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SIST-TS CEN/TS 13130-22:2005

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TECHNICAL SPECIFICATION  
SPÉCIFICATION TECHNIQUE  
TECHNISCHE SPEZIFIKATION

**CEN/TS 13130-22**

February 2005

ICS 67.250

English version

**Materials and articles in contact with foodstuffs - Plastics  
substances subject to limitation - Part 22: Determination of  
ethylene oxide and propylene oxide in plastics**

Matériaux et objets en contact avec les denrées  
alimentaires - Substances dans les matières plastiques  
soumises à des limitations - Partie 22 : Détermination des  
oxydes d'éthylène et de propylène dans les matières  
plastiques

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln  
- Substanzen in Kunststoffen, die Beschränkungen  
unterliegen - Teil 22: Bestimmung von Ethylenoxid und  
Propylenoxid in Kunststoffen

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 13130-22:2005) 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*

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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

Ethylene oxide, C<sub>2</sub>H<sub>4</sub>O, PM/Ref. No 17020, and propylene oxide, C<sub>3</sub>H<sub>6</sub>O, PM/Ref. No 24010 are monomers used in the manufacture of certain plastics materials and articles intended to come into contact with foodstuffs. After manufacture residual ethylene oxide can remain in the finished product and may migrate into foodstuffs coming into contact with that product.

The method has been pre-validated by collaborative trial with two laboratories.

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**CEN/TS 13130-22:2005 (E)****1 Scope**

This document, part of EN 13130, specifies an analytical procedure for the determination of ethylene oxide (EO) or propylene oxide (PO) in polymers.

The method is appropriate for the quantitative determination of ethylene oxide or propylene oxide in an analyte concentration range of approximately 0,2 mg/kg to 2 mg/kg of polymer.

**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****iTeh STANDARD PREVIEW**

The level of ethylene oxide and propylene oxide in polymers is determined by headspace gas chromatography. Quantification is achieved using diethyl ether as an internal standard with calibration against polymer samples fortified with ethylene oxide or propylene oxide according to the standard addition procedure.

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Confirmation of ethylene oxide is carried out using another column with different polarity. Confirmation of propylene oxide is carried out by combined gas chromatography/mass spectrometry (GC/MS).



## 4 Reagents

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

### 4.1 Analytes

**4.1.1 Ethylene oxide**,  $C_2H_4O$  of purity greater than 99 % (w/w).

**4.1.2 Propylene oxide**,  $C_3H_6O$  of purity greater than 99 % (w/w).

**4.1.3 Diethyl ether**,  $C_4H_{10}O$  containing no impurity greater than 1 % by peak area or peak height, which elutes at the same retention time as ethylene oxide or propylene oxide.

### 4.2 Chemical

**N,N-dimethylacetamide**, distilled,  $C_4H_9NO$  free of any interferences (< 1 % area) with the ethylene oxide or propylene oxide and diethyl ether peak.

### 4.3 Solutions

#### 4.3.1 Stock solutions of ethylene oxide or propylene oxide in dimethylacetamide

##### 4.3.1.1 Stock solutions of ethylene oxide in dimethylacetamide (2 mg/ml to 6 mg/ml)

Add 50,0 ml dimethylacetamide (4.2) into a 50 ml sample vial and determine the mass including septa and cap to within 0,1 mg. Introduce 0,1g to 0,3 g ethylene oxide (4.1.1) by bubbling the ethylene oxide gas (4.1.1) through the dimethylacetamide. Weigh the vial to an accuracy of 0,1 mg. Calculate the actual concentration in milligrams of ethylene oxide per millilitre of solution.

Repeat the procedure to provide a second stock solution.

NOTE The stock solution can be stored at  $-20\text{ }^{\circ}\text{C}$  for up to 3 months protected from light in septum capped glass vials with minimum headspace or at  $+4\text{ }^{\circ}\text{C}$  for up to 8 weeks.

Start by comparing the two stock solutions by making just one dilution of each stock solution. If the response factor, calculated from the detector response, and the concentration of the solution agree to within 5 % of one another, further diluted standards shall be made from only one of the stock solutions.

##### 4.3.1.2 Stock solution of propylene oxide in dimethylacetamide (1 mg/ml)

Weigh to the nearest 0,1 mg approximately 50 mg of propylene oxide (4.1.2) into a 50 ml volumetric flask, which contains approximately 30 ml of dimethylacetamide (4.2). Fill the flask to the mark with dimethylacetamide and mix carefully.

Calculate the actual concentration in milligrams of propylene oxide per millilitre of solution.

Repeat the procedure to provide a second stock solution.

NOTE The stock solution can be stored at  $-20\text{ }^{\circ}\text{C}$  for up to 3 months protected from light in septum capped glass vials with minimum headspace or at  $+4\text{ }^{\circ}\text{C}$  for up to 8 weeks.

Start by comparing the two stock solutions by making just one dilution of each stock solution. If the response factor, calculated from the detector response, and the concentration of the solution agree to within 5 % of one another, further diluted standards shall be made from only one of the stock solutions.