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SIST EN 13130-4:2004

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EUROPEAN STANDARD  
NORME EUROPÉENNE  
EUROPÄISCHE NORM

**EN 13130-4**

May 2004

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English version

**Materials and articles in contact with foodstuffs - Plastics  
substances subject to limitation - Part 4: Determination of 1,3-  
butadiene in plastics**

Matériaux et objets en contact avec les denrées  
alimentaires - Substances dans les matières plastiques  
soumises à des limitations - Partie 4 : Détermination du  
1,3-butadiène dans les matières plastiques

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln  
- Substanzen in Kunststoffen, die Beschränkungen  
unterliegen - Teil 4: Bestimmung von 1,3-Butadien in  
Kunststoffen

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.



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## Foreword

This document (EN 13130-4:2004) 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of an identical text or by endorsement, at the latest by November 2004, and conflicting national standards shall be withdrawn at the latest by November 2004.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 13130-4 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 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*
- 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

1,3-Butadiene,  $\text{CH}_2=\text{CH}-\text{CH}=\text{CH}_2$ , is a monomer used in the manufacture of certain plastics materials and articles intended to come into contact with foodstuffs. During the manufacture of 1,3-butadiene copolymers, residual 1,3-butadiene monomer can remain in the polymer and can migrate into food coming into contact with the polymer.

The method described in this part of this standard is to be used in conjunction with part 1 of this standard, which describes the procedure required prior to determination of 1,3-butadiene. The method has been validated by a collaborative trial using acrylonitrile-butadiene-styrene copolymer (ABS).

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**EN 13130-4:2004 (E)****1 Scope**

This part of this European Standard specifies a method for the determination of butadiene monomer in polymers.

The method is applicable to acrylonitrile-butadiene-styrene copolymer (ABS) and to high-impact polystyrene (HIPS) as well as to other 1,3-butadiene polymers and copolymers where these are soluble in N,N-dimethylacetamide or finely dispersed, swollen suspensions in N,N-dimethylacetamide. The level of 1,3-butadiene monomer determined is expressed as milligrams of 1,3-butadiene per kilogram of polymer. The method is appropriate for the quantitative determination of 1,3-butadiene at a level of 0,1 mg/kg in the polymer.

**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*

SIST EN 13130-4:2004

**3 Principle**

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The level of 1,3-butadiene in a polymer is determined by headspace gas chromatography of the polymer sample dissolved in N,N-dimethylacetamide, applying automated sample injection and using flame ionization detection (FID). Quantification is achieved using n-pentane as an internal standard with calibration against polymer samples fortified with 1,3-butadiene according to the standard addition procedure.

If interferences are experienced with the internal standard, n-pentane, calibration is carried out by the external standardization as described in annex A.

If automated headspace sampling cannot be performed, manual injection as described in annex B shall be applied.

Confirmation of 1,3-butadiene levels is carried out by combined gas chromatography/mass spectrometry (GC/MS).

**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** 1,3-Butadiene, CH<sub>2</sub>=CH-CH=CH<sub>2</sub>, purity greater than 99,5 % (w/w).



**4.2** n-Pentane,  $\text{CH}_3-(\text{CH}_2)_3-\text{CH}_3$  containing no impurity at  $> 1\%$  by area which will elute at the same GC retention time as 1,3-butadiene.

**4.3** N,N-Dimethylacetamide,  $\text{CH}_3-\text{CO}-\text{N}(\text{CH}_3)_2$ ,  $d_T = 0,9600 - 0,00094 \times T$  where T is the temperature in °C. The N,N-dimethylacetamide shall be free of any interferences ( $< 1\%$ ) which elute at the same retention times as 1,3-butadiene and n-pentane peaks.

**4.4** Prepare stock solutions of 1,3-butadiene in N,N-dimethylacetamide with defined concentrations in the range 5 mg/g to 10 mg/g as follows:

a) Weigh a 50 ml sample vial (5.4) including septum and cap to an accuracy of 0,01 mg. Pipette 50 ml N,N-dimethylacetamide (4.3) into the vial (5.4), close and weigh again to 0,01 mg. Insert hollow needles for introducing 1,3-butadiene and venting air and weigh again to 0,01 mg. Introduce, under a fume hood, 0,3 g to 0,5 g 1,3-butadiene (4.1) by bubbling through the N,N-dimethylacetamide. Re-weigh the vial plus needles to an accuracy of 0,01 mg and remove then the needles. Calculate the concentration in milligrams of 1,3-butadiene per gram of solution.

b) Repeat item a) to provide a second stock solution.

**NOTE** The stock solutions can be stored at  $-20\text{ °C}$  up to eight weeks protected from light in septum capped glass vials with minimum headspace; storage at  $4\text{ °C}$  with the exclusion of light should not exceed one week.

**4.5** Prepare standard solutions of 1,3-butadiene in N,N-dimethylacetamide with defined concentrations in the range of 0,01 mg/ml to 0,5 mg/ml corresponding to  $0,2\text{ }\mu\text{g}/20\text{ }\mu\text{l}$  to  $10\text{ }\mu\text{g}/20\text{ }\mu\text{l}$  (see the note in 6.2.2) as follows:

a) Weigh a sample vial (5.3), including septum and cap, to an accuracy of 0,01 mg. Pipette 15 ml to 20 ml N,N-dimethylacetamide into the vial, close and weigh again to 0,01 mg. Add 0,2 ml to 5 ml of stock solution (4.4), mix thoroughly and re-weigh to an accuracy of 0,01 mg. Calculate the concentration in micrograms of 1,3-butadiene per gram of solution or in micrograms of 1,3-butadiene per  $20\text{ }\mu\text{l}$  solution (calculate using density according to 4.3). To prepare diluted standard solutions repeat step 4.5.

b) Repeat item a) using the second stock solution prepared in 4.4b) to provide a second set of diluted standard solutions.

**4.6** Dilute standard solution of n-pentane (4.2) in N,N-dimethylacetamide (4.3), with a known concentration of approximately 0,5 mg/ml to 2 mg/ml corresponding to  $10\text{ }\mu\text{g}/20\text{ }\mu\text{l}$  to  $40\text{ }\mu\text{g}/20\text{ }\mu\text{l}$ , prepared by following an analogous procedure to that described in 4.4 and 4.5.

**4.7** Nitrogen, purified, 99,9999 %.

## 5 Apparatus

**NOTE** An instrument or item of apparatus is listed only where it is special, or made to a particular specification, usual laboratory equipment being assumed to be available.

**5.1** Gas chromatograph, equipped with a flame ionization detector and fitted with an automatic headspace sampler.

**5.2** Gas chromatographic column, capable of the separation of N,N-dimethylacetamide from 1,3-butadiene and n-pentane such that the peaks of 1,3-butadiene and n-pentane do not overlap by more than 1 % peak area with other compounds.

**NOTE** The following are examples of GC columns known to be suitable for 1,3-butadiene analysis:

a) 10 m x 0,32 mm internal diameter porous layer open tubular fused silica capillary column with  $10\text{ }\mu\text{m}$  film thickness;