SLOVENSKI STANDARD
SIST EN 1541:2000

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Papir, karton in lepenka v neposrednem stiku z ģivili - Doloļ evanje formaldehida v vodnem ekstraktu

Paper and board intended to come into contact with foodstuffs - Determination of formaldehyde in an aqueous extract

Papier und Pappe vorgesehen für den Kontakt mit Lebensmitteln - Bestimmung von Formaldehyd in einem wässrigen Extrakt

Papier et carton destinés a entrer en contact avec des denrées alimentaires - Détermination du formaldéhyde dans un extrait aqueux

Ta slovenski standard je istoveten z: **EN 1541:1998**

ICS:

67.250	Materiali in predmeti v stiku z živili	Materials and articles in contact with foodstuffs
85.060	Papir, karton in lepenka	Paper and board

SIST EN 1541:2000 **en**

EUROPEAN STANDARD
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EN 1541

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ICS 67.250; 85.060

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Descriptors: paper, paperboards, food products, food-container contact, chemical analysis, determination of content, formaldehyde, aqueous extract, molecular absorption spectrophotometry

English version

Paper and board intended to come into contact with foodstuffs - Determination of formaldehyde in an aqueous extract

Papiers et cartons destinés à entrer en contact avec des denrées alimentaires - Détermination du formaldéhyde dans un extrait aqueux

Papier und Pappe vorgesehen für den Kontakt mit Lebensmitteln - Bestimmung von Formaldehyd in einem wässrigen Extrakt

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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

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Content

Foreword

This European Standard has been prepared by Technical Committee CEN/TC 172 "Pulp, paper and board", the secretariat of which is held by DIN.

This European Standard supersedes EN 1541:1996.

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 December 1998, and conflicting national standards shall be withdrawn at the latest by December 1998.

With regard to EN 1541 : 1996 the following changes have been made:

a) time data in 8.5 have been completed by deviation data;
b) editorial updating.

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 the determination of formaldehyde in aqueous extracts prepared from paper and board intended to come into contact with foodstuffs. The limit of determination is 0,01 mg/dm² or 10 mg/kg paper or board.

For contact at room temperature t,he cold water extract is applied. For paper and board materials intended for boiling and hot filtering purposes, the hot water extract is applied.

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 645
Paper and board intended to come into contact with foodstuffs – Preparation of a cold water extract

EN 647
Paper and board intended to come into contact with foodstuffs – Preparation of a hot water extract

3 Definitions

For the purposes of this Standard the following definitions apply:

3.1 cold water extract: the water solution obtained as a result of cold extraction (see EN 645).

3.2 hot water extract: the water solution obtained as a result of hot extraction (see EN 647).

4 Principle

Formaldehyde reacts with pentane-2,4-dione (acetylacetone) in the presence of ammonium acetate to form 3,5-diacetyl-1,4-dihydrolutidine. The absorbance of the extract is measured at a wavelength of 410 nm.

NOTE: The extract should also be scanned by ultraviolet (UV) spectroscopy for confirmation where the level of formaldehyde exceeds specified limits (see clause 10).

5 Reagents

All reagents shall be of analytical grade and the water shall be distilled or of equivalent purity.

5.1 Anhydrous ammonium acetate

5.2 Acetic acid 99% (d = 1,05)

5.3 Pentane-2,4-dione distilled under reduced pressure (3,33 kPa) at 25° C

5.4 Butan-1-ol

5.5 Hydrochloric acid, 1 mol/l

5.6 Sodium hydroxide solution, 1 mol/l

5.7 Starch solution freshly prepared, 2g/l

5.8 Formaldehyde solution, 370 g/l to 400 g/l

5.9 Standard iodine solution, 0,05 mol/l

5.10 Standard sodium thiosulfate solution, 0,1 mol/l

5.11 Pentane-2,4-dione reagent

In a 100,0 ml volumetric flask dissolve:

- 15,0 g anhydrous ammonium acetate (5.1);
- 0,2 ml pentane-2,4-dione (5.3);
- 0,3 ml acetic acid (5.2);
- ≈ 25 ml of water.

Make up to 100,0 ml with water (pH of final solution about 6,4). This reagent shall be freshly prepared.

5.12 Reagent (5.11) without pentane-2,4-dione

5.13 Formaldehyde-standard: Stock solution

Measure 5,0 ml formaldehyde solution (5.8) into a 1000 ml volumetric flask and make up to 1000 ml with water.

Just before use determine the strength of this solution as follows.

Transfer 10,0 ml of the stock solution to a volumetric flask, add 25,0 ml of a standard iodine solution (5.9) and 10,0 ml of sodium hydroxide solution (5.6).

Allow to stand for 5 min.

Acidify with 11,0 ml of hydrochloric acid (5.5) and determine the excess iodine by titration with a standard sodium thiosulfate solution (5.10), using 0,1 ml of the starch solution (5.7) as indicator.

NOTE: Add the starch solution when the solution to be titrated has become a pale straw colour. Theoretically, 1,0 ml of 0,05 mol/l iodine consumed is equivalent to 1,5 mg formaldehyde.

5.14 Formaldehyde-standard: Dilute solution

Dilute an aliquot of the formaldehyde stock solution (5.13) to 20 times its volume with water, and then further dilute an aliquot of this second solution to 100 times its volume so that 1,0 ml of the final solution contains about 1 μg of formaldehyde.

Use pipettes and volumetric flasks.

Calculate the actual formaldehyde content.

This solution shall be freshly prepared.

6 Apparatus

6.1 Ordinary laboratory apparatus

6.2 Spectrometer for use at the wavelength of 410 nm, with cells having an optical path length of 10 mm

6.3 Scanning ultraviolet (UV) spectrometer in the range of 300 nm to 500 nm (Only for the confirmation steps).

6.4 Thermostatic water-bath capable of maintaining a temperature of (60 ± 2)° C

6.5 Phase separation filter

Phase separation filter capable of separating aqueous and solvent phases, i.e. retaining water and allowing the solvent to flow through.

Chemical resistance: resistance to mineral acids to 4,0 mol/l and alkalis to 0,4 mol/l.

NOTE 1: Pre-washing with solvent is recommended.

NOTE 2: An example of a phase separation filter is Whatman 1 PS[1].

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[1] Whatman is the trade-name of a product supplied by Whatman. This information is given for the convenience of users of this standard and does not constitute an endorsement by CEN of the product named. Equivalent products may be used if they can be shown to lead to the same results.

7 Preparation of sample

Sampling, sample preparation and extraction shall be carried out according to the methods for the preparation of cold water (see EN 645) or hot water extracts (see EN 647).

Two parallel extractions shall be carried out.

The test should be performed not longer than 24 h after extraction.

8 Procedure

8.1 For each extract at least two parallel determinations shall be carried out.

8.2 Sample solution

Into a 50 ml conical flask add

- 25,0 ml extract (V_1) (8.1);
- 5,0 ml pentane-2,4-dione reagent (5.11).

8.3 Reference solution

Possible interference due to coloured substances in the aqueous extract is eliminated by the use of this reference solution.

Into a 50 ml conical flask add

- 25,0 ml extract (8.1);
- 5,0 ml reagent without pentane-2,4-dione (5.12).

8.4 Blank test

This is performed in order to construct the calibration curve.

Into a 50 ml conical flask add

- 25,0 ml water;
- 5,0 ml pentane-2,4-dione reagent (5.11).

8.5 Determination

Shake the solutions 8.2, 8.3 and 8.4 for about 15 s. Immerse the conical flasks in a thermostatic waterbath (6.4) at (60 ± 2)° C for 10 min ± 10 s. Allow to cool for at least 2 min in a bath of iced water.

Measure the absorbance of the reference solution (8.3) against distilled water. If the absorbence exceeds 0,2, a cleaning step using butan-1-ol is necessary. In this case, transfer the contents of the conical flasks into 100 ml separating funnels containing 10,0 ml of butan-1-ol (5.4). Rinse each conical flask with 3,0 ml to 5,0 ml of water. Shake the mixtures vigorously for (30 ± 5) s. Allow the phases to separate. Filter the butan-1-ol phases into the measuring cells (6.2) through a phase separation filter (6.5).

Bring the solutions into the measuring cells (6.2). Measure the absorbance at 410 nm of the sample solution (8.2) with the reference solution (8.3) in the reference cell (A.1). Measure the absorbance of the blank test (8.4) with water in the reference cell, or with butan-1-ol (5.4) if the extraction with butanol has been performed (A.2).

The absorbance measurements shall be made between 35 min and 60 min from the time when the conical flasks were placed in the water bath at 60 °C.

8.6 Calibration curve

Into a 50 ml conical flask add:

- 1,0 ml of the formaldehyde diluted standard solution (5.14);
- 5,0 ml of the pentane-2,4-dione reagent (5.11);
- make up with water to 30,0 ml.

Continue as described in 8.5 and measure the absorbance with butan-1-ol (5.4) or water (see 8.5) in the reference cell.

Repeat the procedure with 5,0 ml; 10,0 ml; 15,0 ml; 20,0 ml and 25,0 ml of the formaldehyde diluted standard solution (5.14).

Construct the calibration curve after subtraction of the blank test value (see 8.5) from each of the absorbances obtained.

9 Expression of results

9.1 Subtract A2 from A1 and read off from the calibration curve (8.6) the amount C in mg of formaldehyde in the sample solution (8.2).

9.2 Calculate the formaldehyde content of sample in mg/dm² or in mg/kg as follows:

$$mg/dm^2 = C \cdot \frac{V_0}{V_1} \cdot \frac{b}{100} \cdot \frac{1}{G} \qquad (1)$$

$$mg/kg = C \cdot \frac{V_0}{V_1} \cdot \frac{1}{G} \cdot \frac{100}{100 - f} \qquad (2)$$

where:

C amount of formaldehyde read from the calibration graph, in mg;
V_0 total volume of extract (250 ml), in ml;
V_1 volume taken for the test (25,0 ml), in ml;
b grammage, in g/m²
G mass of the sample as taken, in g;
f moisture content of the sample, in %.

10 Confirmation

10.1 Requirement for confirmation

Where the level of formaldehyde in the water extract under test (7) exceeds any specified limit, the determination shall be confirmed by scanning ultraviolet (UV) spectroscopy.

10.2 Standard spectrum

Whilst preparing the formaldehyde derivative (8.5), scan the intermediate standard (10,0 ml standard from 8.6) from 300 nm to 500 nm. Record the position and absorbance value at the peak maximum and calculate the ratio of the measurements of the absorbance measured at 20 nm increments either side of the maximum.

The spectrum shall satisfy the following conditions:

a) the maximum shall be in the range from 408 nm to 411 nm;

b) the spectrum shall tend to zero absorbance, that is less than 0,02 absorbance units, below 320 nm.

Examples of the absorbance ratios to be expected are listed in Table 1.

Maximum absorbance : 410 nm

Table 1: Examples of absorbance ratios at corresponding wavelengths

Wavelength pair nm	Ratio
370 / 410	0,520 ± 0,02
390 / 410	0,843 ± 0,01
430 / 410	0,802 ± 0,01
450 / 410	0,386 ± 0,02

10.3 Sample spectrum

Following the procedure of 10.2, record the spectrum of the relevant formaldehyde solution, determining the absorption maxima and the absorption ratios. These ratios shall agree with those found for the standard to within ± 5%. If this criterion is satisfied, the level of formaldehyde found in equation (1) and/or (2) is confirmed.