

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

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

EESTI STANDARDI EESSÕNA

NATIONAL FOREWORD

<p>Käesolev Eesti standard EVS-EN 13130-7:2004 sisaldab Euroopa standardi EN 13130-7:2004 ingliskeelset teksti.</p> <p>Käesolev dokument on jõustatud 23.09.2004 ja selle kohta on avaldatud teade Eesti standardiorganisatsiooni ametlikus väljaandes.</p> <p>Standard on kättesaadav Eesti standardiorganisatsioonist.</p>	<p>This Estonian standard EVS-EN 13130-7:2004 consists of the English text of the European standard EN 13130-7:2004.</p> <p>This document is endorsed on 23.09.2004 with the notification being published in the official publication of the Estonian national standardisation organisation.</p> <p>The standard is available from Estonian standardisation organisation.</p>
--	---

<p>Käsitlusala: 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.</p>	<p>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.</p>
---	---

ICS 67.250

Võtmesõnad:

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

Contents

	page
Foreword.....	3
1 Scope	6
2 Normative references	6
3 Principle.....	6
4 Reagents.....	6
5 Apparatus	7
6 Samples	8
7 Procedure	8
7.1 Preparation of standards	8
7.1.1 Intermediate standards	8
7.1.2 Internal standard, 800 mg/l butan-1,4-diol	8
7.1.3 Working standards for aqueous simulants.....	8
7.1.4 Working standards for olive oil.....	9
7.2 Preparation of calibration graphs	9
7.2.1 Injection of standards	9
7.2.2 Validation of stock monoethylene glycol/diethylene glycol solution	9
7.3 Extraction of olive oil simulant migration test samples	9
7.4 Preparation of aqueous simulant migration test samples	10
7.5 Injection of samples	10
8 Expression of results	10
8.1 Method of calculation.....	10
8.1.1 As milligrams of monoethylene glycol and diethylene glycol per kilogram of food simulant	10
8.1.2 As milligrams of monoethylene glycol and diethylene glycol per square decimetre of surface area of plastic material or article.....	10
8.1.3 Calculation of the specific migration.....	10
8.2 Precision.....	10
8.2.1 Validation.....	10
8.2.2 Repeatability and reproducibility	11
8.2.3 Detection limit.....	12
9 Confirmation of monoethylene glycol and diethylene glycol	12
9.1 Requirement for confirmation	12
9.2 Confirmation by re-analysis using confirmed by gas chromatography - mass spectrometry	12
10 Test report	12
Annex A (informative)	14
Annex B (informative)	15
Bibliography	16

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*

- 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.

This document is a preview generated by EVS

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