INTERNATIONAL STANDARD

ISO 6744-2

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Binders for paints and varnishes — Alkyd resins —

Part 2: Determination of phthalic anhydride content

Liants pour peintures et vernis — Résines alkydes —

Partie 2: Détermination de la teneur en anhydride phtalique

Reference number
ISO 6744-2:1999(E)

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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. 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 6744-2 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 2:
Determination of phthalic anhydride content

1 Scope

This part of ISO 6744 specifies a method for determining the phthalic anhydride content of alkyd resins.

It is only applicable to those alkyd resins which contain only orthophthalic acid as the polybasic acid. It is not applicable to modified alkyd resins (see 3.2 of ISO 6744-1:1999) or to resins containing benzoic acid.

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 3681:1996, *Binders for paints and varnishes — Determination of saponification value — Titrimetric method.*

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 6744-3:1999, *Binders for paints and varnishes — Alkyd resins — Part 3: Determination of unsaponifiable matter 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

A test portion is saponified with potassium hydroxide solution. The potassium phthalate precipitate which forms is filtered, dried and weighed. The filtrate is reserved for the subsequent determinations of unsaponifiable matter and fatty acid contents.

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

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.

4.1 Toluene.

4.2 Solvent mixture, consisting of 1 part by volume of toluene and 1 part by volume of absolute ethanol. Alternatively, a mixture of 1 part by volume of diethyl ether and 1 part by volume of absolute ethanol may be used.

4.3 Potassium hydroxide, approximately 28 g/l solution in absolute ethanol, clear and free from sediment.

5 Apparatus

Ordinary laboratory apparatus and glassware, together with the following:

5.1 Conical flask, of capacity about 250 ml, with ground-glass joint.

5.2 Reflux condenser, with ground-glass joint, fitting on the conical flask (5.1) and the drying tube (5.3).

5.3 Drying tube, filled with anhydrous calcium chloride, fitting on the reflux condenser (5.2).

5.4 Hotplate, fitted with a magnetic stirrer.

5.5 Sintered-glass filter crucible, diameter of the plate 30 mm, pore diameter 15 µm to 40 µm.

5.6 Desiccator, containing silica gel as the desiccant.

5.7 Weighing bottle.

5.8 Drying oven, capable of being maintained at approximately 140 °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.

7.2 Test portion

Weigh, to the nearest 1 mg, a mass (m_0) of the resin or the resin solution containing not more than 2 g of fatty acid (see ISO 6744-4) into the conical flask (5.1).

NOTE The mass of resin to be taken will normally be 3 g to 5 g.

7.3 Determination

Dissolve the test portion (see 7.2) in 10 ml of toluene (4.1) in the conical flask. Add 100 ml of potassium hydroxide solution (4.3), fit the reflux condenser (5.2) on to the conical flask and fit the drying tube (5.3) on to the condenser.

Heat the contents of the flask to boiling and maintain at the boiling point under reflux for 2 h, while stirring (see note). Allow the flask to cool to ambient temperature, rinse the condenser with some solvent and filter the solution through the weighed sintered-glass filter crucible (5.5). Reserve the filtrate for the determination of the unsaponifiable matter in accordance with ISO 6744-3. Wash the potassium phthalate precipitate as rapidly as possible several times with solvent mixture (4.2). Make sure that the precipitate is continuously covered with the solvent mixture and only becomes exposed after the final washing. Afterwards, the precipitate may be placed in an oven maintained at 100 °C for 1 min to 2 min to facilitate evaporation of the solvents. Dry the precipitate in the desiccator (5.6) under vacuum 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 precipitate to the nearest 1 mg (m_1).

As the precipitate is hygroscopic, transfer the crucible and the precipitate immediately after drying to the weighing bottle (5.7). Close the weighing bottle and weigh the precipitate to the nearest 1 mg. Dry the precipitate in the drying oven (5.8) at approximately 140 °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_2).

NOTE Normally, saponification is complete after 2 h. The completeness of saponification can be checked by determining the saponification value under more severe conditions which can be achieved by the use of a longer saponification time, a more concentrated potassium hydroxide solution or a higher-boiling alcohol as solvent (see ISO 3681).

8 Expression of results

Calculate the phthalic anhydride content w_p, expressed as a percentage by mass, by the equations

$$w_p = \frac{m_1 \times 51{,}4}{m_0} \quad (1)$$

and

$$w_p = \frac{m_2 \times 61{,}1}{m_0} \quad (2)$$

where

m_0 is the mass, in grams, of the test portion (see note to 7.2);

m_1 is the mass, in grams, of the precipitate after drying in vacuum at ambient temperature (potassium phthalate monoethanolate);

m_2 is the mass, in grams, of the residue after drying at 140 °C (ethanol-free potassium phthalate);

51,4 is a factor to convert the mass of potassium phthalate monoethanolate to that of phthalic anhydride;

61,1 is a factor to convert the mass of anhydrous potassium phthalate to that of phthalic anhydride.

Calculate the average of the two values and report this average value.

NOTE If the difference between the results calculated by the two equations is greater than 2 % absolute, the method specified in this part of ISO 6744 is not suitable for the alkyd resin under test (see footnote c to Table 1 of ISO 6744-1:1999).

The phthalic anhydride 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 6744-2);

b) all details necessary to identify the product tested;

c) the result of the test, as indicated in clause 8;

d) any deviation from the test method specified;

e) the date of the test.