INTERNATIONAL STANDARD

ISO 11668

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Binders for paints and varnishes — Chlorinated polymerization resins — General methods of test

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

Reference number
ISO 11668:1997(E)

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.

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.

International Standard ISO 11668 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, subcommittee SC 10, *Test methods for binders for paints and varnishes*.

Annex A forms an integral part of this International Standard.

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Binders for paints and varnishes – Chlorinated polymerization resins – General methods of test

1 Scope

This International Standard describes general methods of test for
a) chlorinated rubber and
b) vinyl chloride copolymers
for use in paints, varnishes and similar products.

The test methods to be applied in each individual case shall be the subject of agreement between the interested parties.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 842:1984, *Raw materials for paints and varnishes – Sampling.*

ISO 1158:—[1], *Plastics – Vinyl chloride homopolymers and copolymers – Determination of chlorine content.*

ISO 3219:1993, *Plastics – Polymers/resins in the liquid state or as emulsions or dispersions – Determination of viscosity using a rotational viscometer with defined shear rate.*

ISO 4630:1997, *Binders for paints and varnishes – Estimation of colour of clear liquids by the Gardner colour scale.*

ISO 6271:1997, *Clear liquids – Estimation of colour by the platinum-cobalt scale.*

ISO 8130-2:1992, *Coating powders – Part 2: Determination of density by gas comparison pyknometer (referee method).*

ISO 12058-1:1997, *Plastics – Determination of viscosity using a falling-ball viscometer – Part 1: Inclined-tube method.*

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1) To be published. (Revision of ISO 1158:1984)

3 Definitions

For the purposes of this International Standard the following definitions apply.

3.1 chlorinated rubber: Resin resulting from the action of chlorine on polyisoprene, natural rubber or similar polymers and containing chlorine contents of 64 % to 68 % (*m/m*).

3.2 vinyl chloride copolymer: Resin resulting from the polymerization of vinyl chloride together with other monomers and containing predominantly polyvinyl chloride.

4 Sampling

Take a representative sample of the product to be tested, as described in ISO 842.

5 Test methods

5.1 Colour number

For the determination of the colour number dissolve the resin.

Determine the colour number of the resin solution as described in ISO 4630 (Gardner colour scale). For resin solutions with a Gardner colour number less than 1 determine the colour as described in ISO 6271 (Platinum-cobalt scale).

The test method, the solvent used and the concentration of the resin solution shall be given in the test report.

5.2 Viscosity

Determine the viscosity of the resin solution as described in ISO 3219.

Indicate the solution temperature and the time period from dissolving the resin until the determination because the viscosity of some polymer solutions depends on this time. For referee methods wait at least 24 h before determining the viscosity.

NOTE It should be borne in mind that not only the temperature but also the intensity of stirring (rate of shear), the duration of the dissolving process and the extent of distribution of the polymer in the solvent when beginning the dissolving process influences the viscosity of the solution.

The viscosity may be determined using a falling-ball viscometer as described in ISO 12058-1 if agreed between the interested parties.

5.3 Density

Determine the density of the resin with a gas comparison pyknometer as described in ISO 8130-2.

5.4 Chlorine content

5.4.1 Chlorine content > 50 % (*m/m*)

Determine the chlorine content as described in ISO 1158.

5.4.2 Chlorine content ≤ 50 % (*m/m*)

Determine the chlorine content as described in annex A.

6 Test report

The test report shall contain at least the following information:

a) all details necessary to identify the product tested;

b) a reference to this International Standard (ISO 11668);

c) the colour (Gardner colour number or Platinum-cobalt colour number), the solvent used and the concentration of the solution;

d) the viscosity, the method used, the solvent or thinner used, the concentration of the solution, the temperature of the solution and the time between dissolving the resin and the determination;

e) the density;

f) the chlorine content and the method used;

g) any deviations from the procedures specified;

h) the dates of the tests.

Annex A
(normative)
Determination of chlorine content – Decomposition by the Wickbold method

A.1 Test apparatus
General laboratory apparatus, and

A.1.1 Wickbold combustion apparatus
(see figure A.1)

A.1.2 Bottled hydrogen, oxygen and nitrogen (commercial quality). Bottles must have pressure reducing valves.

A.1.3 Balance with 0,1 mg divisions

A.1.4 Gas burner

A.1.5 250 ml or 100 ml measuring flasks

A.2 Reagents

A.2.1 Absorption solution.

Sodium hydroxide solution, *c* approximately 0,1 mol/l.

Add 5 to 7 drops of 30 % (*m/m*) hydrogen peroxide solution to every litre of sodium hydroxide solution.

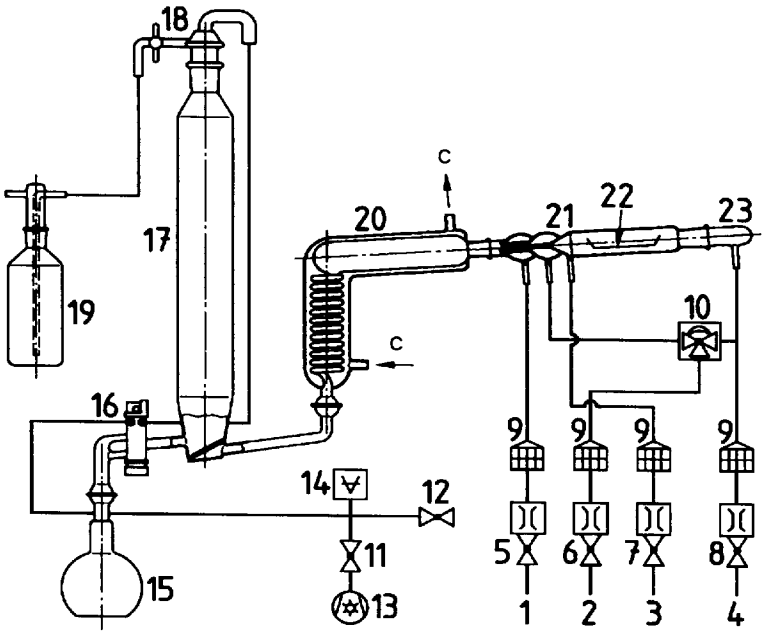
A.2.2 Nitric acid, diluted 1 + 1.

Mix 1 part by volume of nitric acid (ρ = 1,40 g/ml) with 1 part by volume of water.

A.3 Procedure

A.3.1 Safety

- The normal safety regulations concerning gas supply equipment must be observed, especially when handling hydrogen and oxygen.
- The ignition flame must always be adjusted in the presence of excess oxygen.
- A shield made from safety glass or fine wire-mesh must be placed in front of the burner and the combustion chamber.
- Special care must be taken not to allow hydrogen to enter the combustion chamber when the apparatus is being flushed with hydrogen.
- Spectacles with UV filters must be worn.



1 Hydrogen supply
2 Oxygen supply
3 Hydrogen or oxygen supply
4 Nitrogen supply
5 Regulator valve and flow meter for hydrogen
6 Regulator valve and flow meter for oxygen
7 Regulator valve and flow meter for oxygen or hydrogen
8 Regulator valve and flow meter for nitrogen
9 Flashback arresters
10 Mixer valve
11 Vacuum valve
12 Vacuum bypass valve
13 Water ring pump
14 Vacuum gauge
15 Volumetric flask
16 Multiport valve
17 Absorption vessel with fritted-glass filters
18 Rinsing device
19 Flask containing absorption solution
20 Combustion space with condenser
21 Combustion tube
22 Combustion boat
23 Ground-glass joint
C Coolant

Figure A.1 – Diagram of Wickbold combustion apparatus

A.3.2 Weigh the sample into a combustion boat. The quantity must be such that at least 1,0 ml of silver nitrate solution is used during the subsequent argentometric titration. Heat the boat containing the sample strongly in the combustion tube with a flame or electric heat source. This is carried out first of all in the presence of nitrogen, until all the volatile organic components have been evaporated (otherwise there is the danger of an explosion). Afterwards turn on the oxygen supply. The decomposition and combustion gases are drawn through the oxyhydrogen flame, burned and, after cooling, led into the absorption solution. Refer to the manufacturer's operating instructions for details of the Wickbold combustion apparatus.

Place quartz or platinum wool before the burner head to prevent solid particles from passing through the oxyhydrogen flame. Ignite the sample by heating the left side of the combustion tube with a gas burner and slowly move the flame from left to right, i.e., against the flow of oxygen. Combustion is only complete when the combustion tube is free from soot. Afterwards, those parts of the apparatus that have been in contact with the combustion gases are rinsed out with water, collecting the washings.

If account has to be taken of possible chloride in the combustion residue, the combustion boat must be washed out with 1 + 1 nitric acid and rinsed with water (use a volume such that the solution has the correct pH-value for the titration). Collect the washings.

Place the washings and the absorption solution in a volumetric flask and stabilize the temperature of the flask. Fill the flask to the mark with water. Then titrate an aliquot portion of this solution against silver nitrate solution to determine the chloride content. If the chloride content is very low, an accurate determination is often not possible from an aliquot portion. In such a case, concentrate the absorption solution by evaporation and determine the chloride content from the remaining liquid. Evaporation may also prove necessary if, on combustion of a large test sample, large amounts of water accumulate in the absorption solution.

ICS 87.060.20

Descriptors: paints, varnishes, binders (materials), vinyl resins, rubber, tests, determination, colour, viscosity, density (mass/volume), determination of content, chlorine.

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