
INTERNATIONAL STANDARD



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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Paints and varnishes — Determination of volume of dry coating (non-volatile matter) obtained from a given volume of liquid coating

Peintures et vernis — Détermination du volume de revêtement sec (matières non volatiles) obtenu à partir d'un volume donné de produit liquide

First edition — 1974-11-15

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UDC 667.63 : 531.73

Ref. No. ISO 3233-1974 (E)

Descriptors : paints, varnishes, coatings, measurement, dry matter, volume.

FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up 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.

Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 3233 was drawn up by Technical Committee ISO/TC 35, *Paints and varnishes*, and circulated to the Member Bodies in May 1973.

It has been approved by the Member Bodies of the following countries :

Brazil	Iran	South Africa, Rep. of
Bulgaria	Ireland	Spain
Canada	Israel	Sweden
Czechoslovakia	Netherlands	Switzerland
Egypt, Arab Rep. of	New Zealand	Thailand
France	Poland	Turkey
Germany	Portugal	United Kingdom
India	Romania	Yugoslavia

No Member Body expressed disapproval of the document.

Paints and varnishes – Determination of volume of dry coating (non-volatile matter) obtained from a given volume of liquid coating

0 INTRODUCTION

This International Standard is one of a series dealing with the testing of paints, varnishes and related products. It should be read in conjunction with ISO 1512, *Paints and varnishes – Sampling*, ISO 1513, *Paints and varnishes – Examination and preparation of samples for testing*, ISO 1515, *Paints and varnishes – Determination of volatile and non-volatile matter*, and ISO 2811, *Paints and varnishes – Determination of density*.

This method of test is intended to provide a measure of the volume of dry coating obtainable from a given volume of liquid coating. This 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 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, 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 further factor affecting the volume of a dry coating formulation is the degree to which the spaces between pigment particles are filled with binder.

Above 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.

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a standard procedure for determining the volume of non-volatile matter in paints, varnishes and related products for any specified temperature range and period of heating.

2 DEFINITION

volume of non-volatile matter: The volume percentage residue left when the product under test is cured or dried at a specified temperature for a specified period.

3 PRINCIPLE

The mass and volume of a disc (steel or other agreed material, see note in 4.2) are determined. The disc is coated

with the product to be tested and the mass and volume of the disc and the dried coating are then determined by weighing the coated disc in air and in a liquid of known density (see note in 4.6). From the measured masses and volumes of the disc before and after coating, the mass and volume of the dried coating film are calculated. From the density of the liquid coating and the percentage by mass of the non-volatile matter, the volume of the liquid coating deposited on the disc is calculated. The volume of the dried coating divided by the volume of liquid coating, multiplied by 100, is then equal to the percentage by volume of the non-volatile matter in the liquid coating.

4 APPARATUS AND MATERIAL

4.1 Analytical balance, accurate to 0,1 mg.

A single-pan type balance is most convenient and a useful adjunct is to replace the balance pan by a standard counterweight attachment as shown in figure 1.

4.2 Disc, about 60 mm diameter and about 0,7 mm thick, with a small hole 2 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 polyethyleneterephthalate, 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. The dish used for non-volatile determination of the liquid coating (see 6.3) may in some circumstances also be used for this determination in place of the disc.

4.3 Hook, for attaching the disc or dish to the balance during weighing operations. Because of surface tension effects its diameter should not exceed 0,3 mm. A wire of nickel-chromium (80 : 20) about 30 to 40 mm long, is satisfactory.

4.4 Beaker, of size convenient for immersing the disc and which can be accommodated in the balance case. A 600 ml beaker may be found convenient.

4.5 Support, for holding the beaker under the balance stirrup without jamming the pan damper if a counterweight as recommended in 4.1 is not available.

4.6 Liquid, of suitable density and type.

NOTE — The immersion liquid may be water or an organic liquid of low solvent power such as mineral spirits or kerosene. Distilled water is suitable for most products. Exceptions would be dry coatings containing water-leachable constituents and low-gloss coatings, the surfaces of which are poorly wetted by water. Hydrocarbon solvents of low solvent power ($KB^{1)}$ less than 36) are also suitable for most products but must not be used if the dry coating contains ingredients that are readily extracted by the solvent.

5 SAMPLING

A representative sample of the product to be tested shall be taken as specified in ISO 1512. The product shall then be examined and prepared for testing as specified in ISO 1513.

6 PROCEDURE

NOTE — At least two determinations shall be made.

6.1 Volume determination of uncoated discs or dishes

6.1.1 Dry the disc and suspension hook in an oven at $105 \pm 2^{\circ}\text{C}$ for 10 min, cool in a desiccator and weigh the disc in air.

6.1.2 Suspend the disc in the liquid to be used for the immersion (see note 1 below and note in 6.2.4) and again weigh it.

NOTES

1 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 disc.

2 The level of liquid in the beaker necessary for complete immersion of the disc should be marked and should be at least 10 mm above the top of the suspended disc. This level shall be maintained for subsequent weighing when the disc is coated.

6.1.3 Record the temperature of the suspension liquid and obtain its density at this temperature (see 6.4) (preferably the temperature should be $23 \pm 1^{\circ}\text{C}$).

6.1.4 Calculate the volume of the disc V_1 , in millilitres, as follows:

$$V_1 = \frac{m_1 - m_2}{\rho_1}$$

where

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

m_2 is the mass, in grams, of the uncoated disc in liquid;

ρ_1 is the density of liquid, in grams per millilitre, at the test temperature.

6.2 Volume determination of coated discs or dishes (see note in 6.2.1)

6.2.1 Attach the disc to a strong piece of wire and dip it

completely in a representative sample of the product to be tested. Withdraw the disc at a steady rate, drain and remove any thick edge which forms at the bottom of the disc. If any air bubbles form on the surface of the film, burst them with a needle point. The aim should be to have, when dry, a fairly even coating between 30 and 60 μm thick, which will normally represent a volume of between 0,15 and 0,30 ml of dry film. In some cases it may be necessary to dip twice to obtain a thickness between these limits. 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.

Where thixotropic products are involved and there is difficulty in obtaining an even coating, removing bubbles and avoiding thick edges, it is useful to subject the coated disc to a centrifugal action for 20 to 30 s to even out the coating thickness. Figure 2 illustrates a useful means of doing this by attaching two coated discs to a Y-shaped metal support which can be attached to the chuck of an electric motor geared to rotate at about 500 rev/min. The rotation is best done within a suitable enclosure such as a metal drum to trap the discarded material.

NOTE — When it is found convenient to replace the disc by the dish used for the determination of mass of non-volatile matter (see 6.3), it is usually necessary to add a quantity of suitable solvent to the liquid coating, sufficient to allow a satisfactory coating of the required dry film thickness to be deposited evenly over the base of the dish; in this way the two determinations can be combined.

6.2.2 Dry the coated disc in the manner recommended by the manufacturer for the curing of the coating under test. However, in the absence of such information, and unless there are indications to the contrary, a drying schedule of 3 h at $105 \pm 1^{\circ}\text{C}$ shall be used (see note below). Suspend the disc in a suitable oven by the wire or other means used for dipping (the suspension hook should not be used for the purpose).

NOTE — For air-drying products, it is usually recommended that the purchaser and vendor shall agree 3 h at $105 \pm 1^{\circ}\text{C}$ as being a satisfactory drying schedule for this test. For stoving products the drying conditions shall be those which most closely match those recommended by the manufacturer for the curing of the coating under test.

6.2.3 After stoving, detach the coated disc from its support, cool it in a desiccator together with the suspension hook and then weigh it in air.

6.2.4 Weigh the coated disc in the same liquid chosen for immersion of the uncoated disc (see 6.1.2, the notes relating to it, and the note below), taking care to ensure that the temperature of the suspension liquid is exactly the same as when weighing the uncoated disc in the liquid.

NOTE — If the mass changes rapidly due to the absorption of liquid in the paint film, an alternative liquid which is not absorbed in the paint film shall be used and the test repeated.

1) Kauri-butanol value — see ASTM D 1133.

6.2.5 Calculate the volume of the coated disc, V_2 , in millilitres, by the formula

$$V_2 = \frac{m_3 - m_4}{\rho_1}$$

where

m_3 is the mass, in grams, of the coated disc in air;

m_4 is the mass, in grams, of the coated disc in liquid;

ρ_1 is the density of liquid, in grams per millilitre, at the test temperature.

6.3 Determination of the non-volatile content of the liquid coating

Determine the content of non-volatile matter of the liquid coating by the method specified in ISO 1515.

NOTE — The uncoated and coated dish used in this determination may also, in agreed circumstances, be used for the volume determination of the dry coating (see 6.1, 6.2 and the note in 6.2.1).

6.4 Determination of density of the liquid coating

Determine to the nearest 0,001 g/ml the density of a representative sample of the liquid coating by the method specified in ISO 2811.

NOTE — It is important that the density of the liquid coating shall be determined at exactly the same temperature as the density of the immersion liquid and of the dry coating.

7 EXPRESSION OF RESULTS

7.1 Calculations

7.1.1 Calculate the volume of the dry coating, V_3 , in millilitres, by the formula

$$V_3 = V_2 - V_1$$

where

V_1 is the volume, in millilitres, of the uncoated disc (see 6.1.4);

V_2 is the volume, in millilitres, of the coated disc (see 6.2.5).

7.1.2 Calculate the volume of the liquid coating from which the dry coating was formed, V_4 , in millilitres, by the formula

$$V_4 = \frac{m_3 - m_1}{c \times \rho_2} \times 100$$

where

m_3 is the mass, in grams, of the coated disc in air;

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

c is the content of non-volatile matter, as a percentage by mass, of liquid coating (see 6.3);

ρ_2 is the density, in grams per millilitre, of the liquid coating at the test temperature.

7.1.3 Calculate the content of non-volatile matter of the liquid coating, as a percentage by volume, by the formula

$$\frac{V_3 \times 100}{V_4}$$

7.1.4 Calculate the mean of the results obtained.

7.2 Precision

7.2.1 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,

$$0,48 + 0,008 6 (NV)$$

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

7.2.2 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,

$$1,06 + 0,009 6 (NV)$$

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

8 TEST REPORT

The test report shall include the following particulars :

- a reference to this International standard or to a corresponding national standard;
- type and identification of the product under test, and whether a disc or a dish has been used;
- the liquid in which the discs or dishes were suspended;
- the test temperature;
- any deviation, by agreement or otherwise, from the test procedure described;
- the result of the test (the percentage by volume of the non-volatile matter);
- date of test.

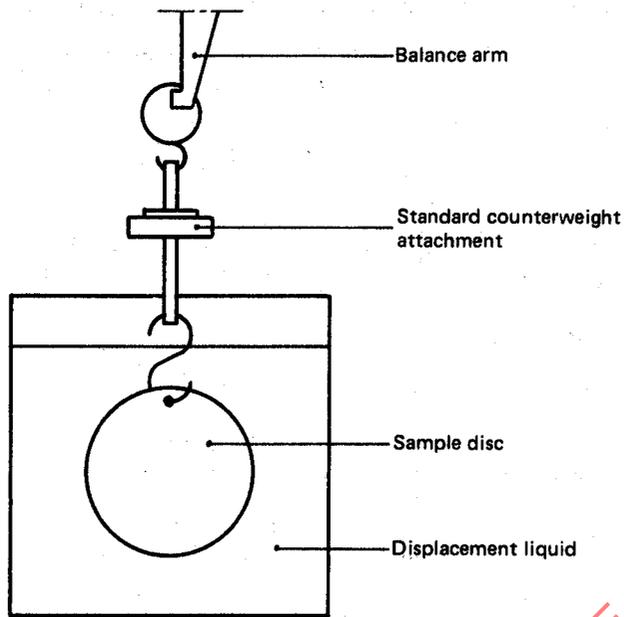


FIGURE 1 — Special balance support

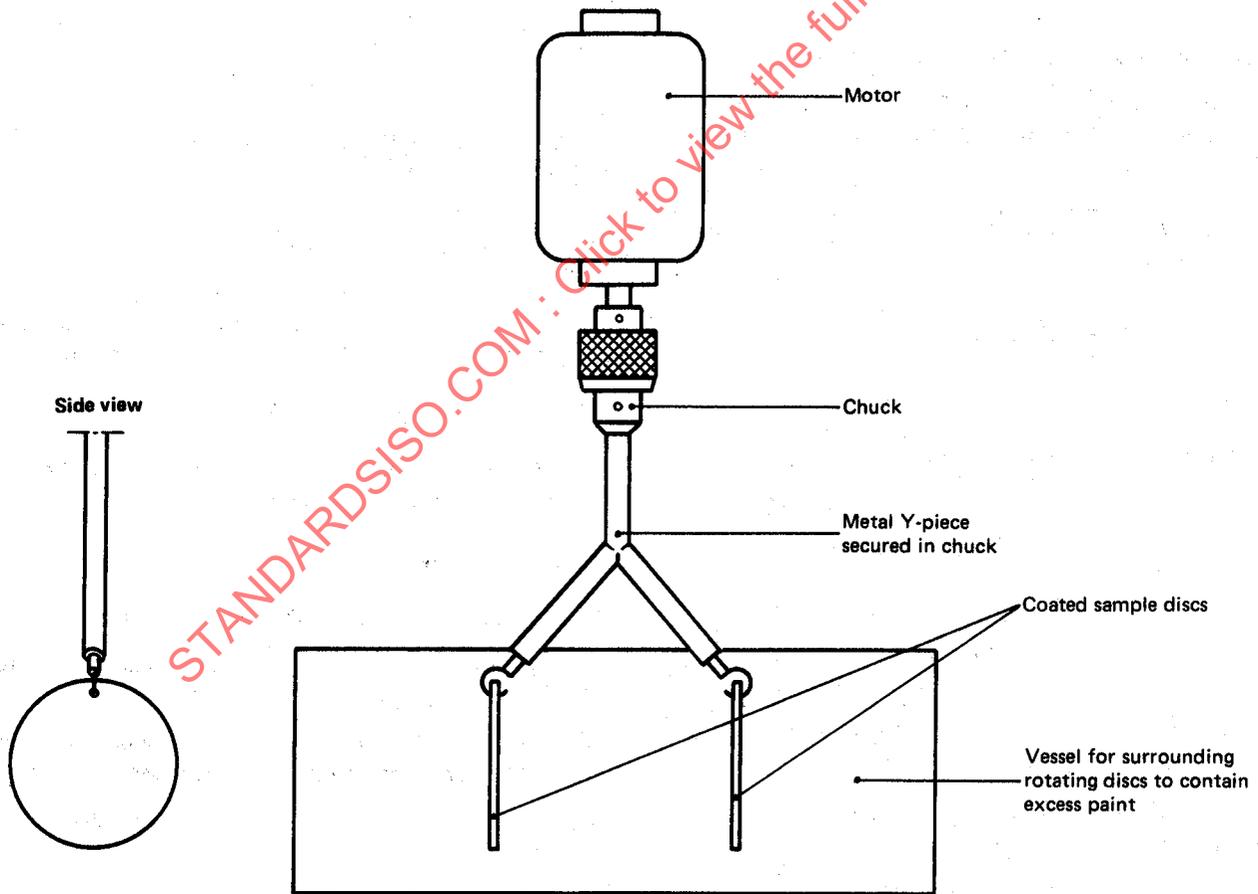


FIGURE 2 — Device for centrifuging discs to ensure a uniform coating of paint