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TECHNICAL SPECIFICATION
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CEN/TS 13130-16

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English version

**Materials and articles in contact with foodstuffs - Plastics
substances subject to limitation - Part 16: Determination of
caprolactam and caprolactam salt in food simulants**

Matériaux et objets en contact avec les denrées
alimentaires - Substances dans les matières plastiques
soumises à des limitations - Partie 16: Détermination du
caprolactame et des sels de caprolactame dans les
simulants d'aliments

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln
- Substanzen in Kunststoffen, die Beschränkungen
unterliegen - Teil 16: Bestimmung von Caprolactam und
Caprolactamsalz 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.

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 (CEN/TS 13130-16:2005) 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

Caprolactam, $C_6H_{11}NO$, PM/Ref. No. 14200, is a monomer used in the manufacture of certain plastics materials and articles intended to come into contact with foodstuffs. After the manufacture residual caprolactam can remain in the finished product and may migrate into foodstuffs coming into contact with that product.

Caprolactam sodium salt, $C_6H_{10}NO Na$, PM/Ref. No 14230, is a monomer used as a catalyst in the anionic polymerisation of caprolactam. After the manufacture residual caprolactam sodium salt can remain in the finished product and may migrate into foodstuffs coming into contact with that product. Caprolactam sodium salt hydrolyzes instantly when in contact with aqueous media.

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

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CEN/TS 13130-16:2005 (E)**1 Scope**

This document, part of EN 13130, specifies an analytical procedure for the determination of caprolactam in the food simulants distilled water, 3 % w/v aqueous acetic acid, 15 % v/v aqueous ethanol and rectified olive oil. The level of caprolactam monomer determined is expressed as mg caprolactam/kg of food or food simulant. The method is appropriate for the quantitative determination of caprolactam in an approximate analyte concentration range of 1,5mg/kg to 30 mg per kilogram of food simulant.

This method is also applicable to the determination of the specific migration of caprolactam sodium salt, PM/Ref No 14230, as caprolactam sodium salt instantaneously hydrolyzes to caprolactam when in contact with aqueous media.

NOTE The method should also be applicable to other aqueous food simulants as well as to other fatty food simulants e.g. sunflower oil, a mixture of synthetic triglycerides and volatile substitutes for fat simulants.

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.*

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3 Principle

The level of caprolactam in aqueous food simulants is determined by gas chromatography. Olive oil test samples are extracted with a mixture of water/ethanol and the resultant solutions analyzed by gas chromatography. Quantification is achieved using an internal standard, caprylolactam. Calibration is achieved by analysis of relevant simulants containing known amounts of caprolactam and caprylolactam.

Confirmation of caprolactam levels is established by gas chromatography using a column of different polarity. Alternatively, confirmation can also be 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 Caprolactam, (6-Caprolactam; ϵ -Caprolactam; Aza-2-cycloheptanone), $C_6H_{11}NO$
molecular weight: 113,16, purity > 98 % (GC).

4.1.2 Aza-2-cyclononanone, (8-aminooctanoic acid lactam; 8-Caprylolactam), $C_8H_{15}NO$
molecular weight 141,2, purity 99 %.

4.2 Chemicals

4.2.1 Ethanol, absolute

4.2.2 n-Heptane

4.2.3 Methanol

4.2.4 Water, deionized

4.3 Solutions

4.3.1 Ethanol/water = 1:2

Measure 200 ml ethanol (4.2.1) and make up with water (4.2.4) to 600 ml.

4.3.2 Standard stock solution of caprolactam in methanol (0,5 mg/ml)

Weigh to the nearest 0,1 mg approximately 50 mg of caprolactam (4.1.1) into a 100 ml volumetric flask. Dissolve the caprolactam in methanol and make up to the mark with methanol (4.2.3).

Calculate the exact concentration in milligrams of caprolactam per millilitre solution.

Repeat the procedure to obtain a second stock solution.

NOTE The solution can be stored in a well-closed container in the dark for a maximum period of 3 months at any temperature between - 20 °C to + 20 °C.

4.3.3 Internal standard stock solution of caprylolactam in methanol (0,35 mg/ml)

Weigh to the nearest 0,1 mg approximately 70 mg of caprylolactam (4.1.2) into a 200 ml volumetric flask. Dissolve the caprylolactam in methanol and make up to the mark with methanol (4.2.3).

Calculate the exact concentration in milligrams caprylolactam per millilitre of solution.

NOTE The solution can be stored in a well-closed container in the dark for a maximum period of 3 months at any temperature between - 20 °C and + 20 °C.

4.3.4 Diluted internal standard solution of caprylolactam in methanol (0,07 mg/ml)

Transfer by pipette 10,0 ml of the internal standard stock solution (4.3.3) into a 50 ml volumetric flask and make up to the mark with methanol (4.2.3).

Calculate the concentration in milligrams of caprylolactam per millilitre of solution.