BS EN 1243:2011

BSI Standards Publication

Adhesives — Determination of free formaldehyde in amino and amidoformaldehyde condensates

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National foreword

This British Standard is the UK implementation of EN 1243:2011. It supersedes BS EN 1243:1999 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PRI/52, Adhesives.

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

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English Version

Adhesives - Determination of free formaldehyde in amino and amidoformaldehyde condensates

Adhésifs - Détermination du formaldéhyde libre dans les condensats de formaldéhyde amino et amido

Klebstoffe - Bestimmung des freien Formaldehydgehaltes in Amino- und Amido-Formaldehyd-Kondensaten

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Foreword

This document (EN 1243:2011) 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of an identical text or by endorsement, at the latest by October 2011, and conflicting national standards shall be withdrawn at the latest by October 2011.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 1243:1998.

The main modifications regarding the previous version are in the Foreword, Normative References and Clause 4.

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1 Scope

This European Standard specifies a method for the determination of the free formaldehyde content in amino and amido-formaldehyde condensate adhesives.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 923:2005+A1:2008, *Adhesives — Terms and definitions*

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

EN ISO 1042:1999, *Laboratory glassware — One-mark volumetric flasks (ISO 1042:1998)*

EN ISO 3696:1995, *Water for analytical laboratory use — Specification and test methods (ISO 3696:1987)*

EN ISO 15605, *Adhesives — Sampling (ISO 15605:2000)*

EN ISO 385:2005, *Laboratory glassware — Burettes (ISO 385:2005)*

ISO 648:2008, *Laboratory glassware — Single-volume pipettes (ISO 648:2008)*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 923:2005+A1:2008 and the following apply.

3.1
free formaldehyde
percentage (m/m) of unreacted formaldehyde in the product

4 Principle

Reaction of the free formaldehyde with sodium sulphite, in the presence of a measured excess of acid, in accordance with the reaction:

$$CH_2O + Na_2SO_3 + H_2O \rightarrow\rightarrow CH_2(OH)SO_3Na + NaOH$$

and alkaline titration of the unreacted excess acid.

The acid-sulphite mixture provides an essentially neutral buffered system which prevents hydrolysis of condensed formaldehyde. A reaction temperature close to 0 °C helps to ensure the absence of side reactions.

5 Reagents

5.1 Hydrochloric acid, aqueous 0,5 M standard volumetric solution.

NOTE Sulphuric acid 0,25 M can be used as an alternative.

5.2 **Sodium sulphite**, aqueous 1 M solution.

5.3 **Hydrochloric acid**, aqueous 0,1 M solution in sodium sulphite.

The solution shall be freshly prepared by mixing 80 ml of sodium sulphite solution (5.2) with 20 ml of hydrochloric acid 0,5 M solution (5.1).

NOTE Sulphuric acid can be used as an alternative.

5.4 **Sodium hydroxide**, aqueous 0,1 M standard volumetric solution.

5.5 **Dimethylsulphoxide**, aqueous 50 % v/v solution, or other solvents such as ethyleneglycol.

NOTE When using a solvent different from dimethylsulphoxide, preliminary comparative tests should be carried out to check possible side reactions.

5.6 **Thymolphthalein**, 0,1 % m/v solution in ethanol

5.7 **Deionized or distilled water**, grade 3 as specified in EN ISO 3696:1995.

6 Apparatus

6.1 Ordinary laboratory apparatus

All volumetric glassware shall be class A, conforming to EN ISO 385:2005, ISO 648:2008 or EN ISO 1042:1999 as appropriate.

6.2 **Analytical balance**, accurate to within 0,1 g.

6.3 **Conical flask**, capacity 300 ml.

6.4 **Ice** and **water bath.**

7 Sampling

Take a significant sample of the adhesive to be tested in accordance with EN ISO 15605. Homogenize as described in EN 1067.

8 Procedure

8.1 Disperse solid adhesives in water (5.7) at 50 % m/m and carry out the analysis within 2 h. Where the solid adhesive is a formulated type (with fillers and acid catalyst), neutralize the dispersion with sodium hydroxide (5.4) before the analysis.

The neutralization of formulate adhesive dispersion shall be done in the following way:

Weight the amount of dispersion to analyse and add sodium hydroxide (5.4) till pH 7,0 ± 0,2 and record the volume used (V_x). Throw away the sample.

Weigh the same amount of another sample of dispersion, add the volume of sodium hydroxide V_x and proceed as described in 8.2, 8.3 and 8.4.

8.2 Weigh, to the nearest 0,1 g, in a 300 ml conical flask (6.3) 5 g of liquid adhesive or dispersion (8.1); add 50 ml of dimethylsulphoxide solution (5.5) previously cooled and put the flask in the ice and water bath (6.4) for 20 min (internal temperature 5 °C).

8.3 Add rapidly, within 5 s, and whilst stirring, 30 ml of hydrochloric acid 0,1 M solution in sodium sulphite (5.3) previously cooled in the ice and water bath (6.4); wait 5 min to ensure complete reaction of the formaldehyde with sulphite; add 1 ml of thymolphthalein solution (5.6) and titrate immediately (in less than 1 min) the excess of acid with the sodium hydroxide 0,1 M solution (5.4) to the blue colour change. Record the volume, V_1, of the sodium hydroxide solution used, in millilitres.

NOTE A potentiometric titration can be used if the temperature and the time conditions are respected.

8.4 Carry out a blank test under the same conditions, but without the adhesive, and note the volume V_2, in millilitres, of sodium hydroxide solution used.

NOTE If the difference V_2 - V_1 is lower than 3 ml, the analysis should be repeated weighing a larger quantity of adhesive (i.e. 10 g).

9 Expression of results

The free formaldehyde content shall be calculated by the following formula:

$$\text{Free formaldehyde \%} = \frac{(V_2 \quad V_1) \times M \times 3{,}002}{m}$$

where

V_1 is the volume, in millilitres, of 0,1 M sodium hydroxide solution (5.4) used for the adhesive test;

V_2 is the volume, in millilitres, of 0,1 M sodium hydroxide solution (5.4) used for the blank test;

M is the molarity of sodium hydroxide solution (5.4);

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

NOTE For the solid adhesives "m" is ½ of the test portion (50 % dispersion).

10 Test report

The test report shall include:

a) reference to this European Standard, i.e EN 1243;

b) the type and designation of the adhesive tested;

c) the result of the test expressed in accordance with Clause 9;

d) any modification to the procedure described and any circumstances affecting the result;

e) date of the test.

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