

Adhesives — Determination of acid value

The European Standard EN 1241:1998 has the status of a
British Standard

ICS 83.180

National foreword

This British Standard is the English language version of EN 1241:1998.

The UK participation in its preparation was entrusted to Technical Committee STI/52, Adhesives, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
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Summary of pages

This document comprises a front cover, an inside front cover, the EN title page, pages 2 to 4, an inside back cover and a back cover.

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English version

Adhesives — Determination of acid value

Adhésifs — Détermination de l'indice d'acidité

Klebstoffe — Bestimmung der Säurezahl

This European Standard was approved by CEN on 2 January 1998.

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CEN

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

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

Foreword

This European Standard has been prepared by Technical Committee CEN/TC 193, Adhesives, the secretariat of which is held by AENOR.

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of an identical text or by endorsement, at the latest by August 1998, and conflicting national standards shall be withdrawn at the latest by August 1998.

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1 Scope

This standard specifies a method for determination of the acid value of adhesives, adhesive components, their basic constituents, and related products.

In some cases, notably when unsaturated polyesters with free anhydrides are present, somewhat lower than theoretical acid values are found, due to the formation of acid esters which precipitate and, are therefore, not determined.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

EN 923, *Adhesives — Terms and definitions.*

EN 1066, *Adhesives — Sampling.*

EN 1067, *Adhesives — Examination and preparation of samples for testing.*

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

ISO 648, *Laboratory glassware — One-mark pipettes.*

ISO 1042, *Laboratory glassware — One-mark volumetric flasks.*

3 Definition

For the purposes of this standard, the definitions in accordance with EN 923 and the following definition apply:

acid value

the number of milligrams of potassium hydroxide (KOH) required to neutralize 1 g of the test product under the test conditions

4 Principle

A weighed quantity of product is dissolved in a mixture of solvents and the product titrated with a standardized ethanolic solution of potassium hydroxide using a suitable indicator.

5 Safety

Persons using this standard shall be familiar with normal laboratory practice.

This standard does not purport to address all safety problems, if any, associated with its use.

It is the responsibility of the user to establish safety and health practices and to ensure compliance with any European and national regulatory conditions.

6 Reagents

6.1 Solvent mixture: mix two parts of toluene by volume, analytical grade, and one part of ethanol by volume, analytical grade.

6.2 Thymol blue (mass concentration $\beta = 0,1\%$) indicator solution: dissolve 0,1 g of Thymol blue in ethanol, analytical grade, and dilute to 100 ml.

6.3 Potassium hydroxide (KOH), 0,1 mol concentration, in ethanol, analytical grade. The solution shall be anhydrous and as free from carbon dioxide as possible.

NOTE Optionally 0,1 mol concentration in sodium hydroxide solution can be used, if required.

6.4 Acetone, analytical grade, containing less than 0,1 % (volume fraction) of water.

6.5 Nitrogen

7 Apparatus

All volumetric glassware shall be class A, in accordance with ISO 385-1, ISO 648 or ISO 1042 as appropriate.

7.1 Analytical balance, with scale divisions of 0,1 mg.

7.2 Conical flasks, capacity 250 ml, with large neck.

7.3 Burette, capacity 50 ml, graduated in 0,05 ml divisions.

7.4 Magnetic stirrer

7.5 Source of nitrogen gas

7.6 Pipettes, one mark, capacity 50 ml.

7.7 Apparatus for potentiometric titration, optional, for example, for dark coloured solutions.

8 Procedure

Take a sample of the product to be tested in accordance with EN 1066. Examine and prepare this sample for testing in accordance with EN 1067.

Weigh, to the nearest 1 mg, a test portion in the range of 0,5 g to 3,0 g \pm 1 mg, the quantity depending on the acid value expected.

Place this test portion in one of the flasks (see 7.2) and add 50 ml of the solvent mixture (see 6.1), using a pipette (see 7.6).

NOTE 1 Depending on the nature of the product under test other suitable solvents, e.g. dimethyl-formamide, or solvent mixture can be used provided the product is dissolved completely without chemical reaction.

For water-borne adhesives no solvent is required and aqueous standard volumetric potassium hydroxide solution free from carbon dioxide may be used. To avoid an excess of carbon dioxide, particularly to products having a low acid number, all products covered with nitrogen gas should be dissolved.

Shake until the product has completely dissolved, warming if necessary on a water bath with a condenser on the flask. If the solubility is poor, or if solution is incomplete after 5 min, prepare another solution by dissolving another test portion in a mixture of 50 ml of the solvent mixture (see 6.1) and 25 ml acetone (see 6.4). Record this change in procedure in the test report.

Cool the solution to (23 ± 2) °C.

Add 5 drops of thymol blue solution (see 6.2). Place the flask on the magnetic stirrer and bubble into the solution a stream of nitrogen.

Titrate with the potassium hydroxide solution (see 6.3) from the burette (see 7.3) to the endpoint where the colour remains blue for 20 to 30 s. Record the volume, V_1 , of KOH solution (see 6.3) used, in millilitres.

Carry out a blank determination using 50 ml of the solvent mixture (see 6.1) [or 50 ml of the solvent mixture (see 6.1) and 25 ml acetone (see 6.4) as described above] and titrate to obtain the same blue coloration as obtained when the product was present. Record the volume, V_2 , of KOH solution (see 6.3) used, in millilitres.

NOTE 2 Potentiometric titration is an option.

9 Calculation and expression of the results

9.1 Calculation

Calculate, for each test, the acid value, A_v , from the equation:

$$A_v = 56,1 \times M \times \frac{(V_1 - V_2)}{m}$$

where:

V_1 is the volume, in millilitres, of KOH solution (see 6.3) used to neutralize the solution of the product;

V_2 is the volume, in millilitres, of KOH solution (see 6.3) used in the blank determination;

M is the molarity of the KOH solution (see 6.3);

m is the mass, in grams, of the test portion.

9.2 Expression of results

Take as the result the mean of at least two determinations, rounded to the nearest whole number.

10 Test report

The test report shall include:

- a) a reference to this European Standard;
- b) a complete identification of the sample;
- c) identification of the solvent used for solution preparation, i.e. toluene/ethanol or toluene/ethanol/acetone and the end point detection used (by an indicator or potentiometry);
- d) test results, the individual acid values and their mean;
- e) any special features noted during the determination;
- f) any operations not specified in this European Standard and all incidents that may have affected the results;
- g) date of test.

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