SLOVENSKI STANDARD
SIST-TS CEN/TS 13130-26:2005

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Materiali in predmeti v stiku z živili - Snovi v polimernih materialih, katerih koncentracija je omejena - 26. del: Določevanje 1-oktena in tetrahidrofurana v modelnih raztopinah za živila

Materials and articles in contact with foodstuffs - Plastics substances subject to limitation - Part 26: Determination of 1-octene and tetrahydrofuran in food simulants

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Substanzen in Kunststoffen, die Beschränkungen unterliegen - Teil 26: Bestimmung von 1-Octen und Tetrahydrofuran in Prüflebensmitteln

Matériaux et objets en contact avec les denrées alimentaires - Substances dans les matieres plastiques soumises a des limitations - Partie 26 : Détermination du 1-octene et du tétrahydrofurane dans les simulants d'aliments

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Materials and articles in contact with foodstuffs - Plastics substances subject to limitation - Part 26: Determination of 1-octene and tetrahydrofuran in food simulants

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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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Contents

page

Foreword

This document (CEN/TS 13130-26:2005) 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

1-octene, C_8H_{16} or CH_2=CH-$(CH_2)_5$ –CH_3, PM/Ref. No 22660, and tetrahydrofuran (THF), C_4H_8O, PM/Ref. No 25150, are monomers used in the manufacture of certain plastics materials and articles intended to come into contact with foodstuffs. After manufacture, residual monomer can remain in the polymer and may migrate into foodstuffs coming into contact with that plastics article.

NOTE However, the following should be taken into account at carrying out a migration test for 1-octene. From migration experiments carried out for 10 d for 40 °C it was recognized that irreproducible loss of 1-octene, 11 % to 69 %, due to volatilization, can arise when using aqueous food simulants.

Method A describes the determination of 1-octene in food simulants.

Method B describes the determination of tetrahydrofuran in food simulants.

The methods have been pre-validated by collaborative trials with two laboratories.

1 Scope

This document, part of EN 13130, specifies analytical procedures for the determination of 1-octene and THF in food simulants water, 3 % w/v aqueous acetic acid, 15 % v/v aqueous ethanol and olive oil. The level of 1-octene and THF monomer determined is expressed as milligrams of monomer per kilogram of food simulant. The methods are appropriate for the quantitative determination of 1-octene in the range of 2 mg/kg to 30 mg/kg in food simulants and of THF in the range of 0,06 mg/kg to 1,5 mg/kg in food simulants.

NOTE The method should also be applicable to other aqueous food simulants as well as to the other fatty food simulants e.g. sunflower oil, corn oil or 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 Method A – Determination of 1-octene in food simulants

3.1 Principle

The level of 1-octene in a food or a food simulant is determined by headspace gas chromatography (HSGC) of the food simulant sample, sealed in headspace glass vials. Headspace gas chromatography is carried out applying automatic injection and flame ionization detection. Quantification is achieved using iso-octane as internal standard with calibration against food simulant samples fortified with known amounts of 1-octene. Confirmation of 1-octene levels is carried out by combined gas chromatography/mass spectrometry (GC/MS).

3.2 Reagents

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

3.2.1 Analytes

3.2.1.1 1-octene, CH_2=CH-$(CH_2)_5$ –CH_3, purity greater than 97,5 % (GC).

3.2.1.2 Iso-octane, $(CH_3)_2$-CH-CH_2-C$(CH_3)_3$, purity greater than 99 % (GC).

3.2.2 Chemical

N,N-dimethylacetamide (DMAA), CH_3-CO-N$(CH_3)_2$, purity greater than 99 %.

3.2.3 Solutions

3.2.3.1 Stock solutions of 1-octene with a defined concentration of approximately 2 mg/ml in DMAA

Weigh a 50 ml volumetric flask, including cap, filled with about 45 ml of DMAA (3.2.2), to an accuracy of 0,1 mg. Add approximately 100 mg of 1-octene (about 150 µl), then reweigh to an accuracy of 0,1 mg. Make up to 50 ml with DMAA. Close and shake.

Calculate the exact concentration of the stock solution in milligrams of 1-octene per millilitre of solution.

Repeat the procedure to provide a second stock solution.

NOTE These stock solutions can be stored at + 4 °C, with the exclusion of light, for up to 3 months.

3.2.3.2 Standard solutions of 1-octene in DMAA with a defined concentration of approximately 200 µg/ml

Place 1,0 ml of the 1-octene stock solution (3.2.3.1) in a 10 ml volumetric flask and make up to the mark with DMAA (3.2.2). Close and mix thoroughly.

Calculate the exact concentration of the standard solution in milligrams of 1-octene per millilitre of solution.

Repeat the procedure using the second stock solution prepared in 3.2.3.1 to provide a second standard solution.

NOTE These standard solutions can be stored at + 4 °C and with the exclusion of light for up to 3 months.

3.2.3.3 Stock solution of internal standard iso-octane in DMAA at a defined concentration of approximately 2 mg/ml

Weigh a 50 ml volumetric flask, including cap, filled with about 45 ml of DMAA (3.2.2), to an accuracy of 0,1 mg. Add approximately 100 mg of iso-octane (about 150 µl), then reweigh to an accuracy of 0,1 mg. Make up to the mark with DMAA close and mix.

Calculate the exact concentration of the stock solution in milligrams of iso-octane per millilitre of solution.

NOTE This stock solution can be stored at + 4 °C, with the exclusion of light, for up to 3 months.

3.2.3.4 Internal standard solution of iso-octane in DMAA at a defined concentration of approximately 120 µg/ml

Place 3,0 ml of internal standard stock solution (3.2.3.3) in a 50 ml volumetric flask and make up to the mark with DMAA. Close and mix thoroughly.

Calculate the exact concentration of the internal standard solution in milligrams of iso-octane per millilitre of solution.

NOTE This internal standard can be stored at + 4 °C, with the exclusion of light, for up to 3 months.

3.3 Apparatus

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

3.3.1 Gas chromatograph, equipped with a flame ionization detector (FID) and fitted with an automatic headspace sampler.

3.3.2 Gas chromatographic column, capable of the separation of DMAA from 1-octene and iso-octane, such that the peaks of 1-octene and iso-octane do not overlap by more than 1 % peak area with other compounds.

NOTE The following are examples of GC columns known to be suitable for 1-octene analysis:

a) 50 m x 0,53 mm i.d. fused silica capillary column, coated with a phenyl-methyl silicone phase, film thickness 2,5 µm.

For guidance, the operating conditions established for the column described above, were the following:

Headspace sampler:	
Sample thermostatting time:	80 min
Sample temperature:	70 °C
Transfer line temperature:	130 °C
Needle temperature:	85 °C
Pressure equilibration time:	3 min
Injection time:	6 sec
Gas chromatograph:	
Injector:	150 °C
Detector:	220 °C
Oven program:	110 °C (11 min), 10 °C/min to 150 °C (5 min)
Carrier gas:	Nitrogen at 250 kPa
Linear velocity:	35 cm/sec
Injection mode:	total injection
FID gases:	to be optimized according to the manufacturer's specification

Alternatively, the following system has been found to be suitable:

b) Column: 30 m x 0,2 mm i.d. fused silica capillary column, coated with a 5 % diphenyl and 95 % dimethyl silicone phase, film thickness 0,33 µm

Headspace sampler:	
Sample thermostatting time:	60 min
Sample temperature:	100 °C
Gas chromatograph:	
Carrier gas:	Helium at 90 kPa and 1 ml/min
Oven program:	75 °C (8 min), 25 °C/min to 250 °C (5 min)
Injection mode:	split (ratio 1:5)

3.3.3 Glass sample vials, 20 ml or another size suitable for the particular autosampler employed, with polytetrafluroethylene (PTFE) coated butyl or silicone rubber septa and aluminium crimp-cap closures.

3.3.4 Volumetric flasks, 50 ml and 10 ml.

3.3.5 Microsyringes, 250 µl and 100 µl.

3.3.6 Volumetric pipettes, 1 ml and 3 ml.

3.4 Samples

3.4.1 General

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

Take into account the possible loss of analyte due to volatilization in aqueous food simulants (see NOTE in the Introduction).

3.4.2 Test sample preparation

3.4.2.1 Aqueous food simulants

Place 1,0 ml of the food simulant obtained from the migration experiment into a sample vial (3.3.3) using a volumetric pipette (3.3.6), close immediately the vial with a septum and cap. Add 200 µl iso-octane internal standard solution (3.2.3.4) followed by 200 µl DMAA (3.2.2) to the food simulant by injection through the septum using the 250 µl microsyringe (3.3.5).

Prepare each test sample at least in duplicate.

3.4.2.2 Olive oil

Weigh 1,0 g ± 0,01 g of the food simulant, as obtained from the migration experiment into a sample vial (3.3.3). Close the vial immediately with a septum and cap. Add 200 µl iso-octane internal standard solution (3.2.3.4) followed by 200 µl DMAA (3.2.2) to the olive oil test sample by injection through the septum using the 250 µl microsyringe (3.3.5) and mix thoroughly.

Prepare each test sample at least in duplicate.

3.4.3 Blank sample preparation

3.4.3.1 Aqueous food simulants

Place 1,0 ml of aqueous 1-octene-free food simulant into a sample vial (3.3.3) using a 1,0 ml volumetric pipette (3.3.6), then cap immediately. Add 200 µl iso-octane internal standard solution (3.2.3.4) followed by 200 µl of DMAA (3.2.2) through the septum using the 250 µl microsyringe (3.3.5).

Prepare each blank sample at least in duplicate.