SLOVENSKI STANDARD
SIST-TS CEN/TS 13130-24:2005

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Materiali in predmeti v stiku z ǵivili - Snovi v polimernih materialih, katerih koncentracija je omejena - 24. del: Dolo ļ evanje maleinske kisline in anhidrida maleinske kisline v modelnih raztopinah za ǵivila

Materials and articles in contact with foodstuffs - Plastics substances subject to limitation - Part 24: Determination of maleic acid and maleic anhydride in food simulants

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Substanzen in Kunststoffen, die Beschränkungen unterliegen - Teil 24: Bestimmung von Maleinsäure und Maleinanhydrid in Prüflebensmitteln

Matériaux et objets en contact avec les denrées alimentaires - Substances dans les matieres plastiques soumises a des limitations - Partie 24 : Détermination de l'acide maléique et de l'anhydre maléique dans les simulants d'aliments

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Materials and articles in contact with foodstuffs - Plastics substances subject to limitation - Part 24: Determination of maleic acid and maleic anhydride in food simulants

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Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Substanzen in Kunststoffen, die Beschränkungen unterliegen - Teil 24: Bestimmung von Maleinsäure und Maleinanhydrid in Prüflebensmitteln

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.

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Foreword

This document (CEN/TS 13130-24:2005) 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

Maleic acid, $C_4H_4O_4$, PM/Ref. No 19540, and maleic anhydride, $C_4H_2O_3$, PM/Ref. No 19960, are monomers used in the manufacture of certain plastics materials and articles intended to come into contact with foodstuffs. After manufacture residual maleic acid and maleic anhydride can remain in the finished product and may migrate into foodstuffs coming into contact with that product.

This method describes the determination of the specific migration of maleic anhydride as maleic acid.

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

1 Scope

This document, part of EN 13130, specifies an analytical procedure for the determination of maleic acid in the four conventional food simulants, water, 3 % w/v aqueous acetic acid, 15 % v/v aqueous ethanol and olive oil or approved substitute. The level of maleic acid determined is expressed as milligrams of maleic acid per kilogram of food or food simulant. The method is appropriate for the quantitative determination of maleic acid in approximate analyte concentration range of 5 mg/kg to 60 mg/kg in the above mentioned food simulants.

This method is also suitable for the determination of maleic anhydride in the four food simulants as the anhydride is hydrolyzed rapidly and completely to maleic acid during sample preparation. The level of maleic anhydride is then expressed as milligrams of maleic acid per kilogram of food simulant.

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. 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 HB 307 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 maleic acid is determined by reverse phase, ion pair high performance liquid chromatography (HPLC) with UV detection at 220 nm. Maleic anhydride hydrolyzes rapidly and completely to maleic acid during sample preparation. Quantification of maleic acid is achieved using citraconic acid, as an internal standard, with calibration against relevant food simulants samples, fortified with known amounts of maleic acid.

Confirmation of the maleic acid level is carried out by ion exchange chromatography with UV detection at 245 nm.

4 Reagents

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

4.1 Analytes

4.1.1 Maleic acid, $HO_2CCH=CHCO_2H$ purity greater than 98 %.

4.1.2 Citraconic acid, $HO_2CC(CH_3)=CHCO_2H$ of purity greater than 98 % w/w and not containing maleic acid.

4.1.3 Mesaconic acid, $HO_2CH=C(CH_3).CO_2H$ of purity greater than 98 % w/w and not containing maleic acid.

NOTE Mesaconic acid may be used as an alternative internal standard.

4.2 Chemicals

4.2.1 Ethanol, absolute.

4.2.2 Sodium hydrogen carbonate

4.2.3 Water, HPLC grade.

4.2.4 Dipotassium hydrogen orthophosphate, $K_2HPO_4.3\ H_2O$.

4.2.5 Potassium dihydrogen orthophosphate, KH_2PO_4.

4.2.6 Orthophosphoric acid (85 %)

4.2.7 Sodium hydroxide, 2 mol/l solution in water.

4.2.8 Cetyl trimethyl ammonium bromide, HPLC grade.

4.2.9 Disodium tetraborate

4.2.10 Ammonium chloride

4.2.11 Acetonitrile

4.3 Solutions

4.3.1 Stock solution of maleic acid in ethanol (7 500 mg/l)

Weigh accurately approximately 0,75 g of the maleic acid (4.1.1) into a tared 100 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 actual concentration of maleic acid in milligrams per litre.

Repeat the procedure to provide a second stock solution.

NOTE The stock solutions can be stored at – 20 °C for up to 2 months in stoppered glass volumetric flasks.