International Standard ISO 3682

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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

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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.

International Standard ISO 3682 was developed by Technical Committee ISO/TC 35, *Paints and varnishes.*

The first edition (ISO 3682-1976) had been approved by the member bodies of the following countries:

Austria	Iran	Romania
Brazil	Ireland	South Africa, Rep. of
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Canada	Mexico	Sweden
Chile	Netherlands	Switzerland
France	New Zealand	Turkey
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The member body of the following country had expressed disapproval of the document on technical grounds:

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This second edition, which cancels and replaces ISO 3682-1976, incorporates draft Amendment 1, which was circulated to the member bodies in December 1981 and has been approved by the member bodies of the following countries:

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The member body of the following country expressed disapproval of the document on technical grounds:

Germany, F.R.

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

1 Scope and field of application

This International Standard specifies a titrimetric method for the determination of the acid value of binders for paints and varnishes. It is not applicable to phenolic resins.

2 References

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

ISO 3251, *Paint media — Determination of volatile and non-volatile matter.*

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3 Definition

For the purpose of this International Standard, the following definition applies.

acid value: The number of milligrams of potassium hydroxide (KOH) required to neutralize the free acids in 1 g of the non-volatile matter of the product.

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NOTE — 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.

4 Sampling

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

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 ethanol, at least 95 % (*V*/*V*),[1] if not otherwise agreed or specified. Neutralize the solvent mixture with the potassium hydroxide solution (5.2) prior to use.

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

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

5.3 Phenolphthalein, indicator solution, 10 g/l in ethanol 95 % (*V*/*V*), methanol or 2-propanol.

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

6 Apparatus

Ordinary laboratory apparatus and

6.1 Conical flask, capacity 250 ml.

6.2 Burette, capacity 50 ml.

If required:

6.3 Device for potentiometric titration, with glass electrode and reference electrode.

6.4 Magnetic stirrer.

7 Procedure

7.1 Test portion

The mass of the test portion depends on the acid value to be expected (see the table for guidance in selecting the appropriate mass). This mass shall be chosen so that the volume of the potassium hydroxide solution (5.2) is in the range 10 to 30 ml.

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

Table

Expected acid value	Mass of test portion
mg KOH/g	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

1) If denatured alcohol, or alcohol of other quality, is used its suitability for the test should be checked.

7.2 Determination

Carry out the determination in duplicate.

Dissolve the test portion (7.1) in 50 ml of the solvent mixture (5.1). Warm, if necessary, but cool the solution to room temperature before carrying out the titration.

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

Add 2 or 3 drops of the phenolphthalein indicator solution (5.3) and titrate rapidly with the potassium hydroxide solution (5.2) until a red coloration that is stable for at least 10 s just appears (see note 1).

If a precipitate is formed during the titration which interferes with the determination of the end-point, add additional solvent as indicated in note 2. When a suitable solvent mixture has been found, repeat the titration using the same solvent mixture.

Use the same final solvent mixture for the blank test (7.3) and record the type and volume of the solvent mixture in the test report (clause 9).

NOTES

1 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 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 preferred using glass electrodes with a suitable response time.

2 The type and volume of the solvent mixture 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.

7.3 Blank test

Carry out a blank test in parallel with the determination, by the same procedure, but omitting the test portion.

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

8 Expression of results

8.1 Calculation

Calculate the acid value, A, by the equation

$$A = 56{,}1 \frac{(V_1 - V_0) \times c}{m \times \mathrm{NV}} \times 100$$

where

A is the acid value, in milligrams of KOH per gram;

V_0 is the volume, in millilitres, of the potassium hydroxide solution (5.2), used for the blank test;

V_1 is the volume, in millilitres, of the potassium hydroxide solution (5.2), used for the determination;

c is the actual concentration, in moles of KOH per litre, of the potassium hydroxide solution (5.2) at the time of use;

m is the mass, in grams, of the test portion (7.1);

NV is the non-volatile matter content, expressed as a percentage by mass, determined according to ISO 3251.

Calculate the arithmetic mean of two determinations, and report the result to one decimal place.

8.2 Precision

8.2.1 Repeatability (r)

The value below which the absolute difference between two single test results on identical material, obtained by one operator in one laboratory using the same equipment within a short interval of time using the standardized test method, may be expected to lie with a 95 % probability, is 3 %.

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8.2.2 Reproducibility (R)

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The value below which the absolute difference between two results, each the mean of two determinations, on identical material, obtained by operators in different laboratories, using the standardized test method, may be expected to lie with a 95 % probability, is 5 %.

9 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 3682);

c) the acid value, expressed in milligrams of KOH per gram of the non-volatile matter of the product (mg KOH/g);

d) the type and volume of the solvent mixture (see 7.2);

e) the temperature and period of heating for the determination of the non-volatile matter content according to ISO 3251;

f) any deviation, by agreement or otherwise, from the procedure specified;

g) the date of the test.