International Standard



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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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Descriptors: paints, varnishes, binders (materials), epoxy resins, tests.

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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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 7142 was prepared by Technical Committee ISO/TC35, Paints and varnishes.

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

Scope and field of application

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

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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 clause 3).

2 References

ISO 385/1, Laboratory glassware — Burettes — Part 1: General requirements.

ISO 648, Laboratory glassware — One-mark pipettes.

ISO 842, Raw materials for paints and varnishes — Sampling.

ISO 1218, Plastics — Polyamides — Determination of "melting point".

ISO 1523, Paints and varnishes, petroleum and related products — Determination of flashpoint — Closed cup equilibrium method.

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

ISO 3001, Plastics — Epoxide compounds — Determination of epoxide equivalent.

ISO 3219, Plastics — Polymers in the liquid, emulsified or dispersed state — Determination of viscosity with a rotational viscometer working at defined shear rate.

ISO 3251, Paint media — Determination of volatile and nonvolatile matter.

150 3679, Paints and varnishes, petroleum and related products 4. Determination of flashpoint — Rapid equilibrium method.

ISO 4583, Plastics — Epoxide resins and related materials — Determination of easily saponifiable chlorine.

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

ISO 4625, Binders for paints and varnishes — Determination of softening point — Ring and ball method.

ISO 4630, Binders for paints and varnishes — Estimation of colour of clear liquids by the Gardner colour scale.

ISO 6271, Clear liquids — Estimation of colour by the platinum-cobalt scale.

3 Definition

epoxy resin: Synthetic resin containing epoxy groups.

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 842.

5 Methods of test

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);
- 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.

Table 1 - Properties and methods of test

| Property | Method of test |
|--|---|
| Colour | ISO 4630 (Gardner colour scale) or ISO 6271 (Platinum-cobalt scale) |
| Viscosity ^{1) 2)} | ISO 3219 or other agreed methods |
| Volatile or non-volatile matter | ISO 3251 ³⁾ |
| Flashpoint ¹⁾ | ISO 1523 ISO 3679 |
| Epoxy equivalent | ISO 3001 |
| Hydroxyl value ITEN STAND | Annex PREVIEW |
| Total chlorine content | ISO 4615 |
| Easily saponifiable chlorine | 150 4583 |
| Melting point | ISO 1218 |
| Softening point https://standards.itch.gi/cotalog/stay | 150 4625 tdord/gst//2608200 h102 4502 8106 |

¹⁾ For epoxy resin solutions, liquid resins and resins in test solutions only. Solid epoxy resins should be tested in a standard test solution of 40 % (m/m) in diethylene glycol mono-n-butylether. The solvent used and the concentration of the test solution should be indicated in the test report.

²⁾ If the flow-time is used for the characterization of the flow behaviour, it should be determined by the method specified in ISO 2431.

³⁾ 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.

Annex

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 a significant colour during the blank test.

A.1.2 Pyridinium perchlorate.

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

A.1.3 Acetic anhydride/pyridine mixturetandards.i

Mix 12 g of 95 % (m/m) acetic anhydride with 88 g of pyridine.

Store the mixture in an airtight bottle and in the dark. Standards/sis fa98678ab6f0/iso-71

- **A.1.4 Potassium hydroxide**, methanolic standard volumetric solution, c(KOH) = 1 mol/I.
- **A.1.5** Phenolphthalein, 10 g/l solution in 95 % (V/V) 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 385/1, 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

Carry out the determination in duplicate.

A.3.1 Test portion

Select the mass of the test portion according to the expected epoxy equivalent (see table 2).

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

Table 2 — Mass of test portion

| Epoxy equivalent | Mass of test portion | Mass of pyridinium perchlorate |
|---------------------|----------------------|-----------------------------------|
| | g | g |
| up to 180/ | W | 4,00 |
| above 180 to 195 | 2,5 | 3,50 |
| above 195 to 215 | | 3,25 |
| above 215 to 240 | | 3,00 |
| 4 above 240 to 290 | | 3,00 |
| 4above 290 to 35045 | 02-8106- | 2,50 |
| above 350 to 425 | | 2,00 |
| above 425 to 515 | 3,0 | 1,75 |
| above 515 to 650 | | 1,35 |
| above 650 to 760 | | 1,10 |
| above 760 | | 1,00 |

A.3.2 Determination

Weigh the corresponding mass of the pyridinium perchlorate (A.1.2) given in table 2 into the conical flask (A.2.1). Add by the pipette (A.2.4) 25 ml of the 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 and heat to boiling and maintain at the boiling point under reflux for 30 min.

Add 2 ml of water and 10 to 15 ml of the 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 the phenolphthalein solution (A.1.5) and titrate with the potassium hydroxide solution (A.1.4).

A.3.3 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

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

Calculate the hydroxyl value by the equation

$$HV = \frac{56,109 \times 1000}{HE} = \frac{56109}{HE}$$

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.3.1);

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

 V_0 is the volume, in millilitres, of the potassium hydroxide solution (A.1.4), required for the blank test (A.3.3);

 V_1 is the volume, in millilitres, of the potassium hydroxide solution (A.1.4), required for the determination (A.3.2).

A.4.2 Precision

A.4.2.1 Repeatability (r)

The value below which the absolute difference between two single test results, on identical material, obtained 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, on identical material, obtained by operators in different laboratories, using the standardized test method, (A.3.1);

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