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English Version

## Foodstuffs - Determination of acrylamide in food and coffee by gas chromatography-mass spectrometry (GC-MS)

Produits alimentaires - Dosage par CG-SM des produits alimentaires et café avec GC-MS

Lebensmittel - Bestimmung von Acrylamid in Lebensmitteln und Kaffee mit Gaschromatographie-Massenspektrometrie (GC-MS)

This Technical Specification (CEN/TS) was approved by CEN on 14 May 2017 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.

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## European foreword

This document (CEN/TS 17083:2017) has been prepared by Technical Committee CEN/TC 275 “Food analysis - Horizontal methods”, the secretariat of which is held by DIN.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

Annexes A, B, C and D are informative.

**WARNING 1 — The use of this Technical Specification can involve hazardous materials, operations and equipment. This document does not purport to address all the safety problems associated with its use. It is the responsibility of the user of this document to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.**

**WARNING 2 — Some precaution is required when using polyacrylamide-based plastics because acrylamide may leach from these materials.**

According to the CEN-CENELEC Internal Regulations, the national standards organizations of the following countries are bound to announce this Technical Specification: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

## 1 Scope

This Technical Specification specifies a method for the determination of acrylamide in cereal-based products, potato-based products and coffee by gas-chromatography mass spectrometry (GC-MS).

The method has been single-laboratory validated via the analysis of spiked samples (French fries (uncooked), bread, water biscuit, infant cereal, biscuit, green coffee, roast coffee and instant coffee), ranging from 30 µg/kg to 1 500 µg/kg acrylamide.

The results from the single laboratory validation were obtained by a laboratory with significant experience in acrylamide analysis. In addition, this method has also been studied by inter laboratory trial via the analysis of samples containing incurred acrylamide, ranging from approximately 200 µg/kg to 2 000 µg/kg. Critical points of the method are identified in 7.5 and Clause 8.

## 2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN ISO 3696:1995, *Water for analytical laboratory use - Specification and test methods (ISO 3696:1987)*

## 3 Principle

The test portion is extracted with hot water and isotopically labelled acrylamide is added as internal standard. High-fat samples are defatted with hexane, cleared with Carrez solution and centrifuged. Sample extracts are brominated and extracted with ethyl acetate. Following removal of the ethyl acetate by evaporation, triethylamine is added to partially debrominate, after which a portion of the sample extract is injected onto a GC-MS system for quantification. The chromatographic separation is obtained on a mid-polarity capillary GC column. The acrylamide derivative is ionized at 70 eV and recorded in selected ion monitoring (SIM) mode, and quantified by comparison with the stable isotopically labelled analogue.

## 4 Reagents

Use only reagents of recognized analytical grade and water complying with grade 1 of EN ISO 3696:1995 (electrical conductivity below 0,1 µS/cm at 25 °C), unless specified otherwise. Standard solutions are preferably prepared gravimetrically. An analytical balance (6.1) is used for the preparation of both native and stable isotope labelled acrylamide.

**WARNING** — Acrylamide has been classified by the International Agency for Research on Cancer (IARC) as probably carcinogenic to humans (see [2]). Bromine is very toxic and corrosive and hydrobromic acid is corrosive and hazardous. The prepared brominating solution (see 4.17) shall be considered as very toxic and corrosive.

Protective equipment such as laboratory coat, disposable gloves and safety glasses shall be used. All handlings of acrylamide, bromine, hydrobromic acid, brominating solution and organic solvents shall be performed in a fume cupboard with adequate air flow.

**IMPORTANT** — Dispose chemical waste according to applicable environmental rules and regulations. Bromine is very toxic to aquatic organisms hence discharge into the environment shall be avoided. Waste shall be disposed of appropriately.

### 4.1 Acrylamide, purity ≥ 99 %.