INTERNATIONAL STANDARD

ISO 6744-3

First edition
1999-12-01

Binders for paints and varnishes — Alkyd resins —

Part 3: Determination of unsaponifiable matter content

Liants pour peintures et vernis — Résines alkydes —

Partie 3: Détermination de la teneur en matière insaponifiable

Reference number
ISO 6744-3:1999(E)

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Printed in Switzerland

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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.

Attention is drawn to the possibility that some of the elements of this part of ISO 6744 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 6744-3 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 10, *Test methods for binders for paints and varnishes*.

Together with the other parts (see below), this part of ISO 6744 cancels and replaces ISO 6744:1984, which has been technically revised.

ISO 6744 consists of the following parts, under the general title *Binders for paints and varnishes — Alkyd resins*:

— *Part 1: General methods of test*

— *Part 2: Determination of phthalic anhydride content*

— *Part 3: Determination of unsaponifiable matter content*

— *Part 4: Determination of fatty acid content*

Binders for paints and varnishes — Alkyd resins —

Part 3: Determination of unsaponifiable matter content

1 Scope

This part of ISO 6744 specifies a method for determining the unsaponifiable matter content of alkyd resins. It is always used in conjunction with ISO 6744-2, which describes the first step of the analysis.

It is not applicable to modified alkyd resins (see 3.2 of ISO 6744-1:1999).

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 6744. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 6744 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

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

ISO 6744-1:1999, *Binders for paints and varnishes — Alkyd resins — Part 1: General methods of test.*

ISO 6744-2:1999, *Binders for paints and varnishes — Alkyd resins — Part 2: Determination of phthalic anhydride content.*

ISO 6744-4:1999, *Binders for paints and varnishes — Alkyd resins — Part 4: Determination of fatty acid content.*

ISO 15528:—[1)], *Paints, varnishes and raw materials for paints and varnishes — Sampling.*

3 Principle

The unsaponifiable matter content of a saponified alkyd resin sample is determined by drying the filtrate obtained in ISO 6744-2 and extraction with diethyl ether or light petroleum ether to remove the fatty acids. The residue is weighed and the percentage is calculated based on the initial sample mass obtained from ISO 6744-2.

4 Reagents

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

1) To be published. (Revision of ISO 842:1984 and ISO 1512:1991)

4.1 Diethyl ether, free from peroxides, to which a crystal of hydroquinone has been added (for the diethyl ether method specified in 7.2).

4.2 Light petroleum, boiling range 30 °C to 50 °C (for the light petroleum method specified in 7.3).

NOTE In some countries, this solvent is known as "petroleum ether 30/50".

4.3 Potassium hydroxide, 56 g/l aqueous solution.

4.4 Acetone.

4.5 Ethanol, 95 % (by volume).

5 Apparatus

Ordinary laboratory apparatus and glassware, together with the following:

5.1 Separating funnels, of capacity 250 ml.

5.2 Distillation apparatus, with a rotary evaporator or water bath.

5.3 Drying oven, capable of being maintained at approximately 105 °C.

6 Sampling

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

7 Procedure

7.1 Number of determinations

Carry out the determination in duplicate.

NOTE When using the diethyl ether method (7.2), emulsions may be formed which are difficult to separate. In such cases, ethanol, saturated sodium chloride solution or a few drops of dilute mineral acid may be added to break the emulsion. In certain cases, it may also be helpful to use the light petroleum method (7.3) instead of the diethyl ether method.

7.2 Diethyl ether method

Vacuum-distill or evaporate the solvent from the filtrate obtained as described in ISO 6744-2:1999, subclause 7.3. Dissolve the residue in 25 ml of ethanol (4.5) and 50 ml of water and transfer to a separating funnel (5.1). Rinse the container with 50 ml of diethyl ether (4.1) and collect all the washings in the separating funnel.

Stopper the separating funnel, shake and allow the layers to separate. Drain off the lower (aqueous) layer into a second separating funnel (5.1) and retain the other layer in the first separating funnel. Continue the extraction of the aqueous layer with two further 50 ml portions of diethyl ether. Combine all the ether extracts in the first separating funnel and reserve the aqueous layer for the determination of the fatty acid content in accordance with ISO 6744-4.

Wash the combined ether extracts with 25 ml portions of water until the pH of the washings is neutral. Add the washings to the aqueous layer obtained by the procedure described above. Evaporate the ether using a suitable apparatus (5.2), either under inert gas using a distillation apparatus with a rotary evaporator or, while observing all required safety precautions, over a water bath. If necessary, remove any water present by addition of acetone (4.4) and evaporating as above.

Dry the residue in the drying oven (5.3) at approximately 105 °C until the difference between the results of two consecutive weighings is not greater than 0,1 %, calculated on the basis of the lower value. Weigh the residue to the nearest 1 mg (m_3).

7.3 Light petroleum method

Vacuum distill or evaporate the solvent from the filtrate obtained as described in ISO 6744-2:1999, subclause 7.3. Transfer the residue with about 100 ml of water to a separating funnel (5.1) and add 50 ml of light petroleum (4.2). Rinse the container with 50 ml of light petroleum and collect all the washings in the separating funnel.

Stopper the separating funnel, shake and allow the layers to separate. Drain off the lower (aqueous) layer into a second separating funnel (5.1) and retain the other layer in the first separating funnel. Continue the extraction of the aqueous layer with two further 50 ml portions of light petroleum. Combine all the light petroleum extracts in the first separating funnel and reserve the aqueous layer for the determination of the fatty acid content in accordance with ISO 6744-4.

Shake the combined light petroleum extracts with a mixture consisting of 15 ml of ethanol (4.5), 15 ml of water and 0,5 ml of potassium hydroxide solution (4.3), allow the layers to separate and drain off the lower (aqueous) layer. Wash the light petroleum extracts with 25 ml portions of water until the pH of the washings is neutral. Add all the aqueous washings to the aqueous layer obtained by the procedure described above. Evaporate the light petroleum using any suitable apparatus (5.2), and remove the water from the residue by addition of acetone (4.4) and evaporate as described above.

Dry the residue in the drying oven (5.3) at approximately 105 °C until the difference between the results of two consecutive weighings is not greater than 0,1 %, calculated on the basis of the lower value. Weigh the residue to the nearest 1 mg (m_3).

8 Expression of results

Calculate the unsaponifiable matter content w_U, expressed as a percentage by mass, by the equation

$$w_U = \frac{m_3}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the test portion (see ISO 6744-2:1999, subclause 7.2);

m_3 is the mass, in grams, of the residue.

The unsaponifiable matter content shall be calculated on the basis of the resin or, for resin solutions, on the basis of the non-volatile matter content of the solution.

9 Test report

The test report shall contain at least the following information:

a) a reference to this part of ISO 6744 (ISO 6744-3);

b) all details necessary to identify the product tested;

c) the result of the test, as indicated in clause 8, and which test method was used;

d) any deviation from the test method specified;

e) the date of the test.

ICS 87.060.20

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