BRITISH STANDARD

BS EN
1246:1998

Adhesives — Determination of ash and sulfated ash

The European Standard EN 1246:1998 has the status of a British Standard

ICS 83.180

National foreword

This British Standard is the English language version of EN 1246:1998.

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;

— monitor related international and European developments and promulgate them in the UK.

A list of organizations represented on this committee can be obtained on request to its secretary.

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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.

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 July 1998

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EUROPEAN STANDARD
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Descriptors: Adhesives, chemical analysis, ash determination, analysis methods

English version

Adhesives — Determination of ash and sulfated ash

Adhésifs — Détermination du taux de cendres total et du taux de cendres sulfatées

Klebstoffe — Bestimmung der Aschegehaltes und des Sulfat-Aschegehaltes

This European Standard was approved by CEN on 24 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

Ref. No. EN 1246:1998 E

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of an identical text or by endorsement, at the latest by August 1998, and conflicting national standards shall be withdrawn at the latest by August 1998.

According to the 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 European Standard specifies two conventional methods for the determination of ash and sulfated ash content of a synthetic or natural adhesive.

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 827, *Adhesives — Determination of conventional solids content and constant mass solids content.*

EN 923, *Adhesives — Terms and definitions.*

EN 1066, *Adhesive — Sampling.*

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

ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method.*

3 Definitions

For the purposes of this standard, the definitions given in EN 923 apply.

4 Principle

4.1 Method A: determination of the percentage of ash

Preliminary drying and combustion of the test sample and ashing in a muffle furnace at a predetermined temperature to constant mass.

4.2 Method B: determination of the percentage of ash as sulfates

Preliminary combustion of the test sample, treatment of the residue with sulfuric acid, heating, neutralization of excess acid by the addition of ammonium carbonate and heating. Then ashing in a muffle furnace at a predetermined temperature to constant mass.

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 appropriate safety and health practices and to ensure compliance with any European and national regulatory conditions.

6 Reagents (used only for sulfated ash)

6.1 *Sulfuric acid*, pure, ρ 1,84 g/ml.

6.2 *Ammonium carbonate*, anhydrous, powdered, analytical reagent grade.

7 Apparatus

7.1 *Crucibles, of silica, porcelain or platinum*, 45 mm to 75 mm diameter and a depth not less than the length of the diameter. Deep crucibles are particulary recommended, especially for the determination of sulfated ash.

7.2 *Bunsen burner or equivalent apparatus.*

7.3 *Muffle furnace, gas or electric*, controlled at (850 ± 20) °C.

7.4 *Oven*, adjustable between 80 °C and 135 °C.

7.5 *Analytical balance*, accurate to 0,0001 g.

7.6 *Desiccator*, containing conventional desiccant.

7.7 *Pipette.*

8 Sampling

A significant sample of the adhesive shall be taken in accordance with EN 1066 and prepared for testing in accordance with EN 1067.

9 Procedure

Carry out the following procedure in duplicate.

Calculate the mass m_1 in grams of ash from the difference in the weighings.

Calculate the mass m_{s1} in grams of sulfated ash from the difference in the weighings.

9.1 Method A: determination of the percentage of ash

9.1.1 Heat the crucible (see **7.1**) at (850 ± 20) °C to constant mass and cool in the desiccator (see **7.6**). Weigh the crucible to the nearest 0,0005 g and add 2 g to 3 g of adhesive. Reweigh the crucible and its contents to the nearest 0,0005 g and calculate the mass m_0 of the adhesive used.

9.1.2 Dry in the oven (see **7.4**) under the same conditions as those used to measure the conventional solids content in accordance with EN 827 for the adhesive involved.

Solid adhesives do not require this operation.

9.1.3 Gently heat the crucible with the Bunsen burner (see **7.2**), or equivalent apparatus, until all volatile matter and any other products of combustion are completely volatized. This is indicated by the disappearance of the black colour from the inside walls of the crucible.

The ashing shall be carried out slowly so that the volatiles do not carry off particles of ash.

9.1.4 Place the crucible in the muffle furnace (see **7.3**) controlled at (850 ± 20) °C and heat for 10 min.

After cooling in the desiccator, weigh the crucible to the nearest 0,0005 g.

Heat again for 10 min, re-cool in the desiccator and reweigh.

Repeat these operations until constant mass is obtained, i.e. until two successive weighings do not differ by more than 0,0005 g.

9.2 Method B: determination of the percentage of sulfated ash

9.2.1 Heat the crucible (see **7.1**) at (850 ± 20) °C to constant mass and cool in the desiccator (see **7.6**). Weigh the crucible to the nearest 0,0005 g and add 2 g to 3 g of adhesive. Reweigh the crucible and its contents to the nearest 0,0005 g and calculate the mass m_0 of the adhesive used.

9.2.2 Dry in the oven (see **7.4**) under the same conditions as those used to measure the conventional solids content in accordance with EN 827 for the adhesive involved.

Solid adhesives do not require this operation.

9.2.3 Gently heat the crucible with the Bunsen burner (see **7.2**) or equivalent apparatus until all volatile matter and any other products of combustion are completely volatized. This stage is indicated by the disappearance of the black colour from the inside walls of the crucible.

The ashing shall be carried out slowly so that the volatiles do not carry off particles of ash.

9.2.4 Allow the crucible and contents to cool.

By means of the pipette (see **7.7**) add to the crucible successive small quantities of sulfuric acid (see **6.1**), warming carefully with the bunsen burner (see **7.2**) until the reaction is completed.

Then, add ammonium carbonate (see **6.2**) to neutralize excess acid, warming until the emission of white fumes ceases.

9.2.5 Place the crucible in the muffle furnace (see **7.3**) controlled at (850 ± 20) °C and heat for 10 min.

After cooling in the desiccator, weigh the crucible to the nearest 0,0005 g.

Heat again for 10 min, re-cool in the desiccator and reweigh.

Repeat these operations until constant mass is obtained, i.e. until two successive weighings do not differ by more than 0,0005 g.

10 Expression of results

For each determination calculate the percentage of ash or sulfated ash to two decimal places by means of the formulae:

$$\text{ash } \% = \frac{m_1}{m_0} \times 100$$

$$\text{sulfated ash } \% = \frac{m_{s1}}{m_0} \times 100$$

where:

m_0 is the mass, in grams, of adhesive used;

m_1 is the mass, in grams, of ash;

m_{s1} is the mass, in grams, of sulfated ash.

Calculate the mean to the nearest 0,0005 grams of the two values finally obtained.

In the test report include the mean as the value of the percentage of ash or sulfated ash.

11 Precision

a) Repeatability (in accordance with ISO 5725-2): 0,10 %

b) Reproducibility (in accordance with ISO 5725-2): 0,20 %

12 Test report

The test report shall include:

a) a reference to this draft European Standard;

b) the type and designation of the adhesive tested;

c) the result of the test expressed in accordance with clause **10**;

d) any circumstances which may have affected the result;

e) date of test.

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