination of the percentage volume of non-volatile matter - Part 1: Method using a coated test panel to determine non-volatile matter and to determine dry film density by the Archimedes principle (ISO 3233-1:2013)

Peintures et vernis - Détermination du pourcentage en volume de matière non volatile - Partie 1: Méthode utilisant un panneau d'essai revêtu pour déterminer la matière non volatile et pour déterminer la masse volumique du feuil sec par le principe d'Archimède (ISO 3233-1:2013)

Beschichtungsstoffe - Bestimmung des Volumens nichtflüchtiger Anteile durch Bestimmung der Trockenfilmdichte (ISO 3233-1:2013)

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Foreword

This document (EN ISO 3233-1:2013) 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of an identical text or by endorsement, at the latest by July 2013, and conflicting national standards shall be withdrawn at the latest by July 2013.

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The text of ISO 3233-1:2013 has been approved by CEN as a EN ISO 3233-1:2013 without any modification.

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Introduction

This method is used to measure the density and to determine the volume of a dry coating obtainable from a given volume of liquid paint. This volume is considered to be the most meaningful measure of the coverage (area of surface covered at a specified dry-film thickness per unit volume) of a paint, varnish or related product. The value obtained by this method might not be the same as that calculated on the basis of the addition of masses and volumes of the raw materials in a formulation. The volume occupied by a combination of resin and solvent can be the same as, greater than or less than the combined volume of the separate components, due to contraction or expansion of the resin and solvent. A second factor affecting the volume of a dry coating formulation is the degree to which the spaces between pigment particles are filled with binder. A third factor is the use of volatile components in reactive systems that, by their reaction, change into non-volatile film-building materials, i.e. amines and reactive solvents in high-build two-component coating materials.

Above and close to the critical pigment volume concentration, the volume of a dry paint film is greater than the theoretical volume, due to an increase in unfilled voids between pigment particles. The porosity of the film means that this method is unsuitable.

The values obtained for the non-volatile matter by volume are dependent on the temperature and time of heating, and these conditions should be carefully considered for the material being tested.

INTERNATIONAL STANDARD **ISO 3233-1:2013(E)**

Paints and varnishes — Determination of the percentage volume of non-volatile matter —

Part 1:
Method using a coated test panel to determine non-volatile matter and to determine dry film density by the Archimedes principle

1 Scope

This part of ISO 3233 describes a procedure for determining the non-volatile matter by volume, NV_V, of coating materials and related products by measuring the density of a dried coating for any specified temperature range and period of drying or curing. This method determines the non-volatile matter immediately after application.

Using the non-volatile matter by volume results obtained in accordance with this part of ISO 3233, it is possible to calculate the spreading rate of coating materials.

The method specified in this part of ISO 3233 is the preferred method for air-drying materials. Its use for other materials still has to be tested.

This part of ISO 3233 is not applicable to coating materials in which the critical pigment volume concentration is exceeded.

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 2811-1, *Paints and varnishes — Determination of density — Part 1: Pyknometer method*

ISO 2811-2, *Paints and varnishes — Determination of density — Part 2: Immersed body (plummet) method*

ISO 2811-3, *Paints and varnishes — Determination of density — Part 3: Oscillation method*

ISO 2811-4, *Paints and varnishes — Determination of density — Part 4: Pressure cup method*

ISO 4618, *Paints and varnishes — Terms and definitions*

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

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 4618 and the following apply.

3.1
non-volatile matter
NV
residue by mass obtained by evaporation under specified conditions

[ISO 4618:2006]

NOTE This part of ISO 3233 and ISO 3251 specify different conditions for the determination of NV. Therefore in this part of ISO 3233, the symbol NV_m is used for non-volatile matter by mass.

3.2
non-volatile matter by volume
NV_V
percentage residue by volume obtained by evaporation under specified conditions

3.3
spreading rate
s
surface area that can be covered by a given quantity of coating material to give a dried film of the required thickness

NOTE It is expressed in m^2/l or m^2/kg.

3.4
practical dry-film density
ρ_p
practically determined density of a dried and cured coating

4 Principle

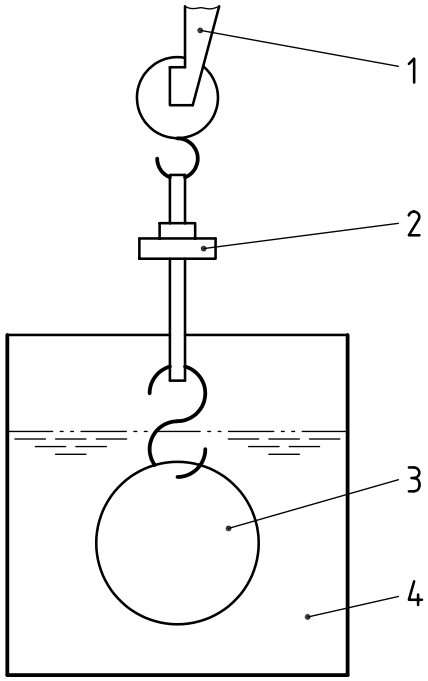
A receptacle (disc or plate) is weighed in air and in water (or other suitable liquid of known density), coated with the product to be tested, dried and reweighed in air and in the same liquid. From these measurements, the mass, the volume and hence the density of the dry coating are calculated. The non-volatile matter by volume is calculated from the quotient of the density of the coating material and the density of the dry film.

5 Apparatus and reagents

Standard laboratory apparatus, together with the following:

5.1 Analytical balance, accurate to 0,1 mg.

A single-pan balance is most convenient, and a useful modification is to replace the balance pan by a standard counterweight attachment as shown in Figure 1.



Key

1 balance arm
2 standard counterweight attachment
3 disc
4 immersion liquid

Figure 1 — Special balance support

5.2 Receptacle (see 7.2).

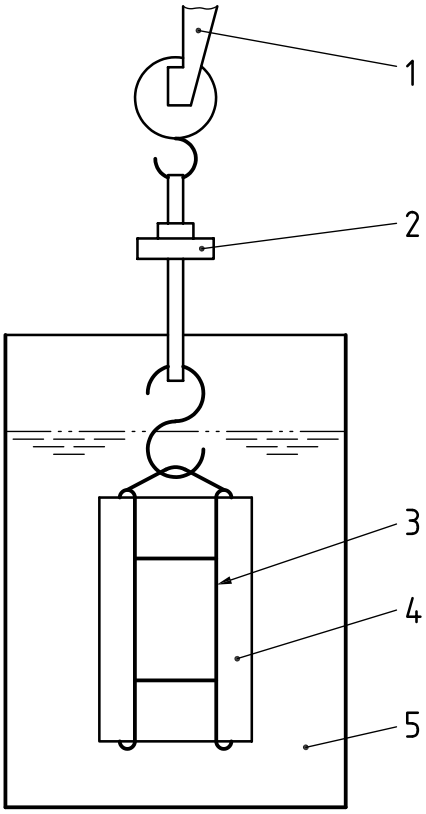
5.2.1 Disc, about 60 mm in diameter and about 0,7 mm thick, with a small hole 2 mm to 3 mm from the edge.

NOTE A stainless-steel disc has been found satisfactory but has the disadvantage of having a density much in excess of normal liquid coatings. Discs of lighter material, including plastics, for example poly(ethylene terephthalate), are permitted provided they do not change in volume by contact with the solvents contained in the liquid coating, or during the heating and drying processes involved.

5.2.2 Plate, of size (75 ± 5) mm by (120 ± 5) mm, with a small hole 2 mm to 3 mm from the shorter side on the longitudinal axis of the panel.

Glass plates may be used as these are very flat. However, drilling a hole is difficult and therefore, if glass plates are used, they should preferably be suspended in a stirrup or cradle of thin wire (see Figure 2). The diameter of the wire shall not exceed 0,3 mm because of surface tension effects.

Plates of this size might be difficult to accommodate in a balance case. Smaller plates may therefore be used provided the coated area is no less than 5 600 mm^2.



Key

1 balance arm
2 standard counterweight attachment
3 wire cradle
4 plate
5 immersion liquid

Figure 2 — Wire cradle support for plate

5.3 Hook, made of stainless steel or synthetic thread, for attaching the receptacle to the balance during weighing operations. The diameter of the wire shall not exceed 0,3 mm because of surface tension effects.

5.4 Beaker, of size convenient for immersing the receptacle with a clearance of at least 10 mm and which can be accommodated in the balance case.

5.5 Support, for holding the beaker under the balance stirrup without jamming the pan damper, if a counterweight as recommended in 5.1 is not available.

5.6 Immersion liquid, of suitable density, in which the receptacle is immersed.

Distilled water is suitable for most coating materials. An organic liquid which does not affect the paint film may also be used.

5.7 Desiccator, containing a drying material.

5.8 Air oven, capable of maintaining the specified or agreed test temperature (see Annex A) to ±2 °C (for temperatures up to 150 °C) or ±3,5 °C (for temperatures above 150 °C and up to 200 °C). An air oven with forced ventilation shall be used.

WARNING — To protect against explosions and fire, careful handling of products containing flammable volatile materials is essential.

Air ovens of the same type shall be used by all parties for referee tests.

6 Sampling

Take a representative sample of the coating material to be tested, as described in ISO 15528.

Examine and prepare the test samples, as described in ISO 1513.

7 Procedure

7.1 Number of determinations and preparation

Carry out the determination in triplicate.

Samples may be applied to discs or plates by dipping, brushing or applicator as described in 7.4.

Examples of test temperatures and times of heating that may be used for various types of coating material are given in Annex A.

7.2 Choice of receptacle

The choice of receptacle (disc or plate) will depend on the type of coating being measured. Discs should preferably be used for paints of low viscosity and paints which are thinned for spray application. Plates may be used for thixotropic and other coatings which can be drawn down with a doctor blade or for paints applied by dipping or by spin coating.

7.3 Determination of volume of uncoated receptacle

7.3.1 Dry the receptacle (5.2) and suspension hook (5.3) in the oven (5.8), if required, at the recommended temperature for 10 min, cool in the desiccator (5.7) and weigh the receptacle in air. Record this mass as m_1.

7.3.2 Place in the beaker (5.4) sufficient of the liquid (5.6) to ensure that it will be at least 10 mm above the top of the suspended receptacle (see Figure 3). Indicate the level on the side of the beaker and check that this level is maintained throughout the determination. The temperature of the liquid should preferably be (23 ± 1) °C. Suspend the receptacle in the liquid (see the note) and again weigh it. Record this mass as m_2.

NOTE If water is used as the immersion liquid, 1 or 2 drops of a suitable wetting agent will help to ensure rapid and thorough wetting of the receptacle.

7.3.3 Record the temperature of the liquid and determine its density at this temperature (see 7.6). Record the density as ρ_1.

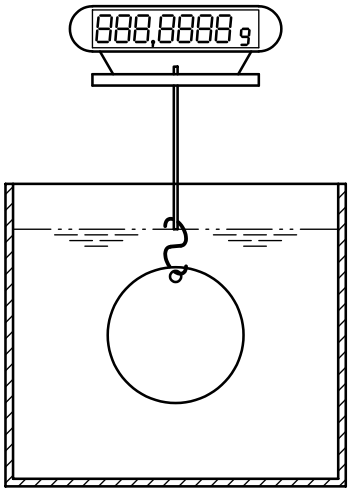


Figure 3 — Assembly for weighing the receptacle in the immersion liquid

7.4 Application

7.4.1 General

Apply, as described in 7.4.2 for discs or 7.4.3 for plates, the approximate amount of coating needed to achieve the specified film thickness, if known.

Viscous materials may be thinned with known amounts of specified thinners of known density.

Thixotropic materials may be applied by syringe or pipette before spreading by a film applicator.

7.4.2 Discs

The preferred method of application is by dipping, but samples may also be applied by brush.

Attach the disc (5.2.1) to a strong piece of wire and immerse it completely in the sample. Withdraw the disc at a steady rate, drain and remove any thick edge which forms at the bottom of the disc. This may be done by drawing a glass rod along the thick edge, rotating the rod at the same time. If any air bubbles form on the surface of the film, burst them with a needle.

NOTE The aim is to have, when dry, a uniform coating of specified thickness. A 30 µm film will normally represent a volume of greater than 0,15 ml of dry film. In some cases, it might be necessary to dip twice to obtain the specified thickness. In others, it might be necessary first to thin the liquid coating slightly with an appropriate thinner to obtain the correct conditions. The mass of dry coating required to conform to the thickness limits will vary according to its density.

Weigh the disc immediately and record this mass as m_3.

7.4.3 Plates

Apply the sample to a plate (5.2.2) by dipping, or by doctor blade or bar applicator, or by spin coating.

Weigh the plate immediately and record this mass as m_3.

7.4.4 Drying

Suspend the coated receptacle by the wire used for dipping the receptacle in the sample, or by any other suitable device. Do not use the suspension hook (5.3) for this purpose. Allow the film to dry under suitable conditions (see Annex A for examples).

7.5 Determination of volume of dry coating

7.5.1 After drying, detach the coated receptacle from the device used to suspend it during drying, cool it in a desiccator together with the suspension hook (5.3), and then weigh it in air. Record this mass as m_4.

7.5.2 Weigh the coated receptacle in the same liquid used for immersion of the uncoated receptacle (see 7.3.2, including its note), taking care to ensure that the temperature of the liquid is exactly the same as when weighing the uncoated receptacle in the liquid. If the mass changes rapidly due to the absorption of liquid by the coating, repeat the determination using another liquid that is not absorbed by the coating. Record this mass as m_5.

7.6 Determination of density of the liquid coating material

Determine, to the nearest 1 mg/ml, the density of the sample by one of the methods specified in ISO 2811-1 to ISO 2811-4, at exactly the same temperature as the density of the immersion liquid. Record this density as ρ_2.

8 Calculation

8.1 Calculation of the practical dry-film density, non-volatile-matter content and non-volatile matter by volume

Calculate the practical dry-film density, ρ_p, in grams per cubic centimetre, using Formula 1:

$$\rho_p = \frac{m_4 - m_1}{m_2 + m_4 - m_1 - m_5} \times \rho_1 \quad (1)$$

Calculate the non-volatile-matter content, NV_m, as a percentage by mass, using Formula 2:

$$NV_m = \frac{m_4 - m_1}{m_3 - m_1} \times 100 \quad (2)$$

Calculate the mean practical dry-film density, $\overline{\rho}_p$, and the mean non-volatile-matter content, $\overline{NV}_m$, of the three individual results.

Calculate the non-volatile matter by volume, NV_V, as a percentage by volume, using Formula 3:

$$NV_V = \overline{NV}_m \times \frac{\rho_2}{\overline{\rho}_p} \quad (3)$$

where

m_1 is the mass, in grams, of the uncoated receptacle in air;

m_2 is the apparent mass, in grams, of the uncoated receptacle immersed in the immersion liquid;

m_3 is the mass, in grams, of the wet coated receptacle;

m_4 is the mass, in grams, of the dry coated receptacle in air;

m_5 is the apparent mass, in grams, of the dry coated receptacle immersed in the immersion liquid;

NV_m is the non-volatile-matter content of the coating material, as a percentage by mass;

$\overline{NV}_m$ is the mean non-volatile-matter content, as a percentage by mass;

NV_V is the non-volatile matter by volume of the coating material, as a percentage by volume;

ρ_p is the practical dry-film density, in grams per cubic centimetre, of the coating at the test temperature;

$\overline{\rho}_p$ is the mean practical dry-film density, in grams per cubic centimetre, of the coating at the test temperature;

ρ_1 is the density, in grams per cubic centimetre, of the immersion liquid at the test temperature;

ρ_2 is the density, in grams per cubic centimetre, of the liquid coating material at the test temperature.

8.2 Calculation of the spreading rate

The spreading rate, s, is a value which is calculated solely from the non-volatile matter by mass or by volume.

It is the quotient of the surface area coated and the mass or volume required for this, in square metres per kilogram or square metres per litre, respectively.

Calculate the spreading rate relative to the mass, s_m, using Formula 4:

$$s_m = \frac{A}{m_0} = \frac{NV_m}{t_d \times \rho_p} \times 10 \quad (4)$$

Calculate the spreading rate relative to the volume, s_V, using Formula 5:

$$s_V = \frac{A}{V_0} = \frac{NV_m \times \rho_2}{t_d \times \rho_p} \times 10 = \frac{NV_V}{t_d} \times 10 \quad (5)$$

where

A is the surface area coated, in square centimetres;

m_0 is the mass required for coating, in grams;

V_0 is the volume required for coating, in litres;

NV_m is the non-volatile-matter content of the coating material, as a percentage by mass;

NV_V is the non-volatile matter by volume of the coating material, as a percentage by volume;

t_d is the dry-film thickness of the coating, in micrometres;

ρ_p is the practical dry-film density, in grams per cubic centimetre;

ρ_2 is the density, in grams per cubic centimetre, of the liquid coating material at the test temperature.

9 Precision

9.1 Repeatability limit

The repeatability limit, r, is the value below which the absolute difference between two test results (each being the average of two valid determinations), obtained on the same test material by the same operator in the same laboratory within a short period of time using the standard test method, can be expected to lie.

Two results for the non-volatile matter by volume, calculated from the practical dry-film density, may be regarded as acceptable, i.e. lying within the repeatability limit, if they do not differ by more than the following value:

$0{,}48 + (0{,}008\,6 \times NV_V)$

9.2 Reproducibility limit

The reproducibility limit, R, is the value below which the absolute difference between two test results (each being the average of two valid determinations), obtained on the same test material by different operators in different laboratories using the standard test method, can be expected to lie.

Two results for the non-volatile matter by volume, calculated from the practical dry-film density, may be regarded as acceptable, i.e. lying within the reproducibility limit, if they do not differ by more than the following value:

$$1{,}06 + (0{,}009\,6 \times NV_V)$$

10 Test report

The test report shall contain at least the following information:

a) all details necessary to identify the coating material tested (manufacturer, product code, batch number, etc.);

b) a reference to this part of ISO 3233 (ISO 3233-1:2012);

c) the method used to determine the density of the coating material (see 7.6);

d) the type of receptacle used (disc or plate);

e) the immersion liquid used;

f) the type of air oven used;

g) the method used to coat the receptacle with the sample under test, including the drying/curing conditions used;

h) the dry-film thickness of the coating, in micrometres;

i) the results of the test, as specified in Clause 8;

j) any deviation from the test method specified;

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

l) the date of testing.

Annex A
(informative)

Examples of test conditions

This annex gives examples of drying conditions that may be used (see Table A.1) for various types of coating material as defined by their mode of drying (see Table A.2).

Table A.1 — Drying conditions for various types of coating material

Drying class	Drying conditions
1	Follow manufacturer's stoving instructions. In the absence of this information, flash dry for 10 min to 15 min and stove at (105 ± 2) °C for 60 min.
2	7 days at (23 ± 2) °C and (50 ± 5) % RH
3	Follow manufacturer's stoving instructions. In the absence of this information, flash dry for 10 min to 15 min, then continue drying for 5 min to 10 min at 70 °C to 80 °C and stove at (125 ± 5) °C for 60 min. If drying is not continued after the flash-drying stage, the stoving period should be at least 120 min.

Table A.2 — Drying modes and drying classes

Drying mode	Drying class
Stoving e.g. alkyd/amino paint	1
Evaporation and oxidative drying e.g. alkyd air-drying paint	2
Evaporation and coalescence e.g. emulsion paint	2
Chemical reaction e.g. two-pack epoxy paint	2
Evaporation only e.g. chlorinated-rubber paint	2
Evaporation and coalescence with cross-linking e.g. Water-borne paint	3

Annex B
(informative)

Overview of the existing methods for determination of non-volatile-matter content and volume of non-volatile matter

Standard	Result	Determined (practical)	Calculated (theoretical)
ISO 3233-1	Percentage volume of non-volatile matter NV_V	Mass of the uncoated disc or plate in air m_1 Mass of the uncoated disc or plate immersed in the immersion liquid m_2 Mass of the wet coated disc or plate m_3 Mass of the dry coated disc or plate in air m_4 Mass of the dry coated disc or plate immersed in the immersion liquid m_5 Density of immersion liquid ρ_1 Density of the coating material ρ_2	Practical dry-film density ρ_p of the test portion, mean value of 3 determinations Non-volatile-matter content NV_m [a] of the test portion, mean value of 3 determinations Non-volatile matter by volume NV_V, calculated from the mean values above Spreading rate relative to the mass s_m Spreading rate relative to the volume s_V
ISO 3233-2	Practical percentage volume of non-volatile matter $NV_{V,p}$	Mass of the uncoated plate in air m_1 Mass of the uncoated plate immersed in the immersion liquid m_2 Mass of the dry coated plate in air m_3 Mass of the dry coated plate immersed in the immersion liquid m_4 Density of immersion liquid ρ_1 Density of the coating material ρ_2 NV of the coating material in accordance with ISO 3251	Practical dry-film density ρ_p of the test portion, 2 determinations Practical non-volatile matter by volume $NV_{V,p}$, mean value of 2 determinations Practical spreading rate relative to the mass $s_{p,m}$ Practical spreading rate relative to the volume $s_{p,V}$
ISO 3251	Non-volatile-matter content NV	Masses of the empty dish m_1, dish with the wet coating material m_2 and dish with the residue after drying m_3 Mean value of duplicates	Non-volatile-matter content NV
ISO 23811 (to become ISO 3233-3)	Theoretical percentage volume of non-volatile matter $NV_{V,t}$	NV of the coating material in accordance with ISO 3251 Density of the coating material ρ_1 Density of the solvents (main solvent) in the coating material ρ_2	Theoretical dry-film density ρ_t of the test portion, single determination Theoretical non-volatile matter by volume NV_V Theoretical spreading rate relative to the mass $s_{t,m}$ Theoretical spreading rate relative to the volume $s_{t,V}$

[a] The subscript “m” has been introduced because ISO 3233-1 and ISO 3251 specify different conditions for the determination of NV.

Bibliography

[1] ISO 3251, *Paints, varnishes and plastics — Determination of non-volatile-matter content*

[2] ISO 23811, *Paints and varnishes — Determination of percentage volume of non-volatile matter by measuring the non-volatile-matter content and the density of the coating material, and calculation of the theoretical spreading rate*

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