



# SLOVENSKI STANDARD

## SIST-TS CEN/TS 13130-10:2005

01-april-2005

Materiali in predmeti v stiku z živilom - Plastične snovi, ki so predmet omejitve - Del 10: Določitev akrilamida v živilskih simuliranih snoveh

Materials and articles in contact with foodstuffs - Plastics substances subject to limitation - Part 10: Determination of acrylamide in food simulants

**iTeh STANDARD PREVIEW**

Werkstoffe und Gegenstände (in Kontakt mit Lebensmitteln) - Substanzen in Kunststoffen, die Beschränkungen unterliegen - Teil 10: Bestimmung von Acrylamid in Prüflebensmitteln

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Matériaux et objets en contact avec les denrées alimentaires - Substances dans les matières plastiques soumises à des limitations - Partie 10 : Détermination de l'acrylamide dans les simulants d'aliments

**Ta slovenski standard je istoveten z: CEN/TS 13130-10:2005**

### **ICS:**

67.250      Materiali in predmeti v stiku z živilom      Materials and articles in contact with foodstuffs

**SIST-TS CEN/TS 13130-10:2005**

**en,fr,de**

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TECHNICAL SPECIFICATION  
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**CEN/TS 13130-10**

February 2005

ICS 67.250

English version

**Materials and articles in contact with foodstuffs - Plastics  
substances subject to limitation - Part 10: Determination of  
acrylamide in food simulants**

Matériaux et objets en contact avec les denrées  
alimentaires - Substances dans les matières plastiques  
soumises à des limitations - Partie 10 : Détermination de  
l'acrylamide dans les simulants d'aliments

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln  
- Substanzen in Kunststoffen, die Beschränkungen  
unterliegen - Teil 10: Bestimmung von Acrylamid 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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COMITÉ EUROPÉEN DE NORMALISATION  
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## Foreword

This document (CEN/TS 13130-10:2005) 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.

[SIST-TS CEN/TS 13130-10:2005](#)

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*

Part 13: *Determination of 2,2-bis(4-hydroxyphenyl)propane (Bisphenol A) in food simulants*

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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

Acrylamide, PM/Ref. No 10630, is a monomer used in the manufacture of certain plastics materials and articles intended to come into contact with foodstuffs. After the manufacture, residual acrylamide can remain in the polymer and may migrate into foodstuffs coming into contact with that product.

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

NOTE 1 The analytical method described in this part of EN 13130 is the result of a study for the determination of the migration of acrylamide from plastic 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 and straightforward method is described in this part of EN 13130. The method was successfully pre-validated by the developing laboratory, using the four official EU food simulants to establish the precision data at the restriction criterion. Also migration tests were performed with samples containing acrylamide as monomer in contact with the four simulants for 10 d at 40 °C. Recovery of acrylamide from fortified simulants at the restriction level was found satisfactory. On testing of the method by a second laboratory it appeared that the testing laboratory was not able to achieve the required detection limit of 0,01 mg/kg. Therefore determination of the reproducibility was not performed by the testing lab. In addition the testing laboratory experienced difficulties with the stability of the base line when operating the HPLC UV detector at 202 nm. Based on the problems encountered by the testing laboratory the method description was modified to make critical points more clear.

NOTE 2 The confirmation method as given in this method description could be followed by the testing laboratory for water and olive oil, although again the detection limit could not be achieved. The lowest possible level was approximately 0,02 mg/kg. In the determination of acrylamide in 15 % v/v aqueous ethanol and 3 % w/v aqueous acetic acid, separation of the simulant and acrylamide was problematic. The testing laboratory concluded that the method was not suitable for the intended purpose. When however, the migration experiment is carried out in a more favourable ratio of contact area to simulant then the level of determination can be increased by -at most- a factor of five. In that case problems with detection limit and base line stability will be much less, and the method may appear to be suitable for the intended purpose.

Within the scope of the Standards, Measurement and Testing project "Monomers" it was not possible to re-test the method and therefore the method described is considered to be a useful analytical method, with limited validation data. Further testing is required to demonstrate that the method can be applied with the required accuracy and limit of detection.

**CEN/TS 13130-10:2005 (E)****1 Scope**

This document, part of EN 13130, specifies an analytical procedure for the determination of acrylamide in the food simulants water, 3 % w/v aqueous acetic acid, 15 % v/v aqueous ethanol and fat simulant. The level of acrylamide monomer determined is expressed as milligrams of acrylamide/kg of food simulant. The method is appropriate for the quantitative determination of acrylamide in approximate analyte concentration range of 0,01mg/kg to 0,1 mg/kg of food simulants.

The method should also be applicable to other fat simulants.

NOTE The suitability of the fat simulant should be assessed prior to setting up migration tests - it may be found necessary to use sunflower oil or a mixture of synthetic triglycerides if unacceptable interferences are found with olive oil.

**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**

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The level of acrylamide in food simulants is determined by direct injection of aqueous food simulants for analysis by high performance liquid chromatography with an ion exclusion column and ultra violet, UV, detection. Fat simulants are extracted with water and the aqueous extracts than analyzed by high performance liquid chromatography (HPLC). Quantification is achieved using external standards.

Confirmation of the identity of acrylamide is established by means of reversed phase HPLC using a column of different polarity but the same detection as used in the quantitative determination.

**4 Reagents**

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

**4.1 Analyte**

**Acrylamide**, CH<sub>2</sub>:CH CONH<sub>2</sub>, molecular weight 71,08, purity greater than 99 %.



## 4.2 Chemicals

4.2.1 Acetonitrile HPLC grade, suitable for low UV wavelength applications.

4.2.2 Methanol HPLC grade

4.2.3 Water HPLC grade

4.2.4 Sulfuric acid 0,05 mol/l in water

## 4.3 Solutions

### 4.3.1 Stock solution of acrylamide in methanol (500 µg/ml)

Weigh to the nearest 0,1 mg approximately 0,05 g of acrylamide into a 100 ml volumetric flask. Dissolve the acrylamide in methanol and fill up to the mark with methanol. Close and mix thoroughly.

Calculate the actual concentration in µg acrylamide/ml solution.

Repeat the procedure to provide a second stock solution.

Store the stock solutions at 5 °C for up to 3 months protected from light in septum capped glass vials with minimum headspace.

### 4.3.2 Diluted stock solution (10 µg/ml)

Using a graduated pipette, transfer 1,0 ml of the acrylamide stock solution (4.3.1) to a 50 ml volumetric flask and fill to the mark with methanol. This solution contains nominally 10 µg per millilitre of acrylamide.

Repeat the procedure using the second stock solution.

### 4.3.3 Intermediate standards

Using graduated pipettes, transfer 0 ml, 0,5 ml, 1,0 ml, 2,0 ml, 3,0 ml and 4,0 ml of the 10 µg/ml diluted stock solution (4.3.2) to a series of 10 ml volumetric flasks. Dilute to the mark with methanol and mix. These standards correspond nominally to 0 µg/ml, 0,5 µg/ml, 1,0 µg/ml, 2,0 µg/ml, 3,0 µg/ml and 4,0 µg/ml acrylamide.

### 4.3.4 HPLC mobile phase

Using a measuring cylinder, transfer 70 ml of 0,05 mol/l sulfuric acid (4.2.4) to a 1 litre volumetric flask and dilute to about 500 ml with water (4.2.3). Add, using a measuring cylinder, 70 ml of acetonitrile (4.2.1) and dilute to the mark with water (4.2.3).

NOTE This solution may require de-gassing prior to use.

## 5 Apparatus

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

5.1 High performance liquid chromatograph, equipped with a ultraviolet detector (UV).

Appropriate operating conditions shall be established for the specific equipment used for the determination.