SLOVENSKI STANDARD
SIST EN 13130-2:2004

01-september-2004

Nadomeģ a:
SIST ENV 13130-2:2000

Materiali in predmeti v stiku z ģivili - Snovi v polimernih materialih, katerih koncentracija je omejena - 2. del: Dolo ev anje tereftalne kisline v modelnih raztopinah za ģivila

Materials and articles in contact with foodstuffs - Plastics substances subject to limitation - Part 2: Determination of terephthalic acid in food simulants

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Substanzen in Kunststoffen, die Beschränkungen unterliegen - Teil 2: Bestimmung von Terephtalsäure in Prüflebensmitteln

Matériaux et objets en contact avec les denrées alimentaires - Substances dans les matieres plastiques soumises a des limitations - Partie 2 : Détermination de l'acide téréphtalique dans les simulants d'aliments

Ta slovenski standard je istoveten z: **EN 13130-2:2004**

ICS:

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

SIST EN 13130-2:2004 **en,fr,de**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 13130-2

May 2004

ICS 67.250

English version

Materials and articles in contact with foodstuffs - Plastics substances subject to limitation - Part 2: Determination of terephthalic acid in food simulants

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This European Standard was approved by CEN on 24 March 2004.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of 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.

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Foreword

This document (EN 13130-2:2004) has been prepared by Technical Committee CEN/TC 194 “Utensils in contact with food”, the secretariat of which is held by BSI.

This document was prepared by Subcommittee SC1 of TC 194 as one of a series of analytical test methods for plastics materials and articles in contact with foodstuffs.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by November 2004, and conflicting national standards shall be withdrawn at the latest by November 2004.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

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.

EN 13130-2 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 other 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 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-trimethylopropane in food simulants*

Parts 1 to 8 are European Standards.

Parts 9 to 28 are Technical Specifications, prepared within the Standards, Measurement and Testing project, MAT1-CT92-0006, "*Development of Methods of Analysis for Monomers*".

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: 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

Terephthalic acid (TA) is a comonomer used in the manufacture of polyester plastics. Residues of terephthalic acid can be present in the plastics after processing to form materials and articles intended to come into contact with foodstuffs. When these plastics are in contact with foodstuffs, the residual terephthalic acid monomer can migrate into the foodstuff.

1 Scope

This part of this European Standard specifies methods for the determination of the monomer terephthalic acid in food simulants; distilled water, 3 % (w/v) acetic acid aqueous solution, 15 % (v/v) ethanol aqueous solution and olive oil and other fatty food simulants, simulants D, e.g. a mixture of synthetic triglycerides or sunflower oil or corn oil. The methods are capable of determining terephthalic acid in the food simulants at the level of the specific migration limit of 7,5 mg of terephthalic acid per kilogram of food simulants.

NOTE This method was developed for the determination of terephthalic acid in 15 % (v/v) aqueous ethanol, as required by the regulations in force at the time the development work was carried out. However, this method, developed for 15 (v/v) aqueous ethanol, should be applicable to the determination in 10 (v/v) aqueous ethanol.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text, and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

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*

EN ISO 1042; *Laboratory glassware – One-mark volumetric flasks (ISO 1042:1998).*

ISO 385 (all parts); *Laboratory glassware – Burettes.*

ISO 648; *Laboratory glassware – One-mark pipettes.*

ISO 835 (all parts); *Laboratory glassware – Graduated pipettes.*

ISO 4788, *Laboratory glassware – Graduated measuring cylinders.*

3 Principle

The aqueous simulant test samples are directly analysed by high performance liquid chromatography (HPLC) with ultraviolet (UV) detection. The olive oil test samples are extracted with dilute sodium hydrogen carbonate and the resultant aqueous solution is acidified and analysed by HPLC.

4 Reagents

WARNING: All chemicals are hazardous to health to a greater or lesser extent. It is beyond the scope of this standard to give instructions for the safe handling of all chemicals, that meet, in full, the legal obligations in all countries in which this standard can be followed. Therefore, specific warnings are not given and users of this standard shall ensure that they meet all the necessary safety requirements in their own country.

NOTE During the analysis, unless otherwise stated, only reagents of recognized analytical grade and only distilled water of equivalent purity should be used.

4.1 Methanol (HPLC grade).

4.2 Sodium acetate, trihydrate.

4.3 Orthophthalic acid.

4.4 Terephthalic acid.

4.5 Orthophosphoric acid 85 % (w/v).

4.6 Water (HPLC grade).

4.7 Sodium hydrogen carbonate solution 0,1 % (w/v).

4.8 Acetic acid, 50 % (v/v) in water.

4.9 Propan-2-ol.

4.10 Heptane.

4.11 pH 3,6 buffer solution.

Dissolve 25,0 g of sodium acetate, trihydrate in 350 ml of water, add 5,0 ml ± 0,1 ml of orthophosphoric acid and adjust to pH (3,6 ± 0,2) with glacial acetic acid (approximately 50 ml). Make up to 500 ml with water.

4.12 Weigh accurately about 0,05 g terephthalic acid and, by continuous stirring, dissolve in about 90 ml of methanol. Make up to 100 ml with methanol in a volumetric flask. Prepare a second terephthalic acid standard stock solution for validation purposes, see 7.3.

NOTE The solution can be warmed to 50 °C to facilitate dissolution of the terephthalic acid, which takes at least 1 h.

4.13 Orthophthalic acid internal standard stock solution.

Weigh about 0,1 g of orthophthalic acid and dissolve in propan-2-ol. Make up to 100 ml with propan-2-ol in a volumetric flask.

4.14 Mobile phase for high performance liquid chromatography.

Using a measuring cylinder add 150 ml of methanol to 150 ml of pH 3,6 buffer and dilute to 1 l with water.

NOTE Degassing the mobile phase can be necessary with some HPLC equipment.

5 Apparatus

5.1 Analytical balance capable of weighing accurately to 0,1 mg.

5.2 pH meter with an accuracy of ± pH 0,1.

5.3 Volumetric flasks, of 25 ml, 50 ml and 100 ml capacity, conforming to the minimum requirements of EN ISO 1042.

5.4 Measuring cylinders, of 5 ml, 25 ml, 50 ml, 100 ml and 250 ml capacity, conforming to the minimum requirements of ISO 4788.

5.5 Pipettes, of 5 ml and 50 ml capacity, conforming to the minimum requirements of ISO 648.

5.6 Graduated pipette, of 2 ml capacity, conforming to the minimum requirements of ISO 835.