TC35

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Paints and varnishes — Preparation of acid extracts from dried paint films

Peintures et vernis — Préparation des extraits acides à partir de films secs

To be withdrawn

Reference number
ISO 6714:1990(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 6714 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*.

Annex A forms an integral part of this International Standard.

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Paints and varnishes — Preparation of acid extracts from dried paint films

1 Scope

This International Standard specifies methods for the preparation of acid extracts required as the test solutions for the determination of the "soluble" metal contents of dried paint films. The acid extracts are prepared in 0,07 mol/l hydrochloric acid solution, which is chosen as an approximation of the strength of the acids in the stomach.

The methods allow test solutions to be prepared from test portions weighing between 25 mg and 250 mg. In certain situations, either it may not be possible to obtain a 25 mg test portion, or larger test portions may be needed and, in both circumstances, appropriate modification of the method will be necessary.

This International Standard is for use in conjunction with the appropriate parts of ISO 3856. The acid extract prepared in accordance with ISO 6714 may be used for the determination of the individual metal contents.

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 565:1990, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings.*

ISO 835-1:1981, *Laboratory glassware — Graduated pipettes — Part 1: General requirements.*

ISO 835-2:1981, *Laboratory glassware — Graduated pipettes — Part 2: Pipettes for which no waiting time is specified.*

ISO 835-3:1981, *Laboratory glassware — Graduated pipettes — Part 3: Pipettes for which a waiting time of 15 s is specified.*

ISO 835-4:1981, *Laboratory glassware — Graduated pipettes — Part 4: Blow-out pipettes.*

ISO 1512:1974, *Paints and varnishes — Sampling.*

ISO 1513:1980, *Paints and varnishes — Examination and preparation of samples for testing.*

ISO 1514:1984, *Paints and varnishes — Standard panels for testing.*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

ISO 3856-1:1984, *Paints and varnishes — Determination of "soluble" metal content — Part 1: Determination of lead content — Flame atomic absorption spectrometric method and dithizone spectrophotometric method.*

ISO 3856-2:1984, *Paints and varnishes — Determination of "soluble" metal content — Part 2: Determination of antimony content — Flame atomic absorption spectrometric method and Rhodamine B spectrophotometric method.*

ISO 3856-3:1984, *Paints and varnishes — Determination of "soluble" metal content — Part 3: Determination of barium content — Flame atomic emission spectrometric method.*

ISO 3856-4:1984, *Paints and varnishes — Determination of "soluble" metal content — Part 4: Determination of cadmium content — Flame atomic absorption spectrometric method and polarographic method.*

ISO 3856-5:1984, *Paints and varnishes — Determination of "soluble" metal content — Part 5: Determination of hexavalent chromium content of the pigment portion of the liquid paint or the paint in powder form — Diphenylcarbazide spectrophotometric method.*

ISO 3856-6:1984, *Paints and varnishes — Determination of "soluble" metal content — Part 6: Determination of total chromium content of the liquid portion of the paint — Flame atomic absorption spectrometric method.*

ISO 3856-7:1984, *Paints and varnishes — Determination of "soluble" metal content — Part 7: Determination of mercury content of the pigment portion of the paint and of the liquid portion of water-dilutable paints — Flameless atomic absorption spectrometric method.*

ISO 4793:1980, *Laboratory sintered (fritted) filters — Porosity grading, classification and designation.*

3 Required supplementary information

For any particular application, the test method specified in this International Standard needs to be completed by the following supplementary information. The items of supplementary information are given in annex A.

4 Principle

A sample of dried paint film is removed from the substrate by means of a sharp scalpel. It is comminuted to pass completely through a 500 µm sieve. The comminuted material or a portion of it is then extracted with 0,07 mol/l hydrochloric acid under specified conditions.

5 Reagents

During the analysis, use only reagents of recognized analytical grade and only water of at least grade 3 purity, complying with ISO 3696.

5.1 Hydrochloric acid, approximately 18 % (m/m), $c(\text{HCl}) \approx 5$ mol/l.

5.2 Hydrochloric acid, dilute, $c(\text{HCl}) = 0{,}07$ mol/l.

5.3 Ethanol, minimum 95 % (V/V).

6 Apparatus

Ordinary laboratory apparatus and glassware together with the following.

6.1 Scalpel.

6.2 Sieves, of nominal aperture 150 µm and 500 µm, complying with ISO 565, made of brass or stainless steel.

6.3 Combined hotplate-stirrer, with appropriate controls (see figure 1).

Dimensions in millimetres

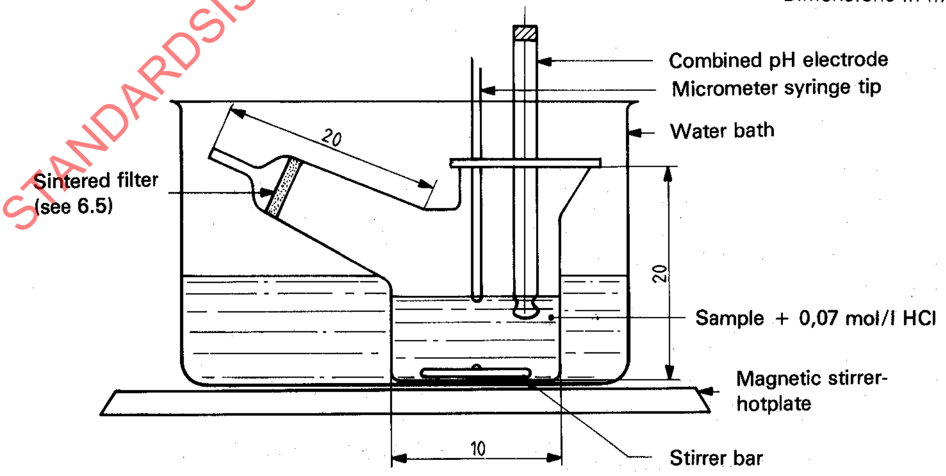

Figure 1 — Extraction apparatus with filter beaker

6.4 Balance, capable of weighing to 0,1 mg.

6.5 Filter beaker, of nominal capacity 7 ml, fitted with a P 4 sintered-glass filter (pore size index 1,6 μm to 4 μm, complying with ISO 4793) and modified as shown in figure 1 by enlargement of the top aperture to accommodate the stem of the pH-meter electrode assembly (6.8) and the tip of the micrometer syringe (6.9).

When test portions of 100 mg or more are available (see 7.1.1), a normal 25 ml beaker may be used, but the extract shall be passed through a P 4 sintered glass filter or a membrane filter.

6.6 Miniature magnetic stirrer bars (10 mm × 5 mm and 5 mm × 3 mm).

6.7 Graduated pipettes, complying with ISO 835,

a) capable of delivering at least 0,5 ml in 0,05 ml steps;

b) capable of delivering at least 10 ml in 2,5 ml steps.

6.8 pH-meter, with combined electrodes having 5 mm outside diameter.

6.9 Micrometer syringe.

6.10 Filter tube, with ground-glass-joint connector and fitted with a side arm to take a vacuum suction line.

6.11 Flexible acid-resistant tubing (for example silicone or butyl rubber tubing).

7 Preparation of the test sample

7.1 From a dry paint film on a substrate

Condition the paint film and substrate at 23 °C ± 2 °C and (50 ± 5) % relative humidity for 1 h.

7.1.1 When the paint film is available in sufficient quantity

When conditioned, remove the paint film from its substrate by means of the scalpel (6.1). Take care not to remove any substrate with the paint. Grind the removed film in a mortar with a pestle and rub it through a 500 μm sieve (6.2) using the pestle and the minimum of force to avoid distorting the mesh apertures of the sieve. Ensure that the whole sample passes through the sieve.

If difficulty is experienced with grinding due to residual plasticity, an alternative method of comminution shall be devised (for example cutting) whereby the particle size is reduced to pass a 1 mm sieve. This shall be recorded in the test report.

On no account shall the paint film be comminuted by a method in which there is a possibility of overheating or excessive cooling as this could adversely affect the material and the extraction.

7.1.2 When the paint film is restricted in quantity

When conditioned, remove the paint film as described in 7.1.1. Comminute the film by cutting or breaking using a pestle and mortar to avoid losses, and then rub it through the mesh of a 500 μm sieve (6.2) using the pestle and the minimum of force to avoid distorting the mesh apertures of the sieve. Ensure that the whole sample passes through the sieve (see 7.1.1).

7.2 From the liquid paint

Take a representative sample of the product to be tested, as described in ISO 1512.

Examine and prepare the sample for testing, as described in ISO 1513. If any skin is present, remove it as far as possible. Thoroughly stir the sample and, if necessary, pass it through a sieve of nominal aperture 150 μm to remove any remaining skin and other extraneous matter.

Unless otherwise specified, prepare smooth glass test panels as described in ISO 1514. Coat each panel by the specified method with the product or system under test. Dry (or, in the case of stoving paints, stove) and age each coated test panel for the specified time and under the specified conditions. Prepare the test sample from the dried film as described in 7.1.

If the method of applying and drying the product under test is not specified, the paint should be applied by whatever method is most suitable and dried at 23 °C ± 2 °C and (50 ± 5) % relative humidity for 28 days.

8 Procedure

8.1 Carry out the extraction in duplicate (if a sufficient quantity of the test sample is not available, carry out a single extraction and report accordingly). During the extraction and the ensuing standing period do not subject the test solution to direct sunlight and, if "soluble" cadmium is to be determined (see ISO 3856-4), protect it as fully as possible from light.

Maintain the extraction temperature at 23 °C ± 2 °C.

8.2 Weigh, to the nearest 0,1 mg, from the comminuted material (see clause 7) a portion that will depend on the quantity of material available. Carry out procedure a) if the quantity of sample is not unduly restricted and b) if the quantity of sample is limited.

a) Quantitatively transfer the weighed portion, preferably 200 mg but not less than 100 mg, to a 25 ml beaker.

b) Quantitatively transfer the weighed portion, which shall be not less than 25 mg, to the filter beaker (6.5).

In either case, add the appropriate miniature magnetic stirrer bar (6.6) and wet the test portion with the ethanol (5.3) using 0,1 ml for every 50 mg of the test portion.

If it is not possible to obtain a test portion of at least 25 mg, use a smaller filter beaker and add not less than 0,05 ml of ethanol to wet the test portion.

8.3 Stand the beaker or filter beaker and contents in the glass dish on the hotplate-stirrer (6.3). Add by means of a pipette (6.7) approximately 50 times the mass of the test sample (i.e. 5 ml for each 100 mg) of the dilute hydrochloric acid (5.2) previously adjusted to a temperature of 23 °C ± 0,1 °C.

Immediately insert the electrodes of the pH-meter (6.8) and commence stirring the liquid at a rate sufficient to maintain the test portion in continuous suspension, taking care not to damage the glass electrode. Continue the stirring for 60 min ± 1 min, adjusting the speed of stirring if necessary. Maintain the pH of the suspension at the value of that of the dilute hydrochloric acid (5.2) by adding small aliquots of hydrochloric acid (5.1) from the micrometer syringe (6.9), as necessary, during the period of stirring.

8.4 At the end of the period of stirring allow the mixture to stand for 60 min ± 1 min.

If a 25 ml beaker has been used, decant the contents through the filter (6.5), connected via the connector to a filter tube (see figure 2), applying suction if necessary, collecting the filtrate in the filter tube (6.10). Immediately stopper the filter tube.

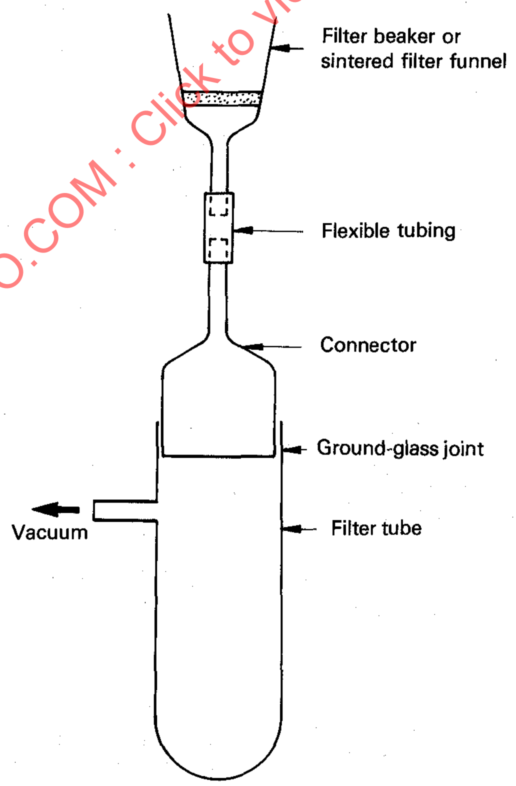

Figure 2 — Filtration of extract using filter tube