# International Standard 6744

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# Binders for paints and varnishes — Alkyd resins — General methods of test

*Liants pour peintures et vernis — Résines alkydes — Méthodes générales d'essai*

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# Foreword

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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.

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# Binders for paints and varnishes — Alkyd resins — General methods of test

## 1 Scope and field of application

This International Standard describes general methods of test for alkyd resins for use in paints, varnishes and similar products. It is also applicable to those solutions and similar preparations made from alkyd resins that are intended for use as binders for paints and varnishes.

The test methods to be applied to an individual alkyd resin shall be the subject of agreement between the interested parties.

The method given in annex A is only applicable to those alkyd resins which contain orthophthalic acid as the polybasic acid. The methods described in annexes A, B and C are not applicable to modified alkyd resins (3.2) and those resins containing benzoic acid.

## 2 References

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

ISO 1523, *Paints, 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 3104, *Petroleum products — Transparent and opaque liquids — Determination of kinematic viscosity and calculation of dynamic viscosity.*

ISO 3219, *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 non-volatile matter.*

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

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

ISO 3682, *Binders for paints and varnishes — Determination of acid value — Titrimetric method.*

ISO 3696, *Water for laboratory use — Specifications.*[1]

ISO 4629, *Paint media — Determination of hydroxyl value — Titrimetric method.*

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

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

## 3 Definitions

**3.1 alkyd resin**: Synthetic resin resulting from the polycondensation of polyacids and fatty acids (or oils) with polyhydric alcohols.

**3.2 modified alkyd resin**: Alkyd resin, modified in order to obtain specific properties, for example with rosin acids, phenolic resins, vinyl toluene, styrene, isocyanates, polyamides, acrylic, epoxy or silicone compounds.

NOTE — An alkyd resin, based on tall oil fatty acids containing not more than 4 % of rosin acids, is regarded as an "alkyd resin", not as a "modified alkyd resin".

## 4 Classification

Alkyd resins are classified into the following groups:

a) according to the content of fatty acid or oil:

| | |
|---|---|
| short oil alkyd resins: | less than about 40 %* |
| medium oil alkyd resins: | about 40 % to 60 %* |
| long oil alkyd resins: | above about 60 %* |

b) according to the content of dibasic acid (see annex A);

c) according to the modifying component (see 3.2).

## 5 Sampling

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

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[1] At present at the stage of draft.

* Content of oil calculated as the triglyceride portion of the non-volatile matter.

## 6 Methods of test

Table — Properties and methods of test

| Property | Method of test |
|---|---|
| Colour | ISO 6271 (Platinum-cobalt scale)<br>ISO 4630 (Gardner colour scale) |
| Flow time[1] | ISO 2431 |
| Viscosity | ISO 3219 or ISO 3104 or other agreed methods |
| Non-volatile matter[1] | ISO 3251 |
| Flashpoint[1] | ISO 1523<br>ISO 3679 |
| Acid value | ISO 3682[2] |
| Hydroxyl value | ISO 4629[2] |
| Phthalic anhydride content | Annex A[3] |
| Unsaponifiable matter | Annex B[3] |
| Fatty acid content | Annex C[3][4] |

1) For alkyd resin solutions only.

2) The acid and hydroxyl values shall be calculated on the basis of the non-volatile matter of the resin solution.

3) These methods are not applicable to alkyd resins that contain benzoic acid and polybasic acids other than *o*-phthalic and/or are modified by compounds such as rosin acids, vinyl toluene, styrene, isocyanates, acrylic, epoxy, or silicone compounds. The general test methods to be specified for these excluded modified alkyd resins will be the subject of a future International Standard.

4) Rosin acids as part of tall oil fatty acids will be found together with the fatty acids. The calculation of these rosin acids as a part of fatty acids is to be agreed between the interested parties.

## 7 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 6744);

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.

# Annex A

# Determination of phthalic anhydride content

(This annex forms an integral part of the Standard.)

## A.1 Principle

Saponification of the test portion with potassium hydroxide solution. Filtration of the remaining potassium phthalate precipitate, followed by drying and weighing. Reservation of the filtrate for the subsequent determinations of unsaponifiable matter and fatty acid contents.

## A.2 Reagents

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

**A.2.1 Toluene.**

**A.2.2 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.

**A.2.3 Potassium hydroxide**, Approximately 28 g/l solution in absolute ethanol, clear and free from sediment.

## A.3 Apparatus

Ordinary laboratory apparatus, and

**A.3.1 Conical flask**, of capacity about 250 ml, with ground glass joint.

**A.3.2 Reflux condenser**, with ground glass joint, fitting on the conical flask (A.3.1) and the drying tube (A.3.3).

**A.3.3 Drying tube**, filled with anhydrous calcium chloride, fitting on the reflux condenser (A.3.2).

**A.3.4 Hot plate**, fitted with a magnetic stirrer.

**A.3.5 Sintered glass filter crucible**, diameter of the plate 30 mm, pore diameter 15 to 40 µm.

**A.3.6 Desiccator**, containing concentrated sulfuric acid as the desiccant.

**A.3.7 Weighing bottle.**

**A.3.8 Drying oven**, capable of being maintained at approximately 140 °C.

## A.4 Procedure

Carry out the determination in duplicate.

### A.4.1 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 annex C), into the conical flask (A.3.1).

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

### A.4.2 Determination

Dissolve the test portion (A.4.1) in 10 ml of the toluene (A.2.1). Add 100 ml of the potassium hydroxide solution (A.2.3), fit the reflux condenser (A.3.2) on to the conical flask (A.3.1) and fit the drying tube (A.3.3) 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 (A.3.5). Reserve the filtrate for the determination of the unsaponifiable matter according to annex B. Wash the potassium phthalate precipitate as rapidly as possible several times with the solvent mixture (A.2.2). Make sure that the precipitate is continuously covered with the solvent mixture and only becomes exposed after the final washing. Afterwards, it is advantageous to place the precipitate in an oven maintained at 100 °C for 1 to 2 min to facilitate evaporation of the solvents. Dry the precipitate in the desiccator (A.3.6) 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 (A.3.7). Close the weighing bottle and weigh the precipitate to the nearest 1 mg. Dry the precipitate in the drying oven (A.3.8) 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, the saponification is completed 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.)

## A.5 Expression of results

Calculate the phthalic anhydride content by the equations

$$c_p = \frac{m_1 \times 51,4}{m_0} \qquad \ldots \ (1)$$

and

$$c_p = \frac{m_2 \times 61,1}{m_0} \qquad \ldots \ (2)$$

where

$c_p$ is the phthalic anhydride content, expressed as a percentage by mass;

$m_0$ is the mass, in grams, of the test portion (see note 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.

NOTES

1 If the difference between the results calculated by both equations is greater than 2 % absolute, the method specified in this annex is not suitable for the alkyd resin under test (see footnote 3 to the table).

2 The phthalic anhydride content should be calculated on the basis of the resin or, for resin solutions, on the basis of the non-volatile matter content of the solution.

# Annex B

## Determination of unsaponifiable matter content

(This annex forms an integral part of the Standard.)

### B.1 Reagents

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

**B.1.1 Diethyl ether**, free from peroxides, to which a crystal of hydroquinone has been added (for the diethyl ether method specified in B.3.1).

**B.1.2 Light petroleum**, boiling range 30 to 50 ° (for the light petroleum method specified in B.3.2).

NOTE — In some countries this reagent is known as "Petroleum ether 30/50 °C".

**B.1.3 Potassium hydroxide**, 56 g/l aqueous solution.

**B.1.4 Acetone.**

**B.1.5 Ethanol**, 95 % (*V*/*V*).

### B.2 Apparatus

Ordinary laboratory apparatus, and

**B.2.1 Separating funnels**, of capacity 250 ml.

**B.2.2 Distillation apparatus, rotary evaporator** or **water bath.**

**B.2.3 Drying oven**, capable of being maintained at approximately 105 °C.

### B.3 Procedure

Carry out the determination in duplicate.

NOTE — When using the diethyl ether method (B.3.1) emulsions may be formed which are difficult to separate. In this case ethanol, saturated sodium chloride solution or some drops of a diluted mineral acid may be added to break the emulsion. In certain cases it may also be helpful to use the light petroleum method (B.3.2) instead of the diethyl ether method.

#### B.3.1 Diethyl ether method

Vacuum distil or evaporate the solvent from the filtrate obtained as described in clause A.4. Dissolve the residue in 25 ml of the ethanol (B.1.5) and 50 ml of water and transfer to a separating funnel (B.2.1). Rinse the container with 50 ml of the diethyl ether (B.1.1) 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 (B.2.1) and retain the other layer in the first separating funnel. Continue the extraction of the aqueous layer with two successive 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 according to annex C.

Wash the final 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 any suitable apparatus (B.2.2), either under inert gas using the distillation apparatus or rotary evaporator or, while observing the required safety precautions, the water bath. If necessary, remove any water present by addition of the acetone (B.1.4) and evaporating as above.

Dry the residue in the drying oven (B.2.3) 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 ($m_3$).

#### B.3.2 Light petroleum method

Vacuum distil or evaporate the solvent from the filtrate obtained as described in clause A.4. Transfer the residue with about 100 ml of water to a separating funnel (B.2.1) and add 50 ml of the light petroleum (B.1.2). Rinse the container with 50 ml of the 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 (B.2.1) and retain the other layer in the first separating funnel. Continue the extraction of the aqueous layer with two successive 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 according to annex C.

Shake the final combined light petroleum extracts with a mixture consisting of 15 ml of ethanol (B.1.5), 15 ml of water and 0,5 ml of the potassium hydroxide solution (B.1.3), 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 (B.2.2), and remove the water from the residue by addition of acetone (B.1.4) and evaporating as described above.

Dry the residue in the drying oven (B.2.3) 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 to the nearest 1 mg ($m_3$).

## B.4 Expression of results

Calculate the unsaponifiable matter content by the equation

$$U = \frac{m_3}{m_0} \times 100 \qquad \ldots (3)$$

where

$m_0$ is the mass, in grams, of the test portion (A.4.1);

$m_3$ is the mass, in grams, of the residue;

$U$ is the unsaponifiable matter content, expressed as a percentage by mass.

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

# Annex C

# Determination of fatty acid content

(This annex forms an integral part of the Standard.)

## C.1 Reagents

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

**C.1.1 Diethyl ether**, free from peroxides, to which a crystal of hydroquinone has been added.

**C.1.2 Acetone.**

**C.1.3 Hydrochloric acid**, approximately 73 g of HCl per litre.

**C.1.4 Silver nitrate**, approximately 10 g/l solution.

## C.2 Apparatus

Ordinary laboratory apparatus and

**C.2.1 Separating funnel**, of capacity 250 ml.

**C.2.2 Distillation apparatus, rotary evaporator** or **water bath.**

**C.2.3 Vacuum drying oven**, capable of being maintained at approximately 60 °C.

## C.3 Procedure

Carry out the determination in duplicate.

If the diethyl ether method for the determination of the unsaponifiable matter content was used, transfer the aqueous solution obtained as described in B.3.1 quantitatively into the separating funnel (C.2.1).

If the light petroleum method for the determination of the unsaponifiable matter content was used, evaporate the aqueous ethanolic solution, obtained after separation of the unsaponifiable matter together with the alkaline washing water, until complete removal of the alcohol. Then transfer with water into the separating funnel (C.2.1).

Acidify the solution with the hydrochloric acid (C.1.3) and extract three times, each time with 50 ml of the diethyl ether (C.1.1).

Wash the combined ether extracts with water until no chloride ions are detectable with the silver nitrate solution (C.1.4). Then evaporate the ether using any suitable apparatus (C.2.2), either under inert gas using the distillation apparatus or rotary evaporator or, while observing the required safety precautions, the water bath. If necessary, remove any water present by addition of acetone (C.1.2) and evaporating as described above.

Dry the residue in the vacuum drying oven (C.2.3) at approximately 60 °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 ($m_4$).

## C.4 Expression of results

Calculate the fatty acid content by the equation

$$F = \frac{m_4}{m_0} \times 100 \qquad \ldots (4)$$

where

$F$ is the fatty acid content, expressed as a percentage by mass;

$m_0$ is the mass, in grams, of the test portion (A.4.1);

$m_4$ is the mass, in grams, of the residue.

NOTE — The fatty acid content should be calculated on the basis of the resin or, for resin solutions, on the basis of the non-volatile matter content of the solution.