INTERNATIONAL STANDARD

ISO 3682

Third edition
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Binders for paints and varnishes — Determination of acid value — Titrimetric method

Liants pour peintures et vernis — Détermination de l'indice d'acide — Méthode titrimétrique

Reference number
ISO 3682:1996(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 3682 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC10, *Test methods for binders for paints and varnishes*.

This third edition cancels and replaces the second edition (ISO 3682:1983), which has been technically and editorially revised. The main change is that the acid value is no longer related to 1 g of non-volatile matter of the product but to 1 g of the product itself.

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Binders for paints and varnishes — Determination of acid value — Titrimetric method

1 Scope

This International Standard specifies a titrimetric method for determining the acid value of binders for paints and varnishes.

It is not applicable to phenolic resins.

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 385-1:1984, *Laboratory glassware — Burettes — Part 1: General requirements.*

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

3 Definition

For the purposes of this International Standard, the following definition applies:

3.1 acid value: The number of milligrams of potassium hydroxide (KOH) required to neutralize the free acids in 1 g of the product tested.

4 Principle

The free acids contained in a test portion are titrated with potassium hydroxide solution, either in the presence of a colour indicator or potentiometrically.

5 Reagents

During the analysis, use only reagents of recognized analytical grade.

5.1 Solvent mixture, consisting of 2 parts by volume of toluene and 1 part by volume of at least 95 % (*V/V*) ethanol (see note 4 to 8.2), unless otherwise agreed or specified. If denaturated alcohol, or alcohol of another quality, is used, its suitability for the test shall be checked. Neutralize, using phenolphthalein as indicator, the solvent mixture with potassium hydroxide solution (5.2) prior to use.

5.2 Potassium hydroxide, standard volumetric solution, c(KOH) = 0,1 mol/l, in 95 % (*V/V*) ethanol (see 5.1) or in methanol, free from carbonates, standardized against potassium hydrogen phthalate.

NOTE 1 A 0,5 mol/l standard volumetric solution of potassium hydroxide may also be used in cases when more than 50 ml of titrant would be required with a 0,1 mol/l solution, thus avoiding the additional errors involved in refilling the 50 ml burette.

Check the concentration of this solution on the day of use.

5.3 Phenolphthalein, 10 g/l solution in 95 % (*V/V*) ethanol, in methanol or in isopropanol.

NOTE 2 Other suitable indicators may be used, for example a 10 g/l solution of bromothymol blue in 95 % (*V/V*) ethanol, in methanol or in isopropanol.

6 Apparatus

Ordinary laboratory apparatus and glassware, together with the following:

6.1 Conical flask, capacity 250 ml.

6.2 Burette, capacity 50 ml, complying with the requirements of ISO 385-1.

6.3 Potentiometric titration apparatus, fitted with a glass electrode and a reference electrode. The use of this apparatus is an optional alternative (see note 3 to 8.2).

6.4 Magnetic stirrer.

7 Sampling

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

8 Procedure

Carry out the determination in duplicate.

8.1 Test portion

By reference to table 1, select the appropriate mass of test portion to be taken. This mass shall be chosen so that the volume of potassium hydroxide solution (5.2) used is in the range 10 ml to 30 ml.

Table 1 — Mass of test portion

Expected acid value mg KOH/g	Approximate mass of test portion g
up to 10	10
above 10 to 25	5
above 25 to 50	2,5
above 50 to 150	1
above 150	0,5

Weigh, to the nearest 1 mg, the test portion into the conical flask (6.1).

8.2 Determination

Dissolve the test portion (8.1), with stirring, in 50 ml of the solvent mixture (5.1).

If free acid anhydrides are present in the binder, as in the case of certain polyester resins, the alcoholic potassium hydroxide solution reacts only partially with the anhydrides. This, however, is generally of little importance with most binders because of the small amount of free anhydrides present. If it is suspected that significant amounts of free anhydrides are present, an aqueous potassium hydroxide solution shall be used.

Warm, if necessary, but cool the solution to room temperature before carrying out the titration.

CAUTION — If the solution is warmed, this shall be carried out in a fume cupboard or well ventilated atmosphere. Avoid overheating.

Add 2 or 3 drops of phenolphthalein solution (5.3) and titrate rapidly with potassium hydroxide solution (5.2) until a red coloration just appears and is stable for at least 10 s while the solution is being stirred.

NOTE 3 With some substances, for example certain polyester resins, no very definite colour change will be obtained with phenolphthalein. In such cases, another indicator, for example bromothymol blue (see note 2 to 5.3) may be used. In all cases of doubt, and especially when the solutions are coloured, a potentiometric titration to pH 7 is to be preferred, using glass electrodes with a suitable response time.

In the case of polybasic acids, there may also be points of inflection above pH 7. In such cases, the point of inflection in the most basic range shall be taken as the end point.

If a precipitate is formed during the titration which interferes with the determination of the end point, add additional solvent as indicated in note 4. When a suitable solvent mixture has been found, repeat the titration using the same solvent mixture.

NOTE 4 The type and volume of solvent mixture to be used depend on whether precipitation occurs during the titration. The volume of solvent mixture may be increased up to 150 ml, or 25 ml of acetone may be added. The purpose of the solvent is to prevent precipitation during titration and not to dissolve the resin initially.

Use the same solvent mixture for the blank test (8.3) and record the type and volume of the solvent mixture in the test report (clause 10).

8.3 Blank test

Carry out a blank test following the same procedure, but omitting the test portion.

NOTE 5 Theoretically, if the neutralization of the solvent mixture (5.1) has been carried out correctly, the result of the blank test will be zero.

9 Expression of results

9.1 Calculation

Calculate the acid value AV, in milligrams of KOH per gram of product, using the equation

$$AV = \frac{(V_1 - V_0) \times c \times 56{,}1}{m}$$