BRITISH STANDARD

BS EN
1541:2001

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

The European Standard EN 1541:2001 has the status of a British Standard

ICS 67.250; 85.060

National foreword

This British Standard is the official English language version of EN 1541:2001. It supersedes BS EN 1541:1998 which is withdrawn.

The UK participation in its preparation was entrusted by Technical Committee CW/47, Materials in contact with food, to Subcommittee CW/47/3, Paper and board in contact with foodstuffs, 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 subcommittee can be obtained on request to its secretary.

Cross-references

The British Standards which implement international or European publications referred to in this document may be found in the BSI Standards Catalogue under the section entitled "International Standards Correspondence Index", or by using the "Find" facility of the BSI Standards Electronic Catalogue.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

This British Standard, having been prepared under the direction of the Consumer Products and Services Sector Committee, was published under the authority of the Standards Committee and comes into effect on 15 June 2001

Summary of pages

This document comprises a front cover, an inside front cover, the EN title page, pages 2 to 8, an inside back cover and a back cover.

The BSI copyright date displayed in this document indicates when the document was last issued.

Amendments issued since publication

Amd. No.	Date	Comments

ISBN 0 580 37328 2

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 1541

April 2001

ICS 67.250; 85.060

Supersedes EN 1541:1998

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 les 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

This European Standard was approved by CEN on 19 February 2001.

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 Management Centre 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 Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

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

Management Centre: rue de Stassart, 36 B-1050 Brussels

Ref. No. EN 1541:2001 E

Contents

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 shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by October 2001, and conflicting national standards shall be withdrawn at the latest by October 2001.

This European Standard supersedes EN 1541:1998.

With regard to EN 1541 : 1998-06 and EN 1541 : 1998/AC the following changes have been made:

a) deletion of the Butan-1-ol step;

b) correction of the formula 2 by incorporation of EN 1541 : 1998/AC;

c) addition of more precise information on the reproducibility, see 11.2;

d) 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 1 mg/kg. This means that for a paper with a grammage of 100 g/m² the limit of determination will be 0,001 mg/dm².

For contact at room temperature the 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 (including amendments).

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 Terms and definitions

For the purposes of this Standard the following terms and definitions apply:

3.1 cold water extract: the water solution obtained as a result of cold extraction [EN 645].

3.2 hot water extract: the water solution obtained as a result of hot extraction [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

5.4 Hydrochloric acid, 1 mol/l

5.5 Sodium hydroxide solution, 1 mol/l

5.6 Starch solution freshly prepared, 2 g/l

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

5.8 Standard iodine solution, 0,05 mol/l

5.9 Standard sodium thiosulphate solution, 0,1 mol/l

5.10 Pentane-2,4-dione reagent

In a 100,0 ml volumetric flask dissolve:

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

Make up to 100,0 ml with water. This reagent shall be freshly prepared.

5.11 Reagent (see 5.10) without pentane-2,4-dione

5.12 Formaldehyde-standard: Stock solution

Measure 5,0 ml formaldehyde solution (see 5.7) into a 1000 ml volumetric flask and make up to 1000 ml with water.

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

Transfer 10,0 ml of the stock solution into a conical flask, add 25,0 ml of a standard iodine solution (see 5.8) and 10,0 ml of sodium hydroxide solution (see 5.5).

Allow to stand for 5 min.

Acidify with 11,0 ml of hydrochloric acid (see 5.4) and determine the excess iodine by titration with a standard sodium thiosulphate solution (see 5.9), using 0,1 ml of the starch solution (see 5.6) as indicator.

NOTE Add the starch solution when the solution being 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.

5.13 Formaldehyde-standard: Dilute solution

Dilute an aliquot of the formaldehyde stock solution (see 5.12) 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 0,001 mg 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 of 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.

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 shall 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) (see 8.1);
- 5,0 ml pentane-2,4-dione reagent (see 5.10).

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 (see 8.1);
- 5,0 ml reagent without pentane-2,4-dione (see 5.11).

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 (see 5.10).

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 (see 6.4) at (60 ± 2)° for 10 min ± 10 s. Allow to cool for at least 2 min in a bath of iced water.

Bring the solutions into the measuring cells (see 6.2). Measure the absorbance at 410 nm of the sample solution (see 8.2) with the reference solution (see 8.3) in the reference cell (A_1).

Measure the absorbance of the blank test (see 8.4) with water in the reference cell (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.

If the value of formaldehyde obtained exceeds the range covered by the calibration solutions, the measurement shall be repeated with a more dilute sample solution and using an equally diluted reference solution.

8.6 Calibration curve

Into a 50 ml conical flask add:

- 1,0 ml of the formaldehyde diluted standard solution (see 5.13);
- 5,0 ml of the pentane-2,4-dione reagent (see 5.10);
- make up with water to 30,0 ml.

Continue as described in 8.5 and measure the absorbance with 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 (see 5.13).

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

9 Calculation and expression of results

9.1 Subtract A_2 from A_1 and read off from the calibration curve (see 8.6) the amount C in mg of formaldehyde in the sample solution (see 8.2).

9.2 Calculate the formaldehyde content of the sample (C_s) or (C_m) as follows:

$$C_s = C \times \frac{V_0}{V_1} \times \frac{b}{100} \times \frac{1}{G} \quad (1)$$

$$C_m = C \times \frac{V_0}{V_1} \times \frac{1}{G} \times \frac{100}{100 - f} \times 1000 \quad (2)$$

where:

C_s amount of formaldehyde soluble of the sample in mg/dm²;
C_m amount of formaldehyde soluble of the sample in mg/kg;
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, in grams, of the sample taken under the same condition as grammage;
f moisture content of the sample, in %.

Report the result with two significant figures.

10 Confirmation

10.1 Requirement for confirmation

Where the level of formaldehyde in the water extract under test (see clause 7) exceeds any specified limit, the determination shall be confirmed by scanning ultraviolet (UV) spectroscopy (see 6.3).

10.2 Standard spectrum

Whilst preparing the formaldehyde derivative (see 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.

11 Repeatability and reproducibility

11.1 Repeatability

From an interlaboratory test (n = 9) with both hot water and cold water extracts the test method described above gave repeatability (r) as shown in Table 2.

Table 2 - Repeatability found in an interlaboratory test

level	coefficient of variation (r)
0,10 mg/dm²	< 10%
0,40 mg/dm²	< 5 %

11.2 Reproducibility

From an interlaboratory test (n = 7) the test method described above gave a reproducibility, measured as the coefficient of variation (CV) between laboratories as shown in table 3. The figures include the variation in the water extraction and in the determination of grammage.

Table 3 - Reproducibility found in an interlaboratory test

Sample	cold water extract		hot water extract	
	mean mg/dm²	CV %	mean mg/dm²	CV %
1	0,024	18	0,097	10
2	0,18	11	0,27	20

NOTE The sample with the higher formaldehyde content was prepared especially for the interlaboratory test.

12 Test report

The test report shall refer to this European Standard and state:

a) extraction method;

b) nature, origin and designation of the sample;

c) date of sampling;

d) date of test;

e) mean result;

f) whether confirmation test has been carried out and if so, its result;

g) any deviations from this European Standard.

blank

BSI — British Standards Institution

BSI is the independent national body responsible for preparing British Standards. It presents the UK view on standards in Europe and at the international level. It is incorporated by Royal Charter.

Revisions

British Standards are updated by amendment or revision. Users of British Standards should make sure that they possess the latest amendments or editions.

It is the constant aim of BSI to improve the quality of our products and services. We would be grateful if anyone finding an inaccuracy or ambiguity while using this British Standard would inform the Secretary of the technical committee responsible, the identity of which can be found on the inside front cover. Tel: 020 8996 9000. Fax: 020 8996 7400.

BSI offers members an individual updating service called PLUS which ensures that subscribers automatically receive the latest editions of standards.

Buying standards

Orders for all BSI, international and foreign standards publications should be addressed to Customer Services. Tel: 020 8996 9001. Fax: 020 8996 7001. Standards are also available from the BSI website at http://www.bsi-global.com.

In response to orders for international standards, it is BSI policy to supply the BSI implementation of those that have been published as British Standards, unless otherwise requested.

Information on standards

BSI provides a wide range of information on national, European and international standards through its Library and its Technical Help to Exporters Service. Various BSI electronic information services are also available which give details on all its products and services. Contact the Information Centre. Tel: 020 8996 7111. Fax: 020 8996 7048.

Subscribing members of BSI are kept up to date with standards developments and receive substantial discounts on the purchase price of standards. For details of these and other benefits contact Membership Administration. Tel: 020 8996 7002. Fax: 020 8996 7001. Further information about BSI is available on the BSI website at http://www.bsi-global.com.

Copyright

BSI
389 Chiswick High Road
London
W4 4AL