
**Paints and varnishes — Determination of
percentage volume of non-volatile matter
by measuring the density of a dried coating**

*Peintures et vernis — Détermination du pourcentage en volume de
matières non volatiles par mesurage de la masse volumique d'un
revêtement sec*

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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 3233 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 9, *General test methods for paints and varnishes*.

This third edition cancels and replaces the second edition (ISO 3233:1984), which has been technically revised.

The technical changes are that plates can also be used as test pieces (receptacles) besides discs, and that the drying conditions for all types of coating material are specified. The plates are used for thixotropic or other coatings which can be drawn down by a doctor blade.

Annex A forms an integral part of this International Standard.

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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 coating. 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 may 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 may be the same as, greater than or less than the combined volume of the separate components, since contraction or expansion of resin solutions in this way is a function of the solubility characteristics of the resin and the 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.

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Paints and varnishes — Determination of percentage volume of non-volatile matter by measuring the density of a dried coating

1 Scope

This International Standard is one of a series of standards dealing with the testing and sampling of paints, varnishes and related products.

It specifies a procedure for determining the volume percentage of non-volatile matter in paints, varnishes and related products by measuring the density of a dried coating for any specified temperature range and period of drying or curing.

The method is not suitable for paints formulated above the critical pigment volume concentration.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard 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 2811-1:1997, *Paints and varnishes — Determination of density — Part 1: Pycnometer method.*

ISO 2811-2:1997, *Paints and varnishes — Determination of density — Part 2: Immersed body (plummet) method.*

ISO 2811-3:1997, *Paints and varnishes — Determination of density — Part 3: Oscillation method.*

ISO 2811-4:1997, *Paints and varnishes — Determination of density — Part 4: Pressure cup method.*

ISO 3251:1993, *Paints and varnishes — Determination of non-volatile matter of paints, varnishes and binders for paints and varnishes.*

3 Definition

For the purposes of this International Standard, the following definition applies.

3.1 volume of non-volatile matter: The volume of the residue obtained when a uniform and specified thickness of the product under test is cured or dried at a specified temperature for a specified period.

4 Principle

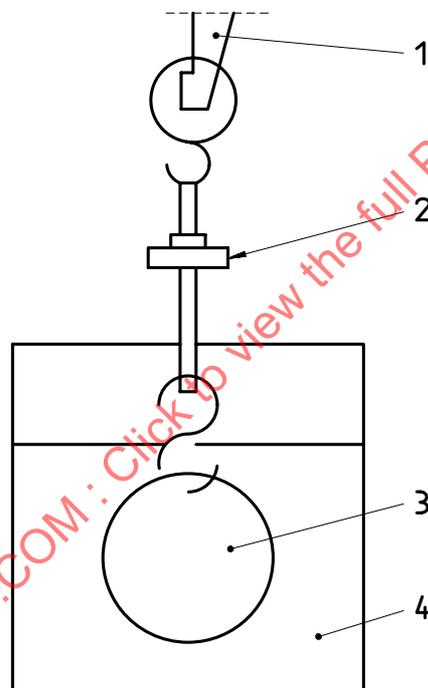
A receptacle (disc or plate) is weighed in air and in water (or other suitable liquid of known density), coated with the product to be tested, dried and reweighed in air and in the same liquid. From these measurements, the mass, the volume and hence the density of the dry coating are calculated. From the determinations of liquid coating density (ISO 2811), non-volatile matter by mass and dry coating density, the non-volatile matter by volume is calculated.

5 Apparatus and materials

Ordinary 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 Receptacles.

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 or other coatings which can be drawn down with a doctor blade or for paints applied by dipping.

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. A plate with a tip may be used for coatings which are applied by dipping (see figure 2).

Glass plates can 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 3). The diameter of the wire shall not exceed 0,3 mm because of surface tension effects.

NOTE — Plates of this size may be difficult to accommodate in a balance case. Smaller plates may be used provided the coated area is no less than $5\,600\text{ mm}^2$.

5.3 Hook, 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.

NOTE — A piece of nickel-chromium (80:20) wire about 30 mm to 40 mm long is satisfactory.

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.

Dimensions in millimetres

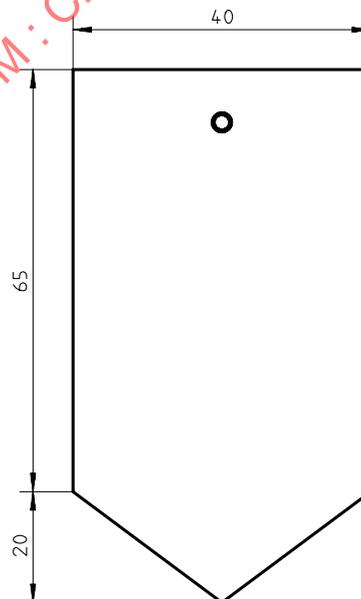
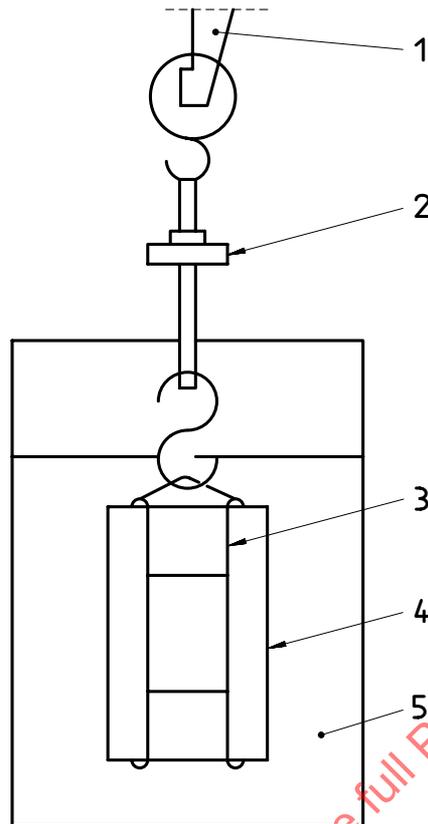


Figure 2 — Suitable plate for dipping
(smaller than the standard rectangular plate since it is coated on both sides)



Key

- 1 Balance arm
- 2 Standard counterweight attachment
- 3 Wire cradle
- 4 Plate
- 5 Immersion liquid

Figure 3 — Wire cradle support for plate

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 and type, to which the coating is inert.

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

5.7 Desiccator, with a drying material, e.g. silica gel.

5.8 Air oven, capable of maintaining the specified or agreed temperature (see annex A) to ± 2 °C (for temperatures up to 150 °C) or $\pm 3,5$ °C (for temperatures above 150 °C and up to 200 °C). The air oven shall be fitted with forced-ventilation equipment and the air speed shall be within the range 0,8 m/s to 1,2 m/s. A method of determining the air speed is given in ISO 3251:1993, annex C.

WARNING — To protect against explosions, it is essential that the number of receptacles in relation to the floor area of the oven be such that the solvent vapour concentration cannot exceed 20 g/m³. Poor air circulation and high concentrations of vapour may also lead to erroneous results.

6 Sampling

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

Examine and prepare the sample for testing, as described in ISO 1513.

7 Procedure

Carry out the determination in duplicate.

Samples may be applied to discs or plates by dipping, brushing or applicator as described in 7.2.

Refer to annex A for the test temperature and time of heating to be used for the particular type of sample.

7.1 Determination of volume of uncoated receptacle

7.1.1 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 .

7.1.2 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. 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 $(23 \pm 1) ^\circ\text{C}$. 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.

7.1.3 Record the temperature of the liquid and determine its density at this temperature (see 7.4). Record the density as ρ_1 .

7.2 Application

7.2.1 General

Apply, as described in 7.2.2 for discs or 7.2.3 for plates, the approximate amount of coating needed to achieve the specified film thickness, if known.

NOTE 1 Viscous materials may be thinned with known amounts of specified thinners of known density.

NOTE 2 Thixotropic materials may be stirred or applied by syringe before spreading by a film applicator.

7.2.2 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 $30 \mu\text{m}$ film will normally represent a volume of greater than 0,15 ml of dry film. In some cases, it may be necessary to dip twice to obtain the specified thickness. In others, it may 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 .

7.2.3 Plates

Apply the sample to a plate (5.2.2) by dipping as shown in figure 2, or by doctor blade or bar applicator.

Weigh the plate immediately and record this mass as m_3 .

7.2.4 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 the conditions given in annex A.

7.3 Determination of volume of dry coating

7.3.1 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 .

7.3.2 Weigh the coated receptacle in the same liquid used for immersion of the uncoated receptacle (see 7.1.2, including its note), taking care to ensure that the temperature of the liquid is exactly the same as when weighing the uncoated disc 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 .

NOTE — This method is not suitable for coatings formulated above the critical pigment volume concentration.

7.4 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 the various parts of ISO 2811, at exactly the same temperature as the density of the immersion liquid. Record this density as ρ_2 .

8 Expression of results

8.1 Calculation

Calculate the density of the dry coating and the percentage by volume of non-volatile matter using the following equations:

Density of dry coating, ρ_0

$$\rho_0 = \frac{m_3 - m_1}{m_2 + m_4 - m_1 - m_5} \times \rho_1$$

Percentage of non-volatile matter by mass, NV_m

$$NV_m = \frac{m_4 - m_1}{m_3 - m_1} \times 100$$

Percentage of non-volatile matter by volume, NV_v

$$NV_v = NV_m \times \frac{\rho_2}{\rho_0}$$

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 percentage non-volatile matter by mass;
- NV_v is the percentage of non-volatile matter by volume;
- ρ_0 is the density, in grams per millilitre, of the dry coating at the test temperature;
- ρ_1 is the density, in grams per millilitre, of the immersion liquid at the test temperature;
- ρ_2 is the density, in grams per millilitre, of the liquid coating material at the test temperature.

Calculate the mean of the two results obtained.

8.2 Replication

If the results of the determination are greater than the repeatability limit, carry out a third determination and take the arithmetic mean of all the results.

If the difference between the result of the third determination and those of the other determinations is greater than $1,5 \times (0,48 + 0,0086 \times NV_v)$, state this and the individual results in the test report.

9 Precision

9.1 Repeatability

The difference between results obtained by the same operator within a short time interval with the same apparatus under constant operating conditions on identical test material shall not exceed, at the 95 % confidence level,

$$0,48 + 0,0086 \times NV_v$$

where NV_v is the content of non-volatile matter, as a percentage by volume, in the liquid coating.

9.2 Reproducibility

The difference between results obtained by different operators working in different laboratories on identical test material shall not exceed, at the 95 % confidence level,

$$1,06 + 0,0096 \times NV_v$$

where NV_v is the content of non-volatile matter, as a percentage by volume, in the liquid coating.