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Standards

Irish Standard
I.S. EN 16618:2015

Food analysis - Determination of acrylamide in food by liquid chromatography tandem mass spectrometry (LC-ESI-MS/MS)

I.S. EN 16618:2015

Incorporating amendments/corrigenda/National Annexes issued since publication:

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English Version

Food analysis - Determination of acrylamide in food by liquid chromatography tandem mass spectrometry (LC-ESI-MS/MS)

Analyse des produits alimentaires - Dosage de l'acrylamide dans les produits alimentaires par chromatographie en phase liquide couplée à la spectrométrie de masse en tandem (CL-ESI-SM-SM)

Lebensmittelanalytik - Bestimmung von Acrylamid in Lebensmitteln mit Flüssigchromatographie und Tandem-Massenspektrometrie (LC-ESI-MS/MS)

This European Standard was approved by CEN on 7 February 2015.

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Foreword

This document (EN 16618:2015) has been prepared by Technical Committee CEN/TC 275 “Food analysis - Horizontal methods”, the secretariat of which is held by DIN.

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of an identical text or by endorsement, at the latest by October 2015 and conflicting national standards shall be withdrawn at the latest by October 2015.

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1 Scope

This European Standard specifies a method for the determination of acrylamide in bakery ware such as bread, toasted bread, crisp bread, butter cookies, and biscuits, as well as potato products such as potato chips, potato crisps, and potato pan cake and roasted coffee, by liquid chromatography in combination with electrospray ionization and tandem mass spectrometry (LC-ESI-MS/MS). This method has been validated in an interlaboratory study via the analysis of both naturally contaminated and spiked samples, ranging from 14,3 µg/kg to 9 083 µg/kg. It was developed at the Swedish National Food Administration and validated in a study organized by the Directorate General Joint Research Centre (DG JRC), Swedish National Food Administration and the Nordic Committee on Food Analysis (NMKL), see [1] and [2].

The limit of quantification (LOQ) depends on the type of instrument used and on the actual performance of the instrument. The majority of the laboratories participating in the validation study were able to determine acrylamide in a butter cookie sample at a level of 14,3 µg/kg. Thus, the validation by interlaboratory study showed that LOQ can be expected to be in the range between below 15 µg/kg and 30 µg/kg.

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 1042:1999, *Laboratory glassware - One-mark volumetric flasks (ISO 1042:1998)*

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

3 Principle

Acrylamide is extracted with water and isotopic labelled acrylamide is added. The extract is centrifuged and the supernatant is cleaned up with two solid phase extraction (SPE) columns. The first SPE column contains silica based C18 groups as well as anion and cation exchangers, and since acrylamide is not retained by the column, the extract is just passed and collected. The reason for using this column is to retain as many matrix components as possible (non-polar compounds as well as anions and cations) without retaining acrylamide, i.e. this first SPE column is used as a chemical filter.

The second SPE column contains a polymer based phase with a relatively high capacity to bind acrylamide. The extract is loaded onto the column, the column is washed with water and finally eluted with a mixture of 60 parts per volume of methanol and 40 parts per volume of water. The purpose of this step, apart from further cleaning of the extract, is to concentrate the extract and to obtain low limits of quantification.

After evaporation of the methanol, the extract is analysed by LC-MS/MS. For this purpose an HPLC column with graphitized carbon as stationary phase is used, since the retention factor (k) is relatively high (k = 4 when no organic solvent is added in the mobile phase) compared to other commercially available columns.

4 Reagents

Use only reagents of recognized analytical grade and water complying with grade 1 of EN ISO 3696:1995, unless otherwise specified. Solvents shall be of quality for HPLC analysis.

4.1 Acrylamide (CAS 79-06-1), purity not less than 99,9 % mass fraction.