

Paints, varnishes and plastics
 Determination of non-volatile-matter content
 (ISO 3251 : 2003)
 English version of DIN EN ISO 3251

DIN

EN ISO 3251

ICS 87.040; 83.080.01

Supersedes September 1995
 edition, DIN ISO 1625, April 2001
 edition, and DIN EN ISO 8618,
 August 1998 edition.

Beschichtungsstoffe und Kunststoffe – Bestimmung des Gehaltes an
 nichtflüchtigen Anteilen (ISO 3251 : 2003)

European Standard EN ISO 3251 : 2003 has the status of a DIN Standard.

A comma is used as the decimal marker.

National foreword

This standard has been published in accordance with a decision taken by CEN/TC 139 to adopt, without alteration, International Standard ISO 3251 as a European Standard.

The responsible German body involved in its preparation was the *Normenausschuss Beschichtungsstoffe und Beschichtungen* (Coatings and Coating Materials Standards Committee), Technical Committees *Lackrohstoffe* and *Allgemeine Prüfverfahren für Beschichtungsstoffe und Beschichtungen*.

The DIN Standards corresponding to the International Standards referred to in clause 2 of the EN are as follows:

ISO Standard	DIN Standard
ISO 123	DIN ISO 123
ISO 124	DIN 53563
ISO 1513	DIN EN ISO 1513
ISO 2431	DIN EN ISO 2431
ISO 15528	DIN EN ISO 15528

Amendments

DIN EN ISO 3251, September 1995 edition, DIN EN ISO 8618, August 1998 edition, and DIN ISO 1625, April 2001 edition, have been combined into one standard.

Previous editions

DIN 53189: 1958-02, 1972-01; DIN 53216: 1972-05; DIN 53216-1: 1983-04; DIN V 53216-1: 1988-05;
 DIN EN ISO 3251: 1995-09; DIN ISO 1625: 2001-04; DIN ISO 8618: 1991-03, 1998-08.

Continued overleaf.

EN comprises 9 pages.

National Annex NA

Standards referred to

(and not included in **Normative references** and **Annex ZA**)

DIN 53563	Determination of total solids content of latex
DIN EN ISO 1513	Paints and varnishes – Examination and preparation of samples (ISO 1513 : 1992)
DIN EN ISO 2431	Determination of flow time of paints and varnishes by use of flow cups (ISO 2431 : 1993 + Corr 1 : 1994)
DIN EN ISO 15528	Paints, varnishes and raw materials for paints and varnishes – Sampling (ISO 15528 : 2000)
DIN ISO 123	Rubber latex – Sampling (ISO 123 : 2001)

English version

Paints, varnishes and plastics
Determination of non-volatile-matter content
(ISO 3251 : 2003)

Peintures, vernis et plastiques – Détermination de l'extrait sec
(ISO 3251 : 2003)

Beschichtungsstoffe und Kunststoffe – Bestimmung des Gehaltes an nichtflüchtigen Anteilen (ISO 3251 : 2003)

This European Standard was approved by CEN on 2003-02-13.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Management Centre or to any CEN member.

The European Standards exist in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, the Czech Republic, Denmark, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, the Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland, and the United Kingdom.

CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Management Centre: rue de Stassart 36, B-1050 Brussels

Foreword

International Standard

ISO 3251 : 2003 Paints, varnishes and plastics – Determination of non-volatile-matter content, which was prepared by ISO/TC 35 'Paints and varnishes' of the International Organization for Standardization, has been adopted by Technical Committee CEN/TC 139 'Paints and varnishes', the Secretariat of which is held by DIN, as a European Standard.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, and conflicting national standards withdrawn, by August 2003 at the latest.

In accordance with the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard:

Austria, Belgium, the Czech Republic, Denmark, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, the Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland, and the United Kingdom.

Endorsement notice

The text of the International Standard ISO 3251 : 2003 was approved by CEN as a European Standard without any modification.

NOTE: Normative references to international publications are listed in Annex ZA (normative).

Contents	Page
Foreword	2
1 Scope	3
2 Normative references	3
3 Terms and definitions	4
4 Apparatus	4
5 Sampling	5
6 Procedure	5
7 Supplementary test conditions	6
8 Expression of results	7
9 Precision	8
10 Test report	8

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 of the 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 3233:1998, *Paints and varnishes — Determination of percentage volume of non-volatile matter by measuring the density of a dried coating*, specifies a method 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

the percentage residue by mass obtained by evaporation under specified conditions

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, (75 ± 5) mm in diameter, 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 to within $\pm 5\%$.

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 be used which are about 0,1 mm thick, cut into rectangles of about (70 ± 10) mm \times (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.

Dishes of different diameters may be used provided the mass of the test portion m , in grams, is calculated from the following equation:

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

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 maintaining the specified or agreed temperature (see Clause 7) to within $\pm 2\text{ }^{\circ}\text{C}$ (for temperatures up to $150\text{ }^{\circ}\text{C}$) or to within $\pm 3,5\text{ }^{\circ}\text{C}$ (for temperatures above $150\text{ }^{\circ}\text{C}$ and up to $200\text{ }^{\circ}\text{C}$). 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 0,1 mg.

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 be 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 $\geq 500 \text{ mPa}\cdot\text{s}$ at a shear rate of 100 s^{-1} or flow time $t \geq 74 \text{ s}$ 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, 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 $(75 \pm 1) \text{ mm}$, 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 be 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, the air flow 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 (see Tables 1 and 2);
- b) the period of heating (see Tables 1 and 2);
- c) the mass of the test portion (see Tables 1 and 2).

Table 1 — Test parameters for paints, varnishes, binders for paints and varnishes and liquid phenolic resins

Period of heating min	Temperature °C	Mass of test portion g	Examples of product classes
20	200	1 ± 0,1 ^a	Powder resins
60	80	1 ± 0,1 ^a	Cellulose nitrate, cellulose nitrate lacquers, polyisocyanate resins ^b
60	105	1 ± 0,1 ^a	Cellulose derivatives, cellulose paints and lacquers, air-drying paints, polyisocyanate resins ^b
60	125	1 ± 0,1 ^a	Synthetic resins (including polyisocyanate resins ^b), stoving (baking) paints, acrylate resins (preferred conditions)
60	150	1 ± 0,1 ^a	Stoving (baking) priming paints, acrylate resins
30	180	1 ± 0,1 ^a	Paints for electrocoating
60	135 ^c	3 ± 0,5	Liquid phenolic resins

^a 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 ± 0,2) g is recommended. For resins containing solvents with boiling points of 160 °C to 200 °C, an oven temperature of 160 °C is recommended. If even higher boiling solvents are present, the conditions shall be agreed between the interested parties.

^b The test parameters will depend on the individual type of polyisocyanate resin under test.

^c An alternative temperature may be used. Recommended alternative temperatures are 120 °C and 150 °C.

Table 2 — Test parameters for polymer dispersions

Period of heating min	Temperature °C	Mass of test portion g	Method ^a
120	80	1 ± 0,2 ^b	A
60	105	1 ± 0,2 ^b	B
60	125	1 ± 0,2 ^b	C
30	140	1 ± 0,2 ^b	D

^a The conditions to be used will depend on the type of polymer dispersion or latex under test and shall be selected by agreement between the interested parties.

^b Test portions other than 1 g may be used by agreement between the interested parties. However, the size of the test portion shall not exceed 2,5 g.

Test portions of 0,2 g to 0,4 g, weighed to the nearest 0,1 mg, may also be used. In this case, the period of heating can be reduced provided it has been established (by measurements on the type of dispersion under test) that the same results are obtained as under the conditions given in this table.

8 Expression of results

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

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

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 % (relative to the mean of the two test results) for paints, varnishes and binders,

0,6 % (relative to the mean of the two test results) 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 % (relative to the mean of the two test results) for paints, varnishes and binders,

1 % (relative to the mean of the two test results) 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.

Annex ZA (normative)

Normative references to international publications with their relevant European publications

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

NOTE Where an International Publication has been modified by common modifications, indicated by (mod.), the relevant EN/HD applies.

<u>Publication</u>	<u>Year</u>	<u>Title</u>	<u>EN</u>	<u>Year</u>
ISO 1513	1992	Paints and varnishes — Examination and preparation of samples for testing	EN ISO 1513	1994
ISO 2431	1993	Paints and varnishes — Determination of flow time by use of flow cups	EN ISO 2431	1996
ISO 15528	2000	Paints, varnishes and raw materials for paints and varnishes — Sampling	EN ISO 15528	2000