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Liants pour peintures et vernis - Résines époxydiques - Méthodes générales d'essai (ISO 7142:2007)

Binders for paints and varnishes - Epoxy resins - General methods of test (ISO 7142:2007)

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Foreword

This document (EN ISO 7142:2007) has been prepared by Technical Committee ISO/TC 35 "Paints and varnishes" in collaboration with Technical Committee CEN/TC 139 "Paints and varnishes", the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by August 2007, and conflicting national standards shall be withdrawn at the latest by August 2007.

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Endorsement notice

The text of ISO 7142:2007 has been approved by CEN as EN ISO 7142:2007 without any modifications.

INTERNATIONAL STANDARD

ISO 7142

Second edition
2007-02-01

Binders for paints and varnishes — Epoxy resins — General methods of test

Liants pour peintures et vernis — Résines époxydiques — Méthodes générales d'essai

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Foreword

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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 7142 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 CEN Technical Committee CEN/TC 139, *Paints and varnishes*.

This second edition cancels and replaces the first edition (ISO 7142:1984), which has been editorially revised and the normative references updated.

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Binders for paints and varnishes — Epoxy resins — General methods of test

1 Scope

This International Standard specifies general methods of test for epoxy resins for use in paints, varnishes and similar products. It is also applicable to those solutions made from epoxy resins that are intended for use as binders for paints and varnishes.

The test methods to be applied to an individual epoxy resin shall be the subject of agreement between the interested parties.

The test methods described in this International Standard are not intended for epoxy esters (see Note to 3.1).

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 385, *Laboratory glassware — Burettes*

ISO 648, *Laboratory glassware — One-mark pipettes*

ISO 1523, *Determination of flash point — Closed cup equilibrium method*

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

ISO 3001, *Plastics — Epoxy compounds — Determination of epoxy equivalent*

ISO 3146, *Plastics — Determination of melting behaviour (melting temperature or melting range) of semi-crystalline polymers by capillary tube and polarizing-microscope methods*

ISO 3219, *Plastics — Polymers/resins in the liquid state or as emulsions or dispersions — Determination of viscosity using a rotational viscometer with defined shear rate*

ISO 3251, *Paints, varnishes and plastics — Determination of non-volatile-matter content*

ISO 3679, *Determination of flash point — Rapid equilibrium closed cup method*

ISO 4615, *Plastics — Unsaturated polyesters and epoxide resins — Determination of total chlorine content*

ISO 4625-1, *Binders for paints and varnishes — Determination of softening point — Part 1: Ring-and-ball method*

ISO 4625-2, *Binders for paints and varnishes — Determination of softening point — Part 2: Cup-and-ball method*

ISO 4630-1, *Clear liquids — Estimation of colour by the Gardner colour scale — Part 1: Visual method*

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ISO 4630-2, *Clear liquids — Estimation of colour by the Gardner colour scale — Part 2: Spectrophotometric method*

ISO 6271-1, *Clear liquids — Estimation of colour by the platinum-cobalt scale — Part 1: Visual method*

ISO 6271-2, *Clear liquids — Estimation of colour by the platinum-cobalt scale — Part 2: Spectrophotometric method*

ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

ISO 21627-2, *Plastics — Epoxy resins — Determination of chlorine content — Part 2: Easily saponifiable chlorine*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
epoxy resin
synthetic resin containing epoxy groups generally prepared from epichlorhydrin and a bisphenol

[ISO 4618:2006]

NOTE Epoxy esters obtained by reacting materials containing epoxy groups with fatty acids or oils which dry by oxidation are not covered by this International Standard.

4 Sampling

Take a representative sample of the product to be tested, as described in ISO 15528.

5 Test methods

See Table 1.

6 Test report

The test report shall contain at least the following information:

a) the type and identification of the product tested;

b) a reference to this International Standard (ISO 7142:2007);

c) the results of the tests, and the methods used;

d) any deviation, by agreement or otherwise, from the procedures specified;

e) the dates of the tests.

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Table 1 — Properties and methods of test

Property	Test method
Colour	ISO 4630-1 or ISO 4630-2 (Gardner colour scale) or ISO 6271-1 or ISO 6271-2 (Platinum-cobalt scale)
Viscosity [a] [b]	ISO 3219 or another agreed method
Volatile or non-volatile matter	ISO 3251 [c]
Flashpoint [a]	ISO 1523 ISO 3679
Epoxy equivalent	ISO 3001
Hydroxyl value	Annex A
Total chlorine content	ISO 4615
Easily saponifiable chlorine	ISO 21627-2
Melting point	ISO 3146
Softening point	ISO 4625-1 or ISO 4625-2

[a] For epoxy resin solutions, liquid resins and resins in test solutions only. Solid epoxy resins shall be tested in a standard test solution of 40 % (by mass) in diethylene glycol mono-*n*-butyl ether. The solvent used and the concentration of the test solution shall be indicated in the test report.

[b] If the flow-time is used for the characterization of the flow behaviour, it shall be determined by the method specified in ISO 2431.

[c] Take a test portion of 5 g and heat it at 140 °C for 3 h without addition of solvent, as described in ISO 3251.

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Annex A
(normative)

Hydroxyl value

A.1 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

WARNING — Take all necessary safety precautions when using the reagents A.1.1 to A.1.3.

A.1.1 Pyridine.

The pyridine shall be clear and shall not cause any significant colour during the blank test.

A.1.2 Pyridinium perchlorate.

Slowly add 144 g of 70 % (by mass) perchloric acid to 120 ml of pyridine, keeping the mixture cool. Recrystallize the precipitate twice in hot water and allow it to dry in air.

A.1.3 Acetic anhydride/pyridine mixture.

Mix 12 g of 95 % (by mass) acetic anhydride with 88 g of pyridine.

Store the mixture in an airtight bottle and in the dark.

A.1.4 Potassium hydroxide, methanolic standard volumetric solution, c(KOH) = 1 mol/l.

A.1.5 Phenolphthalein, 10 g/l solution in 95 % (by volume) ethanol, methanol or 2-propanol.

A.2 Apparatus

Ordinary laboratory apparatus and

A.2.1 Conical flask, of capacity about 250 ml, fitted with a ground-glass joint.

A.2.2 Reflux condenser, with ground-glass joint, fitting on the conical flask (A.2.1).

A.2.3 Burette, of capacity 50 ml, complying with the requirements of ISO 385, for the potassium hydroxide solution (A.1.4).

A.2.4 Pipette, of capacity 25 ml, complying with the requirements of ISO 648.

A.2.5 Suitable heating device.

A.3 Procedure

A.3.1 General

Carry out the determination in duplicate.

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A.3.2 Test portion

Select the mass of the test portion according to the expected epoxy equivalent (see Table A.1).

Weigh, to the nearest 1 mg, the test portion into the conical flask (A.2.1).

Table A.1 — Mass of test portion

Epoxy equivalent	Mass of test portion g	Mass of pyridinium perchlorate g
up to 180	2,5	4,00
above 180 to 195		3,50
above 195 to 215		3,25
above 215 to 240		3,00
above 240 to 290	3,0	3,00
above 290 to 350		2,50
above 350 to 425		2,00
above 425 to 515		1,75
above 515 to 650		1,35
above 650 to 760		1,10
above 760		1,00

A.3.3 Determination

Weigh the corresponding mass of pyridinium perchlorate (A.1.2) given in Table A.1 into the conical flask (A.2.1). Add by the pipette (A.2.4) 25 ml of acetic anhydride/pyridine mixture (A.1.3). Warm the mixture until the test portion is completely dissolved. Fit the reflux condenser (A.2.2) onto the conical flask, heat to boiling and maintain at the boiling point under reflux for 30 min.

Add 2 ml of water and 10 ml to 15 ml of pyridine (A.1.1) to the top of the condenser so as to rinse the condenser tube. Mix the contents of the flask and cool to ambient temperature.

Add 3 drops of phenolphthalein solution (A.1.5) and titrate with potassium hydroxide solution (A.1.4).

A.3.4 Blank test

Carry out a blank test, following the same procedure but omitting the test portion and the pyridinium perchlorate (A.1.2).

A.4 Expression of results

A.4.1 Calculation

Calculate the hydroxyl equivalent by the equation

$$\mathrm{HE} = \frac{1\,000}{\dfrac{5{,}569 \times m_2 + (V_0 - V_1)}{m_1} - \dfrac{2\,000}{\mathrm{EE}}}$$

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Calculate the hydroxyl value by the equation

$$\mathrm{HV} = \frac{56{,}109 \times 1000}{\mathrm{HE}} = \frac{56\,109}{\mathrm{HE}}$$

where

EE is the epoxy equivalent, in grams per mole, determined by the method specified in ISO 3001;

HE is the hydroxyl equivalent, in grams of resin per equivalent of hydroxyl group (OH);

HV is the hydroxyl value, in grams of potassium hydroxide per kilogram;

m_1 is the mass, in grams, of the test portion (A.3.2);

m_2 is the mass, in grams, of pyridinium perchlorate (A.1.2) used;

V_0 is the volume, in millilitres, of potassium hydroxide solution (A.1.4) required for the blank test (A.3.4);

V_1 is the volume, in millilitres, of the potassium hydroxide solution (A.1.4) required for the determination (A.3.3).

A.4.2 Precision

A.4.2.1 Repeatability (r)

The value below which the absolute difference between two single test results, obtained on identical material by one operator in one laboratory using the same equipment within a short interval of time using the standardized test method, may be expected to lie with a 95 % probability is 10 %.

A.4.2.2 Reproducibility (R)

The value below which the absolute difference between two single test results, obtained on identical material by operators in different laboratories using the standardized test method, may be expected to lie with a 95 % probability is 20 %.

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Bibliography

[1] ISO 4618:2006, *Paints and varnishes — Terms and definitions*

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