BRITISH STANDARD

BS EN ISO 3682:1998
BS 6782-3: 1996
Incorporating Amendment No. 1 to BS 6782-3:1996 (renumbers the BS as BS EN ISO 3682:1998)

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

The European Standard EN ISO 3682:1998 has the status of a British Standard

ICS 87.060.20

Committees responsible for this British Standard

The preparation of this British Standard was entrusted to Technical Committee STI/3, Paints, media and related products, upon which the following bodies were represented:

British Coatings Federation Ltd.
European Resin Manufacturers' Association
FOSFA International
Ministry of Defence
Oil and Colour Chemists' Association
Seed Crushers' and Oil Processors' Association

This British Standard, having been prepared under the direction of the Sector Board for Materials and Chemicals, was published under the authority of the Standards Board and comes into effect on 15 November 1996

The following BSI references relate to the work on this standard:
Committee reference STI/3
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Amendments issued since publication

Amd. No.	Date	Comments
10150	November 1998	Renumbers BS 6782-3:1996 as BS EN ISO 3682:1998, with addition of EN ISO title page and foreword, and changes to national foreword

Contents

National foreword

This Part of BS 6782 has been prepared by Technical Committee STI/3 and is the English language version of EN ISO 3682:1998 *Binders for paints and varnishes — Determination of acid value — Titrimetric method*, published by the European Committee for Standardization (CEN). It is identical with ISO 3682:1996, published by the International Organization for Standardization (ISO).

This British Standard supersedes BS 6782-3:1987, which is withdrawn. The difference between this British Standard and BS 6782-3:1987 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.

Cross-references

The British Standards which implement international or European publications referred to in this document may be found in the BSI Standards Catalogue under the section entitled "International Standards Correspondence Index", or by using the "Find" facility of the BSI Standards Electronic Catalogue.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, the EN ISO title page, pages 2 to 5 and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN ISO 3682

April 1998

ICS 87.060.20

Descriptors: Paints, varnishes, binders (materials), tests, chemical tests, determination, acid number, volumetric analysis

English version

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

(ISO 3682:1996)

Liants pour peintures et vernis — Détermination de l'indice d'acide — Méthode titrimétrique
(ISO 3682:1996)

Bindemittel für Beschichtungsstoffe — Bestimmung der Säurezahl — Titrimetrisches Verfahren
(ISO 3682:1996)

This European Standard was approved by CEN on 9 March 1998.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart 36, B-1050 Brussels

Ref. No. EN ISO 3682:1998 E

Foreword

The text of the International Standard from Technical Committee ISO/TC 35, Paints and varnishes, of the International Organization for Standardization (ISO) has been taken over as an European Standard by Technical Committee CEN/TC 139, Paints and varnishes, the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by month of October 1998, and conflicting national standards shall be withdrawn at the latest by October 1998.

According to CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

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

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

where

V_0 is the volume, in millilitres, of potassium hydroxide solution (**5.2**) required for the blank test (**8.3**);

V_1 is the volume, in millilitres, of potassium hydroxide solution (**5.2**) required for the determination (**8.2**);

c is the actual concentration, in moles per litre, of the potassium hydroxide solution (**5.2**);

56,1 is the factor for the conversion of millilitres of potassium hydroxide solution, c(KOH) = 1 mol/l, to milligrams of potassium hydroxide;

m is the mass, in grams, of the test portion (**8.1**).

the two results (duplicates) differ by more than 3 % (relative to the mean), repeat the procedure described in clause **8**.

Report as the final result the mean, rounded to the nearest 0,1 mg KOH/g, of two valid results (replicates).

9.2 Precision

9.2.1 *Repeatability, r*

The value below which the absolute difference between two test results, each the mean of duplicates, obtained on identical material 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 %.

9.2.2 *Reproducibility, R*

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

10 Test report

The test report shall contain at least the following information:

a) all details necessary to identify the product tested;

b) a reference to this International Standard (ISO 3682);

c) the result of the test as indicated in **9.1**;

d) the type and volume of solvent mixture used (see **8.2**);

e) the type of titration: in the presence of a colour indicator (phenolphthalein, bromothymol blue or other) or potentiometric;

f) any deviation from the test method specified;

g) the date of the test.

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