

INTERNATIONAL STANDARD

**ISO
3251**

Second edition
1993-10-01

Paints and varnishes — Determination of non-volatile matter of paints, varnishes and binders for paints and varnishes

*Peintures et vernis — Détermination de l'extrait sec des peintures, des
verniss et des liants pour peintures et vernis*



Reference number
ISO 3251:1993(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 3251 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Sub-Committee SC 10, *Test methods for binders for paints and varnishes*.

This second edition cancels and replaces the first edition (ISO 3251:1974), as well as ISO 1515:1973. During the revision and combination of these two International Standards, the test method was restricted to the determination of non-volatile matter. The "Apparatus" and "Procedure" clauses in the two standards have been combined and the descriptions made more precise. In contrast to ISO 1515:1973, the test conditions (test temperature and period of heating) have to be agreed in every case (if not otherwise specified), and no single test temperature and period of heating are given as preferred test conditions.

Annex B specifies five different combinations of test conditions, and those to be used are selected from these combinations.

Annexes A, B and C form an integral part of this International Standard.

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Paints and varnishes — Determination of non-volatile matter of paints, varnishes and binders for paints and varnishes

1 Scope

This International Standard is one of a series of standards dealing with the sampling and testing of paints, varnishes and related products. It is also applicable to binders for paints and varnishes.

It specifies a test method for determining the non-volatile matter content by mass of paints and varnishes, as well as resins and resin solutions that are intended for use as binders for paints and varnishes.

NOTE 1 The non-volatile matter content of a product is not an absolute quantity but depends upon the temperature and period of heating used for the test. Consequently, when using this method, only relative and not true values for non-volatile matter content are obtained owing to solvent retention, thermal decomposition and evaporation of low molecular mass constituents. The method is therefore primarily intended for testing different batches of the same type of product.

ISO 3233:1984, *Paints and varnishes — Determination of volume of dry coating (non-volatile matter) obtained from a given volume of liquid coating*, specifies a test method for determining the volume of non-volatile matter in paints, varnishes and related products.

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.

1) To be published. (Revision of ISO 842:1984)

Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 842:—¹⁾, *Raw materials for paints and varnishes — Sampling*.

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*.

3 Definition

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

3.1 non-volatile matter: The residue by mass obtained by evaporation under specified conditions of test.

4 Required supplementary information

For any particular application, the test method specified in this International Standard needs to be completed by supplementary information. The items of supplementary information are given in annex A.

5 Apparatus

Ordinary laboratory apparatus, together with the following:

5.1 Flat-bottomed dish, of metal or glass, (75 ± 5) mm in diameter, height of the rim at least 5 mm. Dishes having different diameters may be used by agreement between the interested parties. The agreed dish diameter shall be adhered to ± 5 %.

ISO 3251:1993(E)

5.2 Air oven, capable of maintaining the specified or agreed temperature (see annex B) to $\pm 2\text{ }^{\circ}\text{C}$ (for temperatures up to $150\text{ }^{\circ}\text{C}$) or $\pm 3,5\text{ }^{\circ}\text{C}$ (for temperatures above $150\text{ }^{\circ}\text{C}$ and up to $200\text{ }^{\circ}\text{C}$). The air oven shall be fitted with forced-ventilation equipment and the air speed shall be within the range $0,8\text{ m/s}$ to $1,2\text{ m/s}$. A method for determining the air speed is given in annex C. The air speed shall be tested once every six months.

WARNING — To protect against explosions, it is essential that the number of dishes in relation to the floor area of the oven be such that the solvent vapour concentration cannot exceed 20 g/m^3 .

For referee tests, ovens of equivalent construction shall be used by all parties.

5.3 Analytical balance, capable of weighing to an accuracy of $0,1\text{ mg}$.

5.4 Desiccator, containing a suitable desiccant, for example dried silica gel impregnated with cobalt chloride.

6 Sampling

Take a representative sample of the product to be tested, as described in either ISO 842 (binders) or ISO 1512 (paints and varnishes).

Examine and prepare samples of paints and varnishes for testing, as described in ISO 1513.

7 Procedure

Carry out the determination in duplicate.

Degrease and clean the dish (5.1). Dry the dish in the oven (5.2) at the specified or agreed temperature for the specified or agreed period (see annex B).

Weigh, to the nearest 1 mg , a test portion of $(1 \pm 0,1)\text{ g}$ (m_0) (see note 2) into the dish and distribute it evenly (see note 3). In the case of products that are highly viscous or that form skins, distribute the test portion uniformly with a tared metal wire (for example an uncoated, bent paper-clip), if necessary after addition of 2 ml of suitable solvent.

NOTES

2 Test portions other than 1 g may be used by agreement between the interested parties. If this is the case, a test portion of not more than $(2 \pm 0,2)\text{ g}$ is recommended. It is also recommended that the dish be covered during the weighing procedure.

3 In the case of highly volatile products, it is recommended that a portion of the thoroughly mixed sample

be placed in a stoppered bottle or, alternatively, in a weighing pipette or a 10 ml syringe without a needle. From this, a test portion of $(1 \pm 0,1)\text{ g}$ is weighed by difference to the nearest 1 mg into the dish and distributed evenly over the bottom of the dish.

Allow the dish with the test portion to stand at room temperature for 10 to 15 minutes. Afterwards, transfer it to the air oven, previously brought to the specified or agreed temperature (see annex B). Leave the dish in the oven for the specified or agreed period of heating (see annex B).

When the specified or agreed period of heating is completed, transfer the dish to the desiccator (5.4) and allow to cool to room temperature. Weigh the dish and residue to the nearest 1 mg and calculate the mass of the residue (m_1).

8 Expression of results

Calculate the non-volatile matter content NV, expressed as a percentage by mass, using the equation

$$\text{NV} = \frac{m_1}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of the residue.

If the two results (duplicates) differ by more than 2 % (relative to the mean), repeat the procedure described in clause 7.

Calculate the mean of two valid results (replicates) and report the test result to the nearest 0,1 % (m/m).

9 Precision

9.1 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 2 % (relative to the mean of the two test results).

9.2 Reproducibility (R)

The value below which the absolute difference between two single test results, each the mean of duplicates, obtained on identical material by operators in different laboratories using the standardized test method, may be expected to lie with a 95 % probability is 4 % (relative to the mean of the two test results).

10 Test report

The test report shall contain at least the following information:

- a) all details necessary to identify the product tested;
- b) a reference to this International Standard (ISO 3251);
- c) the type of oven used and appropriate details, for example its effective floor area, the air-replacement rate and the length of time required to reach the test temperature;
- d) the test temperature and the period of heating;
- e) the result of the test, as indicated in clause 8;
- f) any deviation from the test method specified;
- g) the date of the test.

Annex A

(normative)

Required supplementary information

The items of supplementary information listed in this annex shall be supplied as appropriate to enable the method to be carried out.

The information required should preferably be agreed between the interested parties and may be derived, in part or totally, from an international or national

standard or other document related to the product under test.

- a) Test temperature (see annex B).
- b) Period of heating (see annex B).

Annex B

(normative)

Test conditions

The test conditions to be used shall be selected from the combinations given in table B.1.

Table B.1 — Test conditions

Period of heating	Temperature	Examples of product classes
h	°C	
1	80	Polyisocyanate resins ¹⁾
1	105	Cellulose nitrate, cellulose lacquers, air-drying paints, polyisocyanate resins ¹⁾
1	125	Synthetic resins (including polyisocyanate resins ¹⁾), stoving (baking) paints
1	150	Stoving (baking) priming paints
0,5	180	Paints for electrocoating
1) Test conditions will depend on the individual type of polyisocyanate resin to be tested.		

Annex C

(normative)

Determination of the air speed in an air oven with horizontal forced ventilation

C.1 Test conditions

The air oven shall be unheated and room temperature shall be within the range 18 °C to 28 °C.

C.2 Apparatus

C.2.1 Thermal anemometer with sensor probe (heated-wire anemometer or thermoelement anemometer), to determine low speeds up to about 2 m/s.

C.3 Procedure

Determine the usable shelf area of the oven, i.e. the total surface area available for dishes. In the case of ovens with a total shelf area of about 2 000 cm² or

less, divide the surface into nine zones. For ovens with a larger shelf area, divide the surface into 12 to 16 zones.

Take a transparent panel made of suitable heat-resistant material and of the same size as the door of the oven. Drill holes in the panel at locations such that the anemometer sensor probe can be introduced into the oven to measure the air speed at the centre of each zone, 15 mm above the shelf surface (see figure C.1).

Leaving the oven door open, close off the oven interior by placing the transparent panel over the oven doorway. Measure the air speed 15 mm above the centre of each zone.

Record the dispositions of the sensor probe, and the air speed measured at each position, in the test report.

Dimensions en millimetres

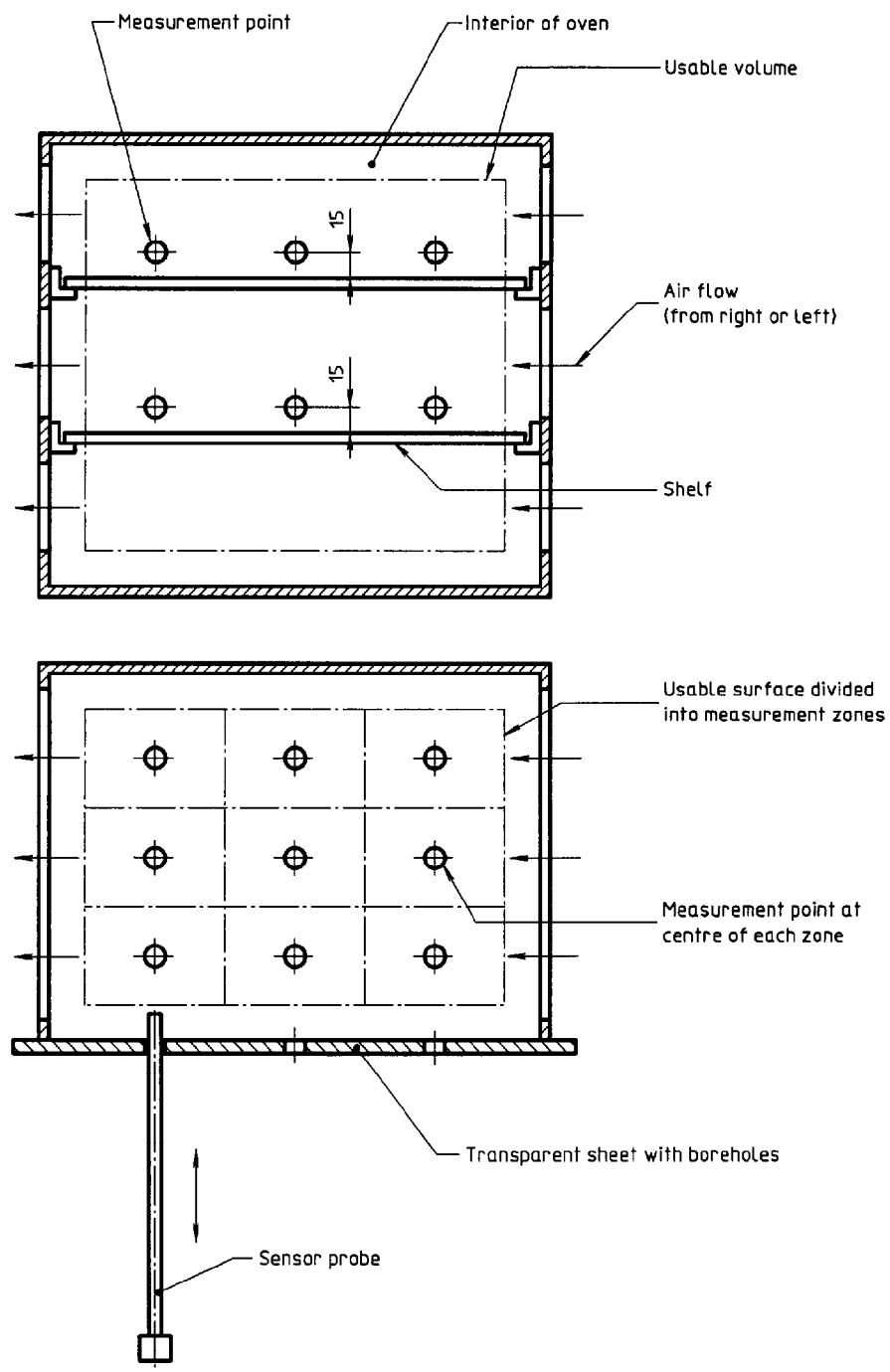


Figure C.1 — Example of the determination of air speed in an oven with horizontal ventilation

ISO 3251:1993(E)

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Descriptors: paints, varnishes, binders (materials), tests, chemical analysis, determination, non-volatile matter.

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