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TECHNICAL SPECIFICATION  
SPÉCIFICATION TECHNIQUE  
TECHNISCHE SPEZIFIKATION

**CEN/TS 13130-28**

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English version

**Materials and articles in contact with foodstuffs - Plastics  
substances subject to limitation - Part 28: Determination of  
1,1,1-trimethylolpropane in food simulants**

Matériaux et objets en contact avec les denrées  
alimentaires - Substances dans les matières plastiques  
soumises à des limitations - Partie 28 : Détermination du  
1,1,1,-triméthylolpropane dans les simulants d'aliments

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln  
- Substanzen in Kunststoffen, die Beschränkungen  
unterliegen - Teil 28: Bestimmung von 1,1,1-  
Trimethylolpropan in Prüflebensmitteln

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

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## Foreword

This document (CEN/TS 13130-28:2005) 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.

Informative Annex A details the relationship of this standard with the European Union Directives.

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 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].

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*

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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

1,1,1-trimethylolpropane (TMP),  $C_6H_{14}O_3$ , PM/Ref. No 25600, is a monomer used in the manufacture of certain plastics materials and articles intended to come into contact with foodstuffs. After manufacture residual 1,1,1-trimethylolpropane can remain in the finished product and may migrate into foodstuffs coming into contact with that product.

NOTE 1 The analytical method described in this part of EN 13130 is the result of a study on the determination of the migration of 1,1,1-trimethylolpropane from plastics materials into food simulants. In the course of the study several problems were encountered and solutions for these problems were incorporated in the final method. The most suitable method is described in this part of EN 13130. The method was successfully pre-validated by the developing laboratory tests, using the four official EU food simulants, to establish the precision data at the restriction criterion. Also migration tests were performed with samples containing 1,1,1-trimethylolpropane as monomer in contact for 10 d at 40 °C with 15 % v/v aqueous ethanol and olive oil.

NOTE 2 During the testing of the method by a second laboratory it appeared that the testing laboratory was not able to reproduce the precision data as obtained by the developing laboratory. Based on the problems encountered by the testing laboratory the method description was modified to make critical points more clear. Within the scope of the project "Development of Methods of Analysis for Monomers" it was not possible to re-test the method and therefore the method described should be considered as a useful analytical method, with limited validation data. Further testing is required to demonstrate that the method can be applied with the required accuracy.

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The method has been pre-validated in a validation experiment only by one laboratory, the developing laboratory.

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**CEN/TS 13130-28:2005 (E)****1 Scope**

This document, part of EN 13130, specifies an analytical procedure for the determination of 1,1,1-trimethylolpropane in food simulants distilled water, 3 % (w/v) acetic acid aqueous solution, 15 % (v/v) ethanol aqueous solution and rectified olive oil. The method is appropriate for the quantitative determination of 1,1,1-trimethylolpropane in approximate analyte concentration range of 0,6 mg/kg to 12 mg/kg food simulant.

NOTE The method 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.

**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**

Aqueous food simulants are saturated with potassium carbonate and extracted with ethanol/ethyl acetate. After evaporation of the solvent, the extracted TMP is silylated with trimethylsilylimidazole in pyridine. The amount of 1,1,1-trimethylolpropane silyl ethers are determined by gas chromatography with flame ionization detection. 1,4-butanediol is used as an internal standard. Fat simulant is extracted with water prior to derivatization.

Confirmation of the identity of 1,1,1-trimethylolpropane is established by combined gas chromatography/mass spectrometry.

**4 Reagents**

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



## 4.1 Analytes

**4.1.1 1,1,1-trimethylolpropane (TMP),  $C_6H_{14}O_3$  or  $CH_3CH_2C(CH_2OH)_3$** , molecular weight 134,18; boiling point: 297 °C; solubility: W, al; purity: > 97 % (GC).

**4.1.2 Internal standard: 1,4-butandiol (BUG) or tetramethyleneglycol,  $C_4H_{10}O_2$  or  $HOCH_2CH_2CH_2CH_2OH$** , molecular weight: 90,12; boiling point: 235,12 °C; density: 1,0171 g/ml; solubility: W, al; purity: > 98 % (GC).

## 4.2 Chemicals

**4.2.1 Water**, deionized or HPLC grade.

**4.2.2 Acetic acid**, 100 %.

**4.2.3 Ethanol**, absolute.

**4.2.4 n-Pentane**, 95 %.

**4.2.5 Ethyl acetate**, 99,5 %.

**4.2.6 Derivatizing reagent, trimethylsilylimidazole**, TMSI in dry pyridine, in 1 ml ampoules.

**4.2.7 Potassium carbonate**, 99,9 %.

**4.2.8 Cation exchange resin, strong acidic, sulfonated polystyrene resins with spherical particles**, capacity 1,7 meq/ml, size 50 mesh to 100 mesh).

The cation exchange resin shall be washed with methanol before use.

**4.2.9 Sodium hydroxide**, 99 %.

## 4.3 Solutions

### 4.3.1 Stock solution of 1,1,1-trimethylolpropane in ethanol (0,75 g/l)

Weigh to the nearest 0,2 mg approximately 75 mg TMP into a 100 ml volumetric flask and fill to the mark with ethanol (4.2.3).

Calculate the actual concentration in milligrams of TMP per millilitre of solution.

Repeat the procedure to obtain a second standard stock solution.

NOTE The stock solutions can be stored in a well closed container in the dark for a maximum period of three months at any temperature between - 20 °C and + 20 °C.

### 4.3.2 Internal standard stock solution of 1,4-butandiol (BUG) in ethanol (0,75 g/l)

Weigh to the nearest 0,2 mg approximately 75 mg BUG into a 100 ml volumetric flask and fill to the mark with ethanol (4.2.3).

Calculate the actual concentration in milligrams of BUG per millilitre of solution.