SLOVENSKI STANDARD
SIST-TS CEN/TS 13130-21:2005

01-april-2005

Materiali in predmeti v stiku z živili - Snovi v polimernih materialih, katerih koncentracija je omejena - 21. del: Določevanje etilendiamina in heksametilendiamina v modelnih raztopinah za živila

Materials and articles in contact with foodstuffs - Plastics substances subject to limitation - Part 21: Determination of ethylenediamine and hexamethylenediamine in food simulants

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Substanzen in Kunststoffen, die Beschränkungen unterliegen - Teil 21: Bestimmung von Ethylendiamin und Hexamethylendiamin in Prüflebensmitteln

Matériaux et objets en contact avec les denrées alimentaires - Substances dans les matieres plastiques soumises a des limitations - Partie 21 : Détermination de l'éthylenediamine et de l'hexaméthylenediamine dans les simulants d'aliments

Ta slovenski standard je istoveten z: **CEN/TS 13130-21:2005**

ICS:

67.250	Materiali in predmeti v stiku z živili	Materials and articles in contact with foodstuffs

SIST-TS CEN/TS 13130-21:2005 **en,fr,de**

TECHNICAL SPECIFICATION

SPÉCIFICATION TECHNIQUE

TECHNISCHE SPEZIFIKATION

CEN/TS 13130-21

February 2005

ICS 67.250

English version

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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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Ref. No. CEN/TS 13130-21:2005: E

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Foreword

This document (CEN/TS 13130-21:2005) 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

Ethylenediamine (EDA), 1,2-Diaminoethane, $C_2H_8N_2$ or $H_2N\text{-}(CH_2)_2\text{-}NH_2$, PM/Ref. No 16960, and hexamethylenediamine (HMDA), 1,6-Diaminohexane, $C_6H_{16}N_2$ or $H_2N\text{-}(CH_2)_6\text{-}NH_2$, PM/Ref. No 18460 are monomers used in the manufacture of certain plastics materials and articles intended to come into contact with foodstuffs. After manufacture, residual EDA monomer and HMDA monomer can remain in the polymer and may migrate into foodstuffs coming into contact with that plastics article.

NOTE Stability tests in olive oil food simulant carried out under test conditions 10 d at 20 °C and 10 d at 40 °C indicated that EDA and HMDA cannot be recovered, presumably due to chemical reaction with the olive oil. Therefore, even though this analytical method works in principle with olive oil as a food simulant, the migration test using olive oil or another fat simulant can provide false-negative results. Therefore, the method should only be applied in case of short exposure periods with olive oil and if applied, a recovery check with spiked olive oil applying the same time/temperature migration test conditions is recommended. Alternatively 95 % ethanol or iso-octane could be used as substitute fatty food simulants.

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

1 Scope

This document, part of EN 13130, specifies an analytical procedure for the determination of EDA monomer and HMDA monomer in the food simulants water, 3 % w/v aqueous acetic acid, 15 % v/v aqueous ethanol and olive oil as well as in the substitute food simulants 95 % v/v ethanol and iso-octane. The level of EDA monomer and HMDA monomer determined are expressed as milligram EDA per kilogram of food simulant and as milligrams HMDA per kilogram of food simulant. The method is appropriate for the quantitative determination of EDA at a minimum level of 1 mg per kilogram of food simulants and for the quantitative determination of HMDA at a minimum level of 0,5 mg per kilogram of food simulants.

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 EDA or HMDA in a food simulant is determined by derivatization of the free diamine, using ethyl chloroformate as derivatization agent followed by analysis of the resulting diurethane by gas chromatography using automated sample injection and flame ionization detection (FID). Quantification is achieved using propylenediamine (PDA) as an internal standard with calibration against relevant food simulants samples fortified with known amounts of EDA or HMDA. Confirmation of EDA or HMDA levels is carried out by combined gas chromatography/mass spectrometry (GC/MS) of the diurethane.

4 Reagents

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

4.1 Analytes

4.1.1 EDA (1,2-diaminoethane), H_2N-$(CH_2)_2$-NH_2, purity greater than 99 %.

4.1.2 HMDA (1,6-diaminohexane), H_2N-$(CH_2)_6$ -NH_2, purity greater than 97 %.

4.1.3 PDA (1,3-diaminopropane), H_2N-$(CH_2)_3$ -NH_2, purity greater than 99 %.

4.2 Chemicals

4.2.1 Ethyl chloroformate, Cl-C(O)-OC_2H_5, purity greater than 97 %.

4.2.2 Toluene

4.2.3 Diethyl ether

4.2.4 Distilled water

4.2.5 25 % (w/w) aqueous solution of ammonia, NH_3 (d^{20} = 0,91kg/l).

4.2.6 Sodium sulfate, anhydrous.

4.2.7 Sodium hydroxide, NaOH, pellets 99 %.

4.2.8 Acetic acid, glacial 100 %.

4.2.9 Purified nitrogen, 99,999 %.

4.3 Solutions

4.3.1 Solution of NH_3 in distilled water [approx. 3 % (w/w)]

Place 200 ml of distilled water (4.2.4) into a 250 ml Erlenmeyer flask, add 30 ml of 25 % aqueous NH_3 solution (4.2.5) and mix thoroughly.