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Paints, varnishes and plastics — Determination of non-volatile-matter content

Peintures, vernis et plastiques — Détermination de l'extrait 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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 3251 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 10, *Test methods for binders for paints and varnishes,* in collaboration with Technical Committee CEN/TC 139, *Paints and varnishes*.

This fourth edition cancels and replaces the third edition (ISO 3251:2003), which has been technically revised. The main changes are as follows:

a) The heating time, heating temperature and test-portion mass have to be agreed in each case. The tables giving values of these parameters have therefore been moved to an informative annex where they are intended to serve as proposals. Values suitable for reactive systems such as car refinish paints have been added.

b) The precision data have been corrected.

Paints, varnishes and plastics — Determination of non-volatile-matter content

1 Scope

This International Standard specifies a method for determining the non-volatile-matter content by mass of paints, varnishes, binders for paints and varnishes, polymer dispersions and condensation resins such as phenolic resins (resols, novolak solutions, etc.).

The method is also applicable to formulated dispersions containing fillers, pigments and other auxiliaries (e.g. thickeners and film-forming agents). For the method to be usable for unplasticized polymer dispersions and rubber latices, the non-volatile residue (which consists essentially of the polymeric material and of small quantities of auxiliaries such as emulsifiers, protective colloids, stabilizers, solvents added as film-forming agents and — especially for rubber latex concentrate — preserving agents) has to be chemically stable under the test conditions. For plasticized samples, the residue, by definition, normally includes the plasticizer.

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 determination. 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.

NOTE 2 This method is suitable for synthetic rubber latices provided heating for a specific period of time is considered appropriate (ISO 124 specifies heating until the loss in mass of a 2 g test portion following successive periods of heating is less than 0,5 mg).

NOTE 3 In-house methods for determining non-volatile matter often include drying with infrared or microwave radiation. Standardization of such methods is not possible, since they are not generally applicable. Some polymer compositions tend to decompose during such treatment and therefore give incorrect results.

ISO 3233 and ISO 23811 specify methods for determining the volume of non-volatile matter in paints, varnishes and related products.

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 123, *Rubber latex — Sampling*

ISO 124, *Latex, rubber — Determination of total solids content*

ISO 1513, *Paints and varnishes — Examination and preparation of samples for testing*

ISO 2431, *Paints and varnishes — Determination of flow time by use of flow cups*

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

[ISO 4618:2006]

4 Apparatus

Ordinary laboratory apparatus, together with the following:

4.1 For paints, varnishes, binders for paints and varnishes and polymer dispersions:

Flat-bottomed dish, of metal or glass, inner diameter of base (75 ± 5) mm, height of 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 within ± 5 %.

NOTE 1 For rubber latices, lipless dishes with covers are recommended.

NOTE 2 For very viscous polymer dispersions or latices, it is recommended that aluminium foils are used which are about 0,1 mm thick, cut into rectangles of about (70 ± 10) mm × (120 ± 10) mm that can be folded in half, thus allowing the viscous liquid to be spread by gently squeezing the halves together.

4.2 For liquid crosslinking resins (phenolic resins):

Flat-bottomed dish, of metal or glass, inner diameter of base (75 ± 1) mm, height of rim at least 5 mm, for use with a test portion of 3 g.

Dishes of different diameters may be used provided the mass of the test portion m, in grams, is calculated from the following equation in order to ensure comparable film thicknesses:

$$m = 3 \times \left(\frac{d}{75} \right)^2 \quad (1)$$

where

d is the diameter, in millimetres, of the dish base;

3 is the nominal mass of the test portion (3 g);

75 is the nominal diameter of the dish (75 mm).

4.3 **Air oven**, designed to carry out the test in safe conditions, and capable of being controlled at the specified or agreed temperature (see Clause 7) to within ± 2 °C (for temperatures up to 150 °C) or ± 3,5 °C (for temperatures above 150 °C and up to 200 °C). The oven shall be fitted with forced-ventilation equipment, except in the case of phenolic resins when an oven with natural convection with a perforated metal shelf placed at one-third of the height of the oven may be used.

WARNING — To protect against explosion or fire, products containing flammable volatile substances should be handled with care. National regulations should be followed.

For certain applications, drying in a vacuum may be preferable. In such cases, the conditions shall be agreed on or the method specified in ISO 124 shall be used. For referee tests, ovens of equivalent construction shall be used by all parties.

4.4 Analytical balance, capable of weighing to an accuracy of 0,000 1 g.

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

5 Sampling

Take a representative sample of paints, varnishes and binders for paints and varnishes, as described in ISO 15528. Take a representative sample of polymer dispersions and rubber latices, as described in ISO 123.

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

6 Procedure

Carry out the determination in duplicate.

Degrease and clean a dish (4.1 or 4.2).

For better precision, it is recommended that the dish is dried in the oven (4.3) at the specified or agreed temperature for the specified or agreed period (see Clause 7) and stored in the desiccator (4.5) until used.

Determine the mass of the clean, dry dish (m_0) to the nearest 1 mg. Weigh a test portion (see Clause 7), to the nearest 1 mg, into the dish (m_1) and distribute it evenly. In the case of products that are highly viscous (viscosity $v \geqslant 500$ mPa·s at a shear rate of 100 s^{-1} or flow time $t \geqslant 74$ s measured with a 6 mm flow cup in accordance with ISO 2431) or that form skins, distribute the test portion uniformly with a tared metal wire (for example a bent, uncoated paper-clip), if necessary after addition of 2 ml of a suitable solvent.

Condensation resins as used for paints and varnishes and other common applications (for example abrasives, friction linings, foundry binders and moulding materials) require higher test-portion masses since materials used for these applications need to be tested in thicker layers so that the monomers of the condensation resins can react during crosslinking. For comparative tests, the thickness of the layer of test portion in the dish shall be constant. Therefore the diameter of the dishes shall be (75 ± 1) mm or the formula given in 4.2 shall be used.

NOTE 1 The non-volatile-matter content of a test portion is influenced greatly by how well and for how long the test portion is distributed in the dish. If a test portion is poorly distributed, e.g. because of high viscosity, the apparent non-volatile-matter content will be higher.

For better precision when testing paints, varnishes and binders for paints and varnishes, it is recommended that 2 ml of a suitable highly volatile solvent is always added.

It is also recommended that the dish is covered during the weighing procedure.

In the case of highly volatile products, it is recommended that a portion of the thoroughly mixed sample is placed in a stoppered bottle or, alternatively, in a weighing pipette or a 10 ml syringe without a needle. From this, the test portion is weighed by difference, to the nearest 1 mg, into the dish and distributed evenly over the bottom of the dish.

If solvent is added, it is recommended that the dish with the test portion is allowed to stand at room temperature for 10 min to 15 min.

Aqueous systems such as polymer dispersions and rubber latices splash when heated, due to surface skinning which could also be influenced by the temperature, the airflow in the oven and possibly the relative

humidity. In such cases, the thickness of the layer of material in the dish shall therefore be kept as low as possible.

After weighing and addition of solvent, transfer the dish to the oven, previously brought to the specified or agreed temperature (see Clause 7). Leave the dish in the oven for the specified or agreed period (see Clause 7).

When the period of heating is completed, transfer the dish to the desiccator and allow to cool to room temperature or, optionally, place the dish in a dust-free atmosphere to cool down.

NOTE 2 The precision of the method can be affected by not using a desiccator.

Weigh the dish and residue (m_2) to the nearest 1 mg.

7 Supplementary test conditions

For any particular application of the method specified in this International Standard, more details in addition to those in the preceding clauses may need to be given.

To enable the method to be carried out, the following test parameters shall be specified, as appropriate:

a) the test temperature;

b) the period of heating;

c) the mass of the test portion.

Commonly used values of these parameters are given in Annex A.

8 Expression of results

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

$$\mathrm{NV} = \frac{(m_2 - m_0)}{(m_1 - m_0)} \times 100 \qquad (2)$$

where

m_0 is the mass, in grams, of the empty dish;

m_1 is the mass, in grams, of the dish with the test portion;

m_2 is the mass, in grams, of the dish with the residue.

If the two results (duplicates) differ by more than 2 % (relative to the mean) for paints, varnishes and binders or by more than 0,5 % for polymer dispersions, e.g. if they are 53,7 % and 53,1 %, repeat the procedure described in Clause 6.

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

9 Precision

9.1 Repeatability limit r

The repeatability limit r is the value below which the absolute difference between two single test results, each the mean of duplicates, can be expected to lie when this method is used under repeatability conditions. In such cases, the test results are obtained on identical material by one operator in one laboratory within a short interval of time using the standardized test method. In this International Standard, r is

2 % (absolute) for paints, varnishes and binders,

0,6 % (absolute) for polymer dispersions,

with a 95 % probability.

9.2 Reproducibility limit R

The reproducibility limit R is the value below which the absolute difference between two single test results, each the mean of duplicates, can be expected to lie when this test method is used under reproducibility conditions. In such cases, the test results are obtained on identical material by operators in different laboratories using the standardized test method. In this International Standard, R is

4 % (absolute) for paints, varnishes and binders,

1 % (absolute) for polymer dispersions,

with a 95 % probability.

10 Test report

The test report shall contain at least the following information:

a) a reference to this International Standard (ISO 3251);

b) all details necessary for complete identification of the product tested (manufacturer, trade name, batch number, etc.);

c) the type of dish used;

d) the type of oven used;

e) the oven temperature and the period of heating;

f) the type of solvent added (if applicable);

g) the result of the test, as indicated in Clause 8;

h) any deviation from the method specified;

i) the date of the test.