



SLOVENSKI STANDARD

SIST EN 1243:2000

01-maj-2000

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Adhesives - Determination of free formaldehyde in amino and amidoformaldehyde condensates

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

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

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Ta slovenski standard je istoveten z: EN 1243:1998

ICS:

83.180

Lepila

Adhesives

SIST EN 1243:2000

en

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EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 1243

September 1998

ICS 83.180

Descriptors: adhesives, chemical tests, determination of content, condensates, formaldehyde, testing conditions

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

This European Standard was approved by CEN on 4 September 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.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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



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

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
EN ISO 3696	Water for analytical laboratory use - Specification and test methods
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

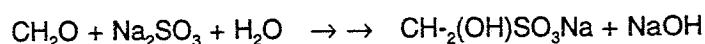
3 Definition

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

3.1 free formaldehyde: The 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:



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 of 0°C helps to ensure the absence of side reactions.

5 Safety

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

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

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

6 Reagents

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

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

6.2 Sodium sulphite, aqueous 1 M solution.

6.3 Hydrochloric acid, aqueous 0,1M solution in sodium sulfite. The solution shall be freshly prepared by mixing 80 ml of sodium sulphite solution (6.2) with 20 ml of hydrochloric acid 0,5 M solution (6.1).

NOTE: Sulphuric acid can be used as an alternative

6.4 Sodium hydroxide, aqueous 0,1M standard volumetric solution.

6.5 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.

6.6 Thymolphthalein, 0,1% m/v solution in ethanol

6.7 Deionized or distilled water, grade 3 as specified in EN ISO 3696.

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7 Apparatus

7.1 Ordinary laboratory apparatus.

All volumetric glassware shall be class A, conforming to ISO 385-1, ISO 648 or ISO 1042 as appropriate.

7.2 Analytical balance, accurate to within 0,1 g

7.3 Conical flask, capacity 300 ml

7.4 Ice and water bath

8 Sampling

Take a significant sample of the adhesive to be tested in accordance with EN 1066. Homogenize as described in EN 1067.

9 Procedure

9.1 Disperse solid adhesives in water (6.7) at 50% m/m and carry out the analysis within two hours. Where the solid adhesive is a formulated type (with fillers and acid catalyst), neutralize the dispersion with sodium hydroxide (6.4) before the analysis.

9.2 Weigh, to the nearest 0,1 g, in a 300 ml conical flask (7.3) 5 g of liquid adhesive or dispersion (9.1); add 50 ml of dimethylsulphoxide solution (6.5) previously cooled and put the flask in the ice and water bath (7.4) for 20 min (internal temperature 5°C).

9.3 Add rapidly, within 5 s, and whilst stirring, 30 ml of hydrochloric acid 0,1M solution in sodium sulphite (6.2) previously cooled in the ice and water bath (7.4); wait 5 min to ensure complete reaction of the formaldehyde with sulphite ; add 1 ml of thymolphthalein solution (6.6) and titrate immediately (in less than 1 min) the excess of acid with the sodium hydroxide 0,1M solution (6.4) 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.

9.4 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).

10 Expression of results

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

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

where

V_1 is the volume, in millilitres, of 0,1M sodium hydroxide solution (6.4) used for the adhesive test

V_2 is the volume, in millilitres, of 0,1M sodium hydroxide solution (6.4) used for the blank test

M is the molarity of sodium hydroxide solution (6.4)

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)

11 Test report

The test report shall include:

- a reference to this European Standard;
- the type and designation of the adhesive tested;
- the result of the test expressed in accordance with clause 10;
- any modification to the procedure described and any circumstances affecting the result;
- the date of the test.