

SLOVENSKI STANDARD

SIST-TS CEN/TS 13130-13:2005

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Materiali in predmeti v stiku z živili - Snovi v polimernih materialih, katerih koncentracija je omejena - 13. del: Določevanje 2,2-bis(4-hidroksifenil)propana (Bisfenola A) v modelnih raztopinah za živila

Materials and articles in contact with foodstuffs - Plastics substances subject to limitation - Part 13: Determination of 2,2-bis(4-hydroxyphenyl)propane (Bisphenol A) in food simulants

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Substanzen in Kunststoffen, die Beschränkungen unterliegen - Teil 13: Bestimmung von 2,2-Bis(4-Hydroxyphenyl)Propan (Bisphenol A) in Prüflebensmitteln

Matériaux et objets en contact avec les denrées alimentaires - Substances dans les matières plastiques soumises à des limitations - Partie 13 : Détermination du 2,2-bis(4-hydroxyphenyl) propane (Bisphénol A) dans les simulants d'aliments

Ta slovenski standard je istoveten z: CEN/TS 13130-13:2005

ICS:

67.250	Materiali in predmeti v stiku z živila	Materials and articles in contact with foodstuffs
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CEN/TS 13130-13

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ICS 67.250

English version

**Materials and articles in contact with foodstuffs - Plastics
substances subject to limitation - Part 13: Determination of 2,2-
bis(4-hydroxyphenyl)propane (Bisphenol A) in food simulants**

Matériaux et objets en contact avec les denrées
alimentaires - Substances dans les matières plastiques
soumises à des limitations - Partie 13 : Détermination du
2,2-bis(4-hydroxyphényl) propane (Bisphénol A) dans les
simulants d'aliments

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln
- Substanzen in Kunststoffen, die Beschränkungen
unterliegen - Teil 13: Bestimmung von 2,2-Bis(4-
Hydroxyphenyl)Propan (Bisphenol A) in Prüflebensmitteln

This Technical Specification (CEN/TS) was approved by CEN on 16 December 2004 for provisional application.

The period of validity of this CEN/TS is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the CEN/TS can be converted into a European Standard.

CEN members are required to announce the existence of this CEN/TS in the same way as for an EN and to make the CEN/TS available promptly at national level in an appropriate form. It is permissible to keep conflicting national standards in force (in parallel to the CEN/TS) until the final decision about the possible conversion of the CEN/TS into an EN is reached.

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Foreword

This document (CEN/TS 13130-13:2005) has been prepared by Technical Committee CEN/TC 194 "Utensils in contact with food", the secretariat of which is held by BSI.

This part of EN 13130 has been prepared within the Standards, Measurement and Testing project, MAT1-CT92-0006, "*Development of Methods of Analysis for Monomers*" and has been prepared by Subcommittee (SC 1) of TC 194 "Utensils in contact with food" as one of a series of test methods for plastics materials and articles in contact with foodstuffs.

This standard is intended to support Directives 2002/72/EC [1], 89/109/EEC [2], 82/711/EEC [3] and its amendments 93/8/EEC [4] and 97/48/EC [5], and 85/572/EEC [6].

At the time of preparation and publication of this part of EN 13130 the European Union legislation relating to plastics materials and articles intended to come into contact with foodstuffs is incomplete. Further Directives and amendments to existing Directives are expected which could change the legislative requirements which this standard supports. It is therefore strongly recommended that users of this standard refer to the latest relevant published Directive(s) before commencement of a test or tests described in this standard.

This part of EN 13130 should be read in conjunction with EN 13130-1.

Further parts of EN 13130, under the general title *Materials and articles in contact with foodstuffs - Plastics substances subject to limitation*, have been prepared, and others are in preparation, concerned with the determination of specific migration from plastics materials into foodstuffs and food simulants and the determination of specific monomers and additives in plastics. The parts of EN 13130 are as follows.

Part 1: *Guide to test methods for the specific migration of substances from plastics to foods and food simulants and the determination of substances in plastics and the selection of conditions of exposure to food simulants*

Part 2: *Determination of terephthalic acid in food simulants*

Part 3: *Determination of acrylonitrile in food and food simulants*

Part 4: *Determination of 1,3-butadiene in plastics*

Part 5: *Determination of vinylidene chloride in food simulants*

Part 6: *Determination of vinylidene chloride in plastics*

Part 7: *Determination of monoethylene glycol and diethylene glycol in food simulants*

Part 8: *Determination of isocyanates in plastics*

Part 9: *Determination of acetic acid, vinyl ester in food simulants*

Part 10: *Determination of acrylamide in food simulants*

Part 11: *Determination of 11-aminoundecanoic acid in food simulants*

Part 12: *Determination of 1,3-benzenedimethanamine in food simulants*

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Part 13: *Determination of 2,2-bis(4-hydroxyphenyl)propane (Bisphenol A) in food simulants*

Part 14: *Determination of 3,3-bis(3-methyl-4-hydroxyphenyl)-2-indoline in food simulants*

Part 15: *Determination of 1,3-butadiene in food simulants*

Part 16: *Determination of caprolactam and caprolactam salt in food simulants*

Part 17: *Determination of carbonyl chloride in plastics*

Part 18: *Determination of 1,2-dihydroxybenzene, 1,3-dihydroxybenzene, 1,4-dihydroxybenzene, 4,4'-dihydroxybenzophenone and 4,4'-dihydroxybiphenyl in food simulants*

Part 19: *Determination of dimethylaminoethanol in food simulants*

Part 20: *Determination of epichlorohydrin in plastics*

Part 21: *Determination of ethylenediamine and hexamethylenediamine in food simulants*

Part 22: *Determination of ethylene oxide and propylene oxide in plastics*

Part 23: *Determination of formaldehyde and hexamethylenetetramine in food simulants*

Part 24: *Determination of maleic acid and maleic anhydride in food simulants*

Part 25: *Determination of 4-methyl-pentene in food simulants*

Part 26: *Determination of 1-octene and tetrahydrofuran in food simulants*

Part 27: *Determination of 2,4,6-triamino-1,3,5-triazine in food simulants*

Part 28: *Determination of 1,1,1-trimethylolpropane in food simulants*

Parts 1 to 8 are European Standards. Parts 9 to 28 are Technical Specifications.

WARNING All chemicals are hazardous to health to a greater or lesser extent. It is beyond the scope of this Technical Specification to give instructions for the safe handling of all chemicals, that meet, in full, the legal obligations in all countries in which this Technical Specification may be followed. Therefore, specific warnings are not given and users of this Technical Specification should ensure that they meet all the necessary safety requirements in their own country.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to announce this CEN Technical Specification: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

Introduction

2,2-bis(4-hydroxyphenyl)propane, hereinafter referred to as Bisphenol A, $C_{15}H_{16}O_2$, PM/Ref. 13480, is a monomer used in the manufacture of certain plastics materials and articles intended to come into contact with foodstuffs. After manufacture residual Bisphenol A can remain in the finished product and may migrate into foodstuffs coming into contact with that product.

The method has been pre-validated by a collaborative trial with three laboratories.

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SIST-TS CEN/TS 13130-13:2005

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CEN/TS 13130-13:2005 (E)**1 Scope**

This document, part of EN 13130, specifies a method for the determination of Bisphenol A in the food simulants water, 3 % w/v acetic acid aqueous, 15 % v/v ethanol aqueous solution and rectified olive oil. The level of Bisphenol A monomer determined is expressed as milligrams Bisphenol A per kilogram of food simulant. The method is applicable to the quantitative determination of Bisphenol A at a minimum level of 0,2 mg/kg to 0,7 mg per kilogram of food simulants.

NOTE The method should also be applicable to other aqueous food simulants as well as to the other fatty food simulants, sunflower oil and a mixture of synthetic triglycerides.

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 13130-1:2004, *Materials and articles in contact with foodstuffs – Plastics substances subject to limitation – Part 1: Guide to test methods for the specific migration of substances from plastics to foods and food simulants and the determination of substances in plastics and the selection of conditions of exposure to food simulants.*

3 Principle

The level of Bisphenol A in aqueous food simulants is determined by high performance liquid chromatography (HPLC) with ultra violet (UV) detection. Olive oil test samples are extracted with a mixture of water/methanol and the resultant solution analyzed by HPLC. Calibration is achieved by analysis of relevant simulants containing known amounts of Bisphenol A. Confirmation of Bisphenol A is carried out by diode array detection.

4 Reagents

NOTE All reagents should be of recognized analytical quality unless otherwise stated.

4.1 Analyte

2,2-bis(4-hydroxyphenyl)propane (Bisphenol A or 4,4'-(methylethylidene)-bisphenol or 4,4'-isopropylidenediphenol), $C_{15}H_{16}O_2$, molecular weight: 228,28, purity > 99 %.

4.2 Chemicals

4.2.1 n-Hexane

4.2.2 Methanol

4.2.3 Water, deionized

4.3 Solutions

4.3.1 Extraction solvent, methanol/water = 1:1

Measure 100 ml of methanol (4.2.2) and 100 ml of water (4.2.3) and mix.

4.3.2 Mobile phase for HPLC, methanol/water = 70:30

Measure 500 ml of methanol (4.2.2) and 215 ml of water (4.2.3) and mix.

4.3.3 Stock solution of Bisphenol A in methanol at a defined concentration of approximately 0,38 mg/ml

Weigh to the nearest 0,1 mg approximately 37,5 mg of Bisphenol A (4.1.1) into a 100 ml volumetric flask. Dissolve the Bisphenol A in methanol and make up to the mark with methanol (4.2.2).

Calculate the concentration in micrograms of Bisphenol A per millilitre of solution.

Repeat the procedure to obtain a second stock solution.

NOTE The solution can be stored in a well closed container in dark for a maximum of 3 months at any temperature between +20 °C and –20 °C.

5 Apparatus

NOTE An instrument or item of apparatus is listed only where it is special or made to a particular specification, usual laboratory glassware and equipment being assumed to be available.

5.1 High performance liquid chromatograph, preferably, equipped with an automatic 20 µl loop injector and a variable wavelength UV detector connected to an integrator.

5.2 HPLC column, capable of separating Bisphenol A fully from peaks originating from the simulants and/or solvents used.

Appropriate operating conditions shall be established for the specific equipment used for the determination.

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NOTE The following column has been found to be suitable:

column: stainless steel 250 x 4,6 mm packed with C18 coated spherical silica gel, particle size 5 µm, (load of 9 % carbon and end-capped);

mobile phase: methanol/water 70 : 30 (4.3.2);

flow rate: 1 ml/min;

detection: UV 280 nm.

5.3 Mechanical shaker (Vortex)**5.4 Micro syringes, 10 µl, 50 µl and 1 000 µl.****5.5 Test tubes, volume 10 ml, size 10 cm x 1,5 cm.****6 Samples****6.1 Test sample preparation****6.1.1 General**

Laboratory samples of the food simulants to be analyzed shall be obtained as described in EN 13130-1. Samples shall be kept refrigerated at + 4 °C in closed containers with the exclusion of light. Analyte-free samples of relevant food simulants of the same type as those to be analyzed shall also be prepared for calibration purposes.

6.1.2 Aqueous solutions

Transfer approximately 1 ml of the food simulants obtained from the migration experiment (see EN 13130-1) into a vial suitable for HPLC injections.

6.1.3 Olive oil

Weigh 1 g ± 0,01 g of olive oil, obtained from the migration experiment (see EN 13130-1), into a test tube (5.5). Add by volumetric pipette 3,0 ml of n-hexane (4.2.1), mix well and add by volumetric pipette 2,0 ml methanol/water (4.3.1). Mix for 1 min with a mechanical shaker (5.3). Allow the phases to separate for 30 min. Retract by means of a pipette a part of the, lower, aqueous layer and transfer the solution into a vial suitable for HPLC injections.

6.2 Blank sample preparation

Treat food simulants which have not been in contact with packaging material in the same way as described in 6.1.

6.3 Calibration sample preparation**6.3.1 Aqueous food simulant calibration samples**

Transfer with a micro syringe (5.4) into a series of six 25 ml volumetric flasks 0 µl, 20 µl, 100 µl, 200 µl, 300 µl and 400 µl of the standard stock solution (4.3.3) and make up to the mark with the appropriate analyte-free food simulant, water, 3 % w/v aqueous acetic acid or 15 % v/v aqueous ethanol, and mix thoroughly. The calibration solutions thus obtained contain 0 µg/ml and approximately 0,30 µg/ml, 1,5 µg/ml, 3,0 µg/ml, 4,5 µg/ml or 6,0 µg of Bisphenol A per millilitre of food simulant.