



SLOVENSKI STANDARD SIST-TS CEN/TS 13130-18:2005

01-april-2005

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Materials and articles in contact with foodstuffs - Plastics substances subject to limitation - Part 18: Determination of 1,2-dihydroxybenzene, 1,3-dihydroxybenzene, 1,4-dihydroxybenzene, 4,4'-dihydroxybenzophenone and 4,4'-dihydroxybiphenyl in food simulants

SIST-TS CEN/TS 13130-18:2005

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Substanzen in Kunststoffen, die Beschränkungen unterliegen - Teil 18: Bestimmung von 1,2-Dihydroxybenzol, 1,3-Dihydroxybenzol, 1,4-Dihydroxybenzol, 4,4'-Dihydroxybenzophenon und 4,4'-Dihydroxybiphenyl in Prüflebensmitteln

Matériaux et objets en contact avec les denrées alimentaires - Substances dans les matières plastiques soumises à des limitations - Partie 18 : Détermination des 1,2-dihydroxybenzene, 1,3-dihydroxybenzene, 1,4-dihydroxybenzene, 4,4'-dihydroxybenzophénone et 4,4'-dihydroxybiphénil dans les simulants d'aliments

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67.250 Materiali in predmeti v stiku z Materials and articles in
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Materials and articles in contact with foodstuffs - Plastics substances subject to limitation - Part 18: Determination of 1,2-dihydroxybenzene, 1,3-dihydroxybenzene, 1,4-dihydroxybenzene, 4,4'-dihydroxybenzophenone and 4,4'-dihydroxybiphenyl in food simulants

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Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Substanzen in Kunststoffen, die Beschränkungen unterliegen - Teil 18: Bestimmung von 1,2-Dihydroxybenzol, 1,3-Dihydroxybenzol, 1,4-Dihydroxybenzol, 4,4'-Dihydroxy-benzophenon und 4,4'-Dihydroxybiphenyl in Prüflebensmitteln

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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.

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.



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Foreword

This document (CEN/TS 13130-18:2005) 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.

[SIST-TS CEN/TS 13130-18:2005](#)

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*

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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

1,2-dihydroxybenzene (1,2-DHB), (pyro)catechol, $C_6H_4(OH)_4$, PM/Ref. No 15880, 1,3-dihydroxybenzene (1,3-DHB), resorcinol, $C_6H_4(OH)_2$, PM/Ref. No 15910, 1,4-dihydroxybenzene (1,4-DHB), hydroquinone, $C_6H_4(OH)_2$, PM/Ref-No. 15940, 4,4'-dihydroxybenzophenone (DHBphen), $HOC_6H_4COC_6H_4OH$, PM/Ref. No 15970 and 4,4'-dihydroxybiphenyl (DHBiph), $HOC_6H_4C_6H_4OH$, PM/Ref. No 16000, are monomers used in the manufacture of certain plastics materials and articles intended to come into contact with foodstuffs. After manufacture, residual monomer(s) can remain in the polymer and may migrate into foodstuffs coming into contact with the plastics material or article.

Method A describes the determination of 1,2-DHB, 1,3-DHB, 1,4-DHB in food simulants.

Method B describes the determination of DHBphen and DHBiph in food simulants. However, in cases where migration occurs, pay attention to the clear identification of the analyte, since under the conditions described in the method DHBphen and DHBiph have the same retention time.

NOTE The following should be taken into account when carrying out a migration test (see EN 13130-1). From migration experiments carried out for 10 d at 40 °C it was recognized that 1,2-DHB, 1,3-DHB and 1,4-DHB are subject to losses due to chemical decomposition. The loss observed for 1,2-DHB was up to 20 % in aqueous simulants, that for 1,3-DHB was 5 % to 12 % in olive oil and that for 1,4-DHB was 14 % to 68 %, in both aqueous food simulants and olive oil.

For DHBiph a small loss of 5 % to 12 % due to chemical decomposition was observed when using 15 % v/v aqueous ethanol as food simulant.

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

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CEN/TS 13130-18:2005 (E)**1 Scope**

This document, part of EN 13130, specifies two methods as follows.

Method A is an analytical procedure for the determination of the monomers 1,2-DHB, 1,3-DHB and 1,4-DHB in the food simulants water, 3 % w/v aqueous acetic acid, 15 % v/v aqueous ethanol and olive oil. The levels of monomer determined are expressed in milligrams of monomer per kilogram of food simulant. The method is appropriate for the quantitative determination of 1,2-DHB and 1,3-DHB at a minimum level of 0,15 mg/kg in food simulants and, of 1,4-DHB at a minimum level of 0,10 mg/kg in food simulants.

Method B is an analytical procedure for the determination of DHBphen and DHBiph in the food simulants water, 3 % w/v aqueous acetic acid, 15 % v/v aqueous ethanol and olive oil. The method is appropriate for the quantitative determination of DHBphen or DHBiph at minimum levels of 0,2 mg/kg and 0,25 mg/kg, respectively, in food simulants.

NOTE The methods 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.

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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 1,2-dihydroxybenzene, 1,3-dihydroxybenzene and/or 1,4-dihydroxybenzene in food simulants

3.1 Principle

The aqueous simulant test samples are analyzed directly by high performance liquid chromatography (HPLC) with ultra violet (UV) detection. The olive oil test samples are extracted with distilled water and the resultant aqueous solution analyzed by HPLC. Quantification is achieved using 2-methyl-1,3-dihydroxybenzene as an internal standard with calibration against relevant food simulant samples fortified with known amounts of 1,2-DHB, 1,3-DHB or 1,4-DHB. Confirmation of 1,2-DHB, 1,3-DHB or 1,4-DHB is carried out by diode array detection.

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3.2 Reagents

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

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3.2.1 Analytes <https://standards.iteh.ai/catalog/standards/sist/b6dbe2fc-a10a-4e02-8612-b03c9facfa86/sist-ts-cen-ts-13130-18-2005>

3.2.1.1 1,2-dihydroxybenzene (1,2-DHB), C₆H₄(OH)₂, purity greater than 99 %.

3.2.1.2 1,3-dihydroxybenzene (1,3-DHB), C₆H₄(OH)₂, purity greater than 99 %.

3.2.1.3 1,4-dihydroxybenzene (1,4-DHB), C₆H₄(OH)₂, purity greater than 99 %.

3.2.1.4 2-methyl-1,3-dihydroxybenzene (2-methylresorcinol) (2-MR), CH₃C₆H₃(OH)₂, internal standard, purity greater than 98 %.

3.2.2 Chemicals

3.2.2.1 Hexane, glass distilled grade.

3.2.2.2 Methanol, glass distilled grade.

3.2.2.3 Water, glass distilled grade.

3.2.2.4 Acetonitrile, HPLC grade.

3.2.2.5 Water, distilled, HPLC grade.

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3.2.2.6 Sodium dihydrogen orthophosphate, $\text{NaH}_2\text{PO}_4 \cdot 2\text{H}_2\text{O}$.

3.2.2.7 Glacial acetic acid.

3.2.2.8 Mobile phase for high performance liquid chromatography, prepared as follows.

Dissolve 7,5 g of sodium dihydrogen orthophosphate (3.2.2.6) in 800 ml of water (3.2.2.5), add 150 ml acetonitrile (3.2.2.4) using a measuring cylinder and adjust to pH ($3,6 \pm 0,2$) with glacial acetic acid (3.2.2.7) (approximately 5 ml). Make up to 1 l with water (3.2.2.5).

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

3.2.3 Solutions

NOTE The solutions prepared in 3.2.3.1 to 3.2.3.7 can be stored in the dark at $-20\text{ }^\circ\text{C}$ for up to 6 weeks.

3.2.3.1 Standard stock solution 1,2-DHB and/or 1,4-DHB in methanol at a defined concentration of approximately 0,5 g/l

Weigh $50\text{ mg} \pm 2\text{ mg}$ of 1,2-DHB, or 1,4-DHB into a 100 ml volumetric flask to an accuracy of 0,2 mg and dilute to the mark with methanol (3.2.2.2). Close and mix thoroughly.

Calculate the exact concentration of 1,2-DHB and/or 1,4-DHB in milligrams per litre.

Repeat the procedure to provide a second standard stock solution.

3.2.3.2 Standard stock solution 1,3-DHB in methanol at a defined concentration of approximately 0,125 g/l

Weigh $12,5\text{ mg} \pm 2\text{ mg}$ of 1,3-DHB into a 100 ml volumetric flask to an accuracy of 0,2 mg and dilute to the mark with methanol (3.2.2.2). Close and mix thoroughly.

Calculate the exact concentration of 1,3-DHB in milligrams per litre.

Repeat the procedure to provide a second standard stock solution.

3.2.3.3 Stock internal standard solution of 2-MR in methanol at a defined concentration of approximately 1 g/l

Weigh $100\text{ mg} \pm 2\text{ mg}$ of 2-MR into a 100 ml volumetric flask to an accuracy of 0,2 mg and dilute to the mark with methanol (3.2.2.2). Close and mix thoroughly.

Calculate the exact concentration of 2-MR in milligrams per litre.

3.2.3.4 Intermediate standard solutions of 1,2-DHB and 2-MR in methanol

Into six 25 ml volumetric flasks, pipette 0 ml, 2,0 ml, 4,0 ml, 8,0 ml, 16,0 ml and 20,0 ml of the 1,2-DHB standard stock solution (3.2.3.1). Add 5,0 ml of the 2-MR stock internal standard solution (3.2.3.3) and dilute to the mark with methanol. The resulting intermediate standards contain approximately 0 mg/l, 40 mg/l, 80 mg/l, 160 mg/l, 320 mg/l and 400 mg/l 1,2-DHB and 200 mg/l 2-MR.

Calculate the exact concentrations of 1,2-DHB and 2-MR in milligrams per litre.

Repeat the procedure using the second solution prepared in 3.2.3.1 to provide a second set of intermediate standards.

3.2.3.5 Intermediate standard solutions of 1,3-DHB and 2-MR in methanol

Into six 25 ml volumetric flasks, pipette 0 ml, 2,0 ml, 4,0 ml, 8,0 ml, 16,0 ml and 20,0 ml of the 1,3-DHB standard stock solution (3.2.3.2). Add 5,0 ml of the 2-MR stock internal standard solution (3.2.3.3) and dilute to the mark with methanol. The resulting intermediate standards contain approximately 0 mg/l, 10 mg/l, 20 mg/l, 40 mg/l, 80 mg/l and 100 mg/l 1,3-DHB and 200 mg/l 2-MR.

Calculate the exact concentrations of 1,3-DHB and 2-MR in milligrams per litre.

Repeat the procedure using the second solution prepared in 3.2.3.2 to provide a second set of intermediate standards.

3.2.3.6 Intermediate standard solutions of 1,4-DHB and 2-MR in methanol

Into a 100 ml volumetric flask, pipette 10 ml of the 1,4-DHB standard stock solution (3.2.3.1) and dilute to the mark with methanol thus obtaining a diluted stock solution of 1,4-DHB of approximately 50 mg/l.

Into six 25 ml volumetric flasks, pipette 0 ml, 1,5 ml, 5,0 ml, 7,5 ml, 10,0 ml and 15,0 ml of this diluted 1,4-DHB stock solution. Add 5,0 ml of the 2-MR stock internal standard solution (3.2.3.3) and dilute to the mark with methanol. These standards contain approximately 0 mg/l, 3 mg/l, 10 mg/l, 15 mg/l, 20 mg/l and 30 mg/l 1,4-DHB and 200 mg/l 2-MR.

Calculate the exact concentrations of 1,4-DHB and 2-MR in milligrams per litre of solution.

Repeat the procedure using the second stock solution prepared in 3.2.3.1 to provide a second set of intermediate standards.

3.2.3.7 Intermediate internal standard solution

Pipette 5 ml of the 2-MR stock internal standard solution (3.2.3.3) into a 25 ml volumetric flask and dilute to the mark with methanol. This solution contains approximately 200 mg/l 2-MR.

Calculate the exact concentration of 2-MR in milligrams per litre of solution. The calculated concentration shall be the same as in 3.2.3.4, 3.2.3.5 and 3.2.3.6.

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 High performance liquid chromatograph, with a 20 μ l injection loop, and a variable wavelength UV detector, set to 280 nm, connected to a strip chart recorder or integrator.

3.3.2 HPLC column, capable of fully resolving 1,2-DHB, 1,3 DHB and/or 1,4-DHB from 2-MR, such that the peaks of 1,2-DHB, 1,3-DHB and/or 1,4-DHB and 2-MR do not overlap by more than 1 % peak area with each other and with interferences arising from the injection media.

Appropriate operating conditions shall be established for the specific equipment used for the determination.

NOTE The following column has been found to be suitable:

Column	250 mm x 4,6 mm I.D. octadecylsilane, 10 % carbon loading with Hypersil 5 μ m packing
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