SLOVENSKI STANDARD
SIST-TS CEN/TS 13130-14:2005

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Materiali in predmeti v stiku z živili - Snovi v polimernih materialih, katerih koncentracija je omejena - 14. del: Določevanje 3,3-bis(3-metil-4-hidroksifenil)-2-indolinona v modelnih raztopinah za živila

Materials and articles in contact with foodstuffs - Plastics substances subject to limitation - Part 14: Determination of 3,3-bis(3-methyl-4-hydroxyphenyl)-2-indoline in food simulants

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Substanzen in Kunststoffen, die Beschränkungen unterliegen - Teil 14: Bestimmung von 3,3-Bis(3-Methyl-4-Hydroxyphenyl)-2-Indolinon in Prüflebensmitteln

Matériaux et objets en contact avec les denrées alimentaires - Substances dans les matieres plastiques soumises a des limitations - Partie 14 : Détermination de la 3,3-bis(3 -méthyl-4-hydroxyphenyl)-2-indolinone dans les simulants d'aliments

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Materials and articles in contact with foodstuffs - Plastics substances subject to limitation - Part 14: Determination of 3,3-bis(3-methyl-4-hydroxyphenyl)-2-indoline in food simulants

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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 13130-14:2005) 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*

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

3,3-bis(3-methyl-4-hydroxyphenyl)-2-indolinone, PM/Ref. 13600, $C_{22}H_{19}NO_3$, is a monomer used in the manufacture of certain plastics materials and articles intended to come into contact with foodstuffs. After manufacture residual 3,3-bis(3-methyl-4-hydroxyphenyl)-2-indolinone can remain in the finished product and may migrate into foodstuffs coming into contact with that product.

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

1 Scope

This document, part of EN 13130, specifies an analytical procedure for the determination of 3,3-bis(3-methyl-4-hydroxyphenyl)-2-indolinone in the food simulants water, 3 % w/v aqueous acetic acid, 15 % v/v aqueous ethanol and olive oil or approved substitute. The level of 3,3-bis(3-methyl-4-hydroxyphenyl)-2-indolinone determined, is expressed as milligrams of 3,3-bis(3-methyl-4-hydroxyphenyl)-2-indolinone per kilogram of food or food simulant. The method is applicable to the quantitative determination of 3,3-bis(3-methyl-4-hydroxyphenyl)-2-indolinone in an approximate analyte concentration range of 0,18 mg/kg to 4/ kg of food simulants.

NOTE 1 The method should also be applicable to other aqueous food simulants as well as to the other fatty food simulants e.g. a mixture of synthetic triglycerides or sunflower oil.

NOTE 2 The suitability of the fat simulant should be assessed prior to setting up migration tests. Olive oil has been found to give unacceptable interference in some cases.

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 3,3-bis(3-methyl-4-hydroxyphenyl)-2-indolinone in aqueous food simulants is determined by reverse phase high performance liquid chromatography (HPLC) with UV detection at 235 nm. Fat simulants are extracted with 80 % aqueous acetonitrile prior to HPLC analysis. Quantification is achieved by calibration against samples of relevant food simulants, fortified with known amounts of 3,3-bis(3-methyl-4-hydroxyphenyl)-2-indolinone. The level of 3,3-bis(3-methyl-4-hydroxyphenyl)-2-indolinone determined, is expressed as milligrams per kilogram of food simulant.

Confirmation of 3,3-bis(3-methyl-4-hydroxyphenyl)-2-indolinone is carried out by reverse phase HPLC using an analytical column of different polarity with UV detection at 280 nm.

4 Reagents

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

4.1 Analyte

3,3-bis(3-methyl-4-hydroxyphenyl)-2-indolinone, ($C_{22}H_{19}NO_3$) purity greater than 98 % w/w.

4.2 Chemicals

4.2.1 Ethanol, absolute

4.2.2 Acetonitrile, chromatography grade

4.2.3 Water, HPLC grade

4.2.4 Orthophosphoric acid, 80 %

4.3 Solutions

4.3.1 Stock solution of 3,3-bis(3-methyl-4-hydroxyphenyl)-2-indolinone in ethanol (approximately 2 500 mg/l)

Weigh to the nearest 0,1 mg approximately 0,06 g of 3,3-bis(3-methyl-4-hydroxyphenyl)-2-indolinone (4.1) into a tared 25 ml volumetric flask. Half fill the flask with ethanol (4.2.1), shake thoroughly and make the volume up to the mark with ethanol (4.2.1).

Calculate the exact concentration of 3,3-bis(3-methyl-4-hydroxyphenyl)-2-indolinone in milligrams per litre.

Repeat the procedure to provide a second stock solution.

NOTE The stock solutions may be stored in the dark at room temperature for up to 3 months in stoppered glass volumetric flasks.

4.3.2 Intermediate standard solutions of 3,3-bis(3-methyl-4 -hydroxyphenyl)-2-indolinone in ethanol

Into six 25 ml volumetric flasks, add by pipette 0 ml, 0,1 ml, 0,5 ml, 1,0 ml, 2,0 ml and 4,0 ml of the stock solution (4.3.1). Dilute to the mark with ethanol (4.2.1) to give standard solutions containing nominal concentrations of approximately 0 mg/l, 10 mg/l, 50 mg/l, 100 mg/l, 200 mg/l and 400 mg per litre of 3,3-bis(3-methyl-4-hydroxyphenyl)-2-indolinone. Calculate the exact concentrations of the standard solutions in milligrams per litre.

Repeat the procedure using the second stock solution prepared in 4.3.1 to give a second set of intermediate standard solutions.

Store the standard solutions of 3,3-bis(3-methyl-4-hydroxyphenyl)-2-indolinone stored for up to 3 months in the stoppered glass flasks, protected from light, at room temperature.

4.3.3 Solvent A, HPLC mobile phase

Dissolve 0,7 ml of 80 % orthophosphoric acid (4.2.4) in 620 ml of water (4.2.3) and shake thoroughly to mix. Add 380 ml of acetonitrile (4.2.2) to the mixture.

4.3.4 Acetonitrile in water, 80 %

Transfer using a measuring cylinder 100 ml of water (4.2.3) and 400 ml of acetonitrile (4.2.2) to a 500 ml screw cap bottle, shake to mix thoroughly.