



# SLOVENSKI STANDARD SIST EN 13130-7:2004

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Materials and articles in contact with foodstuffs - Plastics substances subject to limitation - Part 7: Determination of monoethylene glycol and diethylene glycol in food simulants

**iTeh STANDARD PREVIEW**

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Substanzen in Kunststoffen, die Beschränkungen unterliegen - Teil 7: Bestimmung von Monoethylenglycol und Diethylenglycol in Prüflebensmitteln

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Matériaux et objets en contact avec les denrées alimentaires - Substances dans les matières plastiques soumises a des limitations - Partie 7 : Détermination du monoéthylène glycol et du diéthylène glycol dans les simulants d'aliments

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EUROPEAN STANDARD  
NORME EUROPÉENNE  
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**EN 13130-7**

May 2004

ICS 67.250

English version

## Materials and articles in contact with foodstuffs - Plastics substances subject to limitation - Part 7: Determination of monoethylene glycol and diethylene glycol in food simulants

Matériaux et objets en contact avec les denrées alimentaires - Substances dans les matières plastiques soumises à des limitations - Partie 7 : Détermination du monoéthylène glycol et du diéthylène glycol dans les simulants d'aliments

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Substanzen in Kunststoffen, die Beschränkungen unterliegen - Teil 7: Bestimmung von Monoethylenglykol und Diethylenglykol in Lebensmittel-Simulantien

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

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

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## Foreword

This document (EN 13130-7:2004) 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 13130-7 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 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 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*

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- 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-trimethylpropane 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

Monoethylene glycol (MEG) and diethylene glycol (DEG) are monomers used in the manufacture of plastics materials and articles intended to come into contact with foodstuffs. Residues of monoethylene glycol and diethylene glycol can remain in the plastic after processing to form materials and articles for food contact use, and can migrate into foodstuffs.

NOTE Commission Directive 2002/72/EC lists a combined specific migration limit of 30 mg/kg (T) of monoethylene glycol and diethylene glycol in foods or food simulants.

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**EN 13130-7:2004 (E)****1 Scope**

This part of this European Standard specifies methods for the determination of monoethylene glycol and diethylene glycol in the food simulants; water, 3 % (w/v) acetic acid, 15 % (v/v) ethanol 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 monoethylene glycol and diethylene glycol in food simulants separately, or combined, at the specific migration limit SML (T) of 30 mg/kg.

**NOTE** This method was developed for the determination of monoethylene glycol and diethylene glycol 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 835 (all parts); *Laboratory glassware – Graduated pipettes.*

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

**3 Principle**

After addition of an internal standard the aqueous food simulants or the olive oil water extract is directly injected for gas chromatographic analysis using a cold on-column injector. Detection limits are approximately 1 mg/kg in the food simulants. The concentrations of monoethylene glycol and diethylene glycol are measured by comparison of peak height or area ratios against standards.

**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 may 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.

4.1 Methanol

4.2 Monoethylene glycol



- 4.3 Diethylene glycol
- 4.4 Butan-1,4-diol, internal standard
- 4.5 Water (HPLC or deionized)
- 4.6 Heptane
- 4.7 Prepare a monoethylene glycol and diethylene glycol standard stock solution as follows:

Weigh accurately about 0,75 g each of monoethylene glycol and diethylene glycol into a beaker, dissolve in methanol and transfer with washings to a 100 ml volumetric flask. Dilute to the mark with methanol. This solution is stable for 1 month if stored in the dark at 5 °C in a stoppered flask. Prepare a second stock solution for validation purposes, see 7.2.2.

- 4.8 Prepare a butan-1,4-diol internal standard stock solution as follows:

Weigh about 1 g of butan-1,4-diol into a beaker, dissolve in methanol and make up to 100 ml mark with methanol in a volumetric flask. This solution is stable for 1 month if stored in the dark at 5 °C in a stoppered flask.

## 5 Apparatus

- 5.1 Analytical balance capable of weighing to 0,1 mg.
- 5.2 Graduated pipettes, conforming to the minimum requirements of ISO 835 and of 1 ml and 2 ml capacity.
- 5.3 Burette, conforming to the minimum requirements of ISO 385 and of 25 ml capacity.
- 5.4 Volumetric flasks, conforming to the minimum requirements of EN ISO 1042 and of 25 ml, 50 ml and 100 ml capacity.
- 5.5 Separating funnels with polytetrafluoroethylene stopcock, of 250 ml capacity.
- 5.6 Glass syringe, of 50 ml capacity.
- 5.7 0,2 µm disposable HPLC filters, or disposable C18 solid phase extraction cartridges 400 mg size.
- 5.8 Measuring cylinders, conforming to the minimum requirements of ISO 4788 and of 25 ml and 50 ml capacity.
- 5.9 Gas chromatograph (GC) fitted with a flame ionization detector (FID) and a cold on-column injector.

NOTE 1 This method has been developed for use with cold on-column injectors, which are available from all major GC suppliers. Previous studies at other laboratories have shown that split/splitless injection gives unreliable results. Higher than optimum carrier gas flow rates have been found to give less peak tailing and increased column lifetime for polyethylene glycol stationary phases. The GC column should be capable of resolving monoethylene glycol, diethylene glycol and butan-1,4-diol from each other, and from ethanol and acetic acid present in the simulants. GC capillary columns using a polyethylene glycol stationary phase have been found to be most suitable for example:

- a) 15 m X 0,53 mm internal diameter, film thickness 1 µm;
- Temperature                    100 °C hold 2 min ramped to 150 °C at 10 °C/min, hold 4 min;
- Carrier gas                      Helium 50 KPa, 18 ml/min;