SLOVENSKI STANDARD
oSIST prEN ISO 3251:2018

01-september-2018

Barve, laki in plastične mase - Določevanje nehlapnih snovi (ISO/DIS 3251:2018)

Paints, varnishes and plastics - Determination of non-volatile-matter content (ISO/DIS 3251:2018)

Beschichtungsstoffe und Kunststoffe - Bestimmung des Gehaltes an nichtflüchtigen Anteilen (ISO/DIS 3251:2018)

Peintures, vernis et plastiques - Détermination de l'extrait sec (ISO/DIS 3251:2018)

Ta slovenski standard je istoveten z: **prEN ISO 3251**

ICS:

83.080.01	Polimerni materiali na splošno	Plastics in general
87.040	Barve in laki	Paints and varnishes

oSIST prEN ISO 3251:2018 **en,fr,de**

DRAFT INTERNATIONAL STANDARD

ISO/DIS 3251

ISO/TC **35**/SC **9** Secretariat: **BSI**

Voting begins on: **2018-07-09**

Voting terminates on: **2018-10-01**

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

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

ICS: 83.080.01; 87.040; 87.060.20

This document is circulated as received from the committee secretariat.

ISO/CEN PARALLEL PROCESSING

Reference number
ISO/DIS 3251:2018(E)

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Published in Switzerland

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

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

This fifth edition cancels and replaces the fourth edition (ISO 3251:2008), which has been technically revised.

The main changes compared to the previous edition are as follows:

— a general reference to ISO 4618 on terms and definitions has been added to Clause 3;

— the definition of non-volatile matter has been updated in accordance with the latest edition of ISO 4618 (i.e. ISO 4618:2012);

— the example of the desiccant in 4.5 has been changed to silica gel orange because the use of cobalt chloride as indicator is no longer allowed;

— the precision data of polymer dispersions has been corrected: the figures given in the 2008 edition were ± data which now have been converted correctly into percentages;

— the common test parameters for coating powders (powder resins) have been deleted from Table A.1 because ISO 8130-7 can be used instead;

— Common test parameters for waterborne coating materials have been added to Table A.1.

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

1 Scope

This document 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, film-forming agents). For the method to be usable for unplasticized polymer dispersions and rubber lattices, 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. Several polymer compositions tend to decompose during such treatment and therefore give incorrect results.

ISO 3233 (all parts) specifies test methods for determining the volume of non-volatile matter in paints, varnishes and related products.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 test samples*

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

ISO 4618:2014, *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.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

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

Note 1 to entry: Instead of the term "non-volatile matter" different terms, such as solid, dry residue, dry matter, solid matter, stoving residue are being used commonly with the respective abbreviations. The term "non-volatile matter" which is also applied in ISO 3251 should be used together with the abbreviation "NV" instead of these terms.

[SOURCE: ISO 4618:2014, 2.176]

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 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 %.

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 the rim at least 5 mm, for a test portion of 3 g.

For obtaining comparable film thicknesses, dishes of different diameters may be used provided the mass of the test portion m, in grams, is calculated from Equation (1):

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

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) ± 2 °C (for temperatures up to 150 °C) or ± 3,5 °C (for temperatures above 150 °C and up to 200 °C). The air oven shall be fitted with forced-ventilation equipment, except 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 orange.

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 ($\nu \geq 500$ mPa · s or flow time $t \geq 74$ 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 an uncoated, bent 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, 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 temperature, airflow in the oven and possibly 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.

8 Expression of results

Calculate the non-volatile-matter content NV, expressed as a percentage by mass, using Equation (2):

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

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

