SLOVENSKI STANDARD
SIST-TS CEN/TS 13130-9:2005

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Materiali in predmeti v stiku z živili - Snovi v polimernih materialih, katerih koncentracija je omejena - 9. del: Določevanje vinilacetata v modelnih raztopinah za živila

Materials and articles in contact with foodstuffs - Plastics substances subject to limitation - Part 9: Determination of acetic acid, vinyl ester in food simulants

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Substanzen in Kunststoffen, die Beschränkungen unterliegen - Teil 9: Bestimmung von Essigsäurevinylester in Prüflebensmitteln

Matériaux et objets en contact avec des denrées alimentaires - Substances dans les matieres plastiques soumises a des limitations - Partie 9 : Détermination du vinyl ester d'acide acétique dans les simulants d'aliments

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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 13130-9:2005) 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

Acetic acid, vinyl ester (vinyl acetate) $CH_3CO_2CH=CH_2$, PM/Ref. No 10120, is a monomer used in the manufacture of certain materials and articles intended to come into contact with foodstuffs. After manufacture, residual vinyl acetate can remain in the polymer and may migrate into foodstuffs coming into contact with the plastic material or product.

NOTE The following should be taken into account when carrying out a migration test. Vinyl acetate is known to hydrolyze in aqueous media. In acetic acid solutions the hydrolysis is almost complete after 10 d at 40 °C. Vinyl acetate is stable in olive oil.

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

1 Scope

This document, part of EN 13130, specifies an analytical procedure for the determination of vinyl acetate in the four conventional EU food simulants, water, 10 % (v/v) ethanol aqueous solution; 3 % (w/v) acetic acid aqueous solution and olive oil or an approved substitute. The level of vinyl acetate monomer determined is expressed as milligrams of vinyl acetate per kilogram of food simulant. The method is appropriate for the quantitative determination of vinyl acetate in approximate analyte concentration range of 1,2 mg/kg to 24 mg/kg food simulant.

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

NOTE 2 The suitability of the fat simulant should be assessed prior to setting up migration tests - it may be found necessary to use sunflower oil or a mixture of synthetic triglycerides if unacceptable interferences are found with olive oil.

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 vinyl acetate in food simulants is determined by headspace capillary gas chromatography, using flame ionization detection. Quantification is achieved using propionic acid, methyl ester (methyl propionate), as an internal standard with calibration against relevant food simulants samples, fortified with known amounts of vinyl acetate.

Confirmation of vinyl acetate levels is carried out by combined headspace gas chromatography/mass spectrometry, or by repeating the analytical procedure using a gas chromatograph capillary column of different polarity.

If automated headspace sampling cannot be performed, manual injection may be used ensuring that the gas syringe and needle are preheated to the same temperature as the headspace vials before the headspace is sampled.

4 Reagents

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

4.1 Analytes

4.1.1 Vinyl acetate, $CH_3CO_2CH=CH_2$, purity greater than 99 %.

4.1.2 Methyl propionate, $CH_3CH_2CO_2CH_3$, purity greater than 99 %, which is free of interfering substances which elute at the same retention time as vinyl acetate.

4.2 Chemicals

4.2.1 Water, HPLC grade.

4.2.2 N,N-dimethylacetamide (99,9 %)

4.3 Solutions

4.3.1 Stock solution of vinyl acetate in dimethylacetamide (6 mg/ml)

Weigh accurately approximately 0,15 g of the vinyl acetate into a tared stoppered 25 ml volumetric flask containing about 10 ml of dimethylacetamide, and dilute to the mark with dimethylacetamide. Calculate the exact concentration of vinyl acetate in milligrams per litre.

Repeat the procedure to provide a second standard stock solution.

NOTE The stock solutions can be stored in a well closed containers in the dark for a maximum period of three months at any temperature between 5 °C and 20 °C.

4.3.2 Internal standard stock solution of propionic acid, methyl ester (methyl propionate) in dimethylacetamide (6 mg/ml)

Weigh approximately 0,6 g of propionic acid methyl ester (methyl propionate), into a tared stoppered 100 ml volumetric flask containing about 20 ml of dimethylacetamide, and dilute to the mark with dimethylacetamide.

NOTE The stock solutions can be stored in well closed containers in the dark for a maximum period of three months at any temperature between 5 °C and 20 °C.

4.3.3 Standard solutions of vinyl acetate in dimethylacetamide

Into six 25 ml volumetric flasks, each containing approximately 5 ml of dimethylacetamide, add by pipette 0 ml, 0,5 ml, 1,0 ml, 2,0 ml, 3,0 ml and 5,0 ml of the stock solution (3.3.1). Pipette in 5,0 ml of internal standard solution (4.3.2). Dilute to the mark with dimethylacetamide to give standard solutions containing a nominal concentration of 0 mg/l, 120 mg/l, 240 mg/l, 480 mg/l, 720 mg/l and 1200 mg/l vinyl acetate. Shake thoroughly to mix.

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 standard solutions.

NOTE The standard solutions can be stored in well closed containers in the dark for a maximum period of three months at any temperature between 5 °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, the usual laboratory glassware and equipment being assumed to be available.