SLOVENSKI STANDARD

oSIST prEN 13130-13:2006

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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 2,2bis(4-hydroxyphenyl)propane (Bisphenol A) in food simulants

Werstoffe 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 matieres plastiques soumises a des limitations - Partie 13 : Détermination du 2,2-bis(4-hydroxyphenyl)propane (Bisphénol A) dans les simulants d'aliments

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

Werstoffe 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 draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 194.

If this draft becomes a European Standard, 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.

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Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

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Foreword

This document (prEN 13130-13:2006) has been prepared by Technical Committee CEN/TC 194 "Utensils in contact with food", the secretariat of which is held by BSI.This document was prepared by Subcommittee SC 1 of TC 194 as one of a series of analytical test methods for plastics materials and articles in contact with foodstuffs.

This document is currently submitted to the CEN Enquiry.

This document will supersede CEN/TS 13130-13:2005.

At the time of preparation and publication of this standard the European Union legislation relating to plastics materials and articles intended to come into contact with foodstuffs is incomplete. Further Regulations, 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.

EN 13130-13 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 with the determination of specific monomers and additives in plastics. The other Parts of EN 13130 are as follows.

Part 1: *Guide to the test methods for the specific migration of substances from plastics into food 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 ethylene 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*

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-1-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*

Introduction

2,2-Bis(4-hydroxyphenyl)propane, herein 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.

1 Scope

This document, part of EN 13130, specifies a method for the determination of Bisphenol A in the food simulants distilled water, 3 % w/v acetic acid aqueous, 10 % 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 0,002 mg/kg in aqueous food simulants and 0,009 mg/kg in olive oil.

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

This European Standard incorporates by dated and 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 and 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 13130-1:2004, *Materials and articles in contact with foodstuffs – Plastics substances subject to limitation– Part 1: Guide to the test methods for the specific migration of substances from plastics into food 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 fluorescence (FLD) detection. Olive oil test samples are extracted with a mixture of water/methanol and the resultant solution analysed by HPLC. Calibration is achieved by analysis of relevant simulants containing known amounts of Bisphenol A.

4 Reagents

NOTE All reagents should be of recognised 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, deionised

4.3 Solutions

4.3.1 Extraction solvent, methanol/water = 85:100

Measure 85 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 0,75 mg/ml

Weigh to the nearest 0,1 mg approximately 75 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 (3.2.2).

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

Store the solution in a well closed container in the dark for a maximum period of 3 months at any temperature between +20 °C and –20 °C.

Repeat the procedure to provide a second stock solution. The two solutions should be cross-checked against one another. However, if other in-house quality systems are in place then these may be applicable instead.

4.3.4 Standard solution of Bisphenol A in methanol at a defined concentration of approximately 0,075 mg/ml

Transfer 2 ml of the stock solution of Bisphenol A (4.3.3) into a 20 ml volumetric flask and make up to the mark with methanol (3.2.2).

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

Repeat the procedure to obtain a second standard solution if applicable.

4.3.5 Dilute solution of Bisphenol A in methanol at a defined concentration of approximately 0,0075 mg/ml

Transfer 2 ml of the standard solution of Bisphenol A (4.3.4) into a 20 ml volumetric flask and make up to the mark with methanol (3.2.2).

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

Repeat the procedure to obtain a second dilute solution if applicable.

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 fluorescence 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.

NOTE For guidance, column and the parameters established for the column selected are as follows:.

column: stainless steel 150 × 3.0 mm packed with C18 coated spherical silica gel, particle size 5 µm, (load of 17,5 % carbon and end-capped)

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

flow rate: 0,5 ml/min

detection: fluorescence detection

excitation: 235 nm

emission: 317 nm

Alternative fluorescence excitation and emission wavelengths are:

Excitation: 275 nm

Emission: 305 nm

5.3 Mechanical shaker (Vortex)

5.4 Micro syringes, 10 µl, 50 µl, 100 µl, 250 µl and 500 µl

5.5 Test tube, 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 analysed 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). Transfer with a micro syringe (5.4) 160 µl methanol (4.2.2) to each of the test tubes. 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 eight 25 ml volumetric flasks 0 µl, 10 µl, 20 µl, 40 µl, 100 µl, 200 µl, 300 µl and 400 µl of the standard Bisphenol A solution (4.3.4) and make up to the mark with the appropriate analyte-free food simulant, water, 3 % w/v aqueous acetic acid or 10 % v/v aqueous ethanol, and mix thoroughly. The calibration solutions thus obtained contain 0 µg/ml and approximately 0,030 µg, 0,060 µg, 0,12 µg, 0,30 µg, 0,60 µg, 0,90 µg or 1,2 µg of Bisphenol A per millilitre of food simulant.

Calculate the exact concentrations of Bisphenol A in the calibration samples in micrograms per millilitre corresponding directly to milligrams per kilogram.

NOTE Commission Directive 2002/72/EC [2] states that the specific gravity of all simulants should conventionally be assumed to be ‘1’. Milligrams of substance released per litre of simulant will thus correspond numerically to milligrams of substance released per kilogram of simulant and, taking into account of the provisions laid down in Directive 82/711/EEC [3], to milligrams of substance released per kilogram of foodstuff.

For at least one food simulant, repeat the procedure using the second standard solution (4.3.4), if applicable.

6.3.2 Olive oil calibration samples

Weigh 1 g ± 0,01 g of olive oil into a series of eight test tubes. Transfer with a micro syringe (5.4) the following volumes of the dilute Bisphenol A solution (4.3.5) and methanol (4.2.2) to the test tubes:

Test tube number	1	2	3	4	5	6	7	8
Volume (µl) dilute Bisphenol A solution (4.3.5)	0	4	8	16	40	80	120	160
Volume (µl) methanol (4.2.2)	160	156	152	144	120	80	40	0

The calibration samples thus obtained contain 0 µg and approximately 0,030 µg, 0,060 µg, 0,12 µg, 0,30 µg 0,60 µg, 0.90 µg and 1,2 µg of Bisphenol A per gram of olive oil.

Add by volumetric pipette 3,0 ml of n-hexane (4.2.1), mix and add by volumetric pipette 2,0 ml of methanol/water (4.3.1). Shake for 1 min using a mechanical shaker (5.3). Allow the phases to separate. Retract by means of a pipette a part of the, lower, aqueous layer and transfer this solution into a vial suitable for HPLC injections.

Calculate the exact concentrations of Bisphenol A in the calibration samples in micrograms per gram olive corresponding directly to milligrams per kilogram.