SLOVENSKI STANDARD
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Materiali in predmeti v stiku z živili - Snovi v polimernih materialih, katerih koncentracija je omejena - 12. del: Določevanje 1,3-benzendimetanamina v modelnih raztopinah za živila

Material and articles in contact with foodstuffs - Plastics substances subject to limitation - Part 12: Determination of 1,3-benzenedimethanamine in food simulants

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Substanzen in Kunststoffen, die Beschränkungen unterliegen - Teil 12: Bestimmung von 1,3-Benzendimethanamin in Prüflebensmitteln

Matériaux et objets en contact avec les denrées alimentaires - Substances dans les matieres plastiques soumises a des limitations - Partie 12 : Détermination du 1,3-benzénediméthanamine dans les simulants d'aliments

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Material and articles in contact with foodstuffs - Plastics substances subject to limitation - Part 12: Determination of 1,3-benzenedimethanamine in food simulants

Matériaux et objets en contact avec les denrées alimentaires - Substances dans les matières plastiques soumises à des limitations - Partie 12 : Détermination du 1,3-benzénediméthanamine 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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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: rue de Stassart, 36 B-1050 Brussels

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Contents

page

Foreword 3
Introduction 5
1 Scope 6
2 Normative references 6
3 Principle 6
4 Reagents 6
5 Apparatus 9
6 Samples 9
7 Procedure 10
8 Expression of results 12
9 Confirmation 13
10 Test report 14
Bibliography 15

Foreword

This document (CEN/TS 13130-12:2005) 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

1,3-benzenedimethanamine (also known as 3-(aminomethyl)-benzylamine or m-xylylene diamine (m-XDA)), $C_8H_{12}N_2$, PM/Ref-No. 13000, hereinafter referred to as m-XDA, is a monomer used in the manufacture of certain plastics materials and articles intended to come into contact with foodstuffs. After manufacture, residual m-XDA monomer can remain in the polymer and may migrate into foodstuffs coming into contact with the plastics material or article.

NOTE However the following should be taken into account when carrying out a migration test: Stability tests in spiked olive oil food simulant carried out under test conditions 10 d at 40 °C indicated that m-XDA is not stable in the fat simulant, presumably due to chemical reaction with the olive oil and/or oxygen. Typically a recovery of only 25 % was obtained under these test conditions. 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, see also the NOTE in Clause 1.

The method has been tested by a second laboratory.

1 Scope

This document, part of EN 13130, specifies a procedure for the determination of 1,3-benzenedimethanamine (also known as 3-(aminomethyl)-benzylamine or m-xylylene diamine (m-XDA)) monomer, hereinafter referred to as m-XDA, in the food simulants water, 3 % w/v aqueous acetic acid, 15 % v/v aqueous ethanol and olive oil. The level of m-XDA monomer determined is expressed as milligrams of m-XDA per kilogram of food simulant in the above mentioned food simulants.

NOTE In addition to the instability of m-XDA observed in olive oil, the test laboratory could not detect any m-XDA peak from the olive oil work-up procedure. Based on some control checks, the assumption of the test laboratory was that the extraction from the olive oil does not work. Therefore, it is recommended that the food simulant 95 % ethanol or iso-octane be applied as substitute fatty simulants instead of olive oil. It is believed that 95 % ethanol and iso-octane can be treated in the same way as 15 % v/v aqueous ethanol and olive oil simulant, respectively. Corresponding investigations were not possible within the project.

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

m-XDA in aqueous simulant test samples is determined, following bis-derivatization with fluorescamine, by high performance liquid chromatography (HPLC) with fluorescence detection. m-XDA in olive oil test samples is extracted with 3 % w/v aqueous acetic acid and determined in the same way. Quantification is relative to external standards. The identity of m-XDA is confirmed by mono-derivatization with less fluorescamine leading to a second peak in the HPLC chromatograms obtained from samples, and by comparison with authentic samples.

4 Reagents

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

4.1 Analyte

1,3-benzenedimethanamine (also known as 3-(aminomethyl)-benzylamine or m-xylylene diamine (m-XDA), $C_8H_{12}N_2$, purity 99 %.

4.2 Other chemicals

4.2.1 Distilled water (HPLC grade), saturated with nitrogen (4.2.10).

4.2.2 Methanol (HPLC grade).

4.2.3 Tetrahydrofuran (HPLC grade).

4.2.4 Acetone

4.2.5 Heptane

4.2.6 Fluorescamine, $C_{17}H_{10}O_4$, purity 98 %.

4.2.7 Disodium tetraborate 10-hydrate, $Na_2B_2O_7 \cdot 10H_2O$.

4.2.8 Sodium hydroxide

4.2.9 Acetic acid glacial 100 %

4.2.10 Purified nitrogen, 99,9999 %.

4.3 Solutions

4.3.1 3 % w/v acetic acid in distilled water

Weigh 15,0 g of acetic acid (4.2.9) with an accuracy of 0,1 g into a 500 ml volumetric flask and make up to the mark with distilled water (4.2.1).

4.3.2 90 % (v/v)Tetrahydrofuran

Pour 90 ml of tetrahydrofuran (4.2.3) into a 100 ml volumetric flask and make up to the mark with water (4.2.1).

4.3.3 Fluorescamine solution, 2 mg/ml

Weigh 10 mg of fluorescamine (4.2.6) into a 5 ml volumetric flask and make up to the mark with acetone (4.2.4).

NOTE The solution can be stored for up to 1 week in the dark at 5 ºC.

4.3.4 5 M sodium hydroxide, NaOH

Weigh 20,0 g of sodium hydroxide (4.2.8) into a 100 ml volumetric flask and make up to the mark with water (4.2.1).

4.3.5 0,15 M borate buffer (pH = 9,2)

Weigh 14,3 g of disodium tetraborate 10-hydrate (4.2.7) into a 250 ml volumetric flask and make up to the mark with water (4.2.1).

Any precipitate caused by a fall in temperature shall be totally re-dissolved before the solution is used.