

Foodstuffs - Determination of melamine and cyanuric acid in foodstuffs by liquid chromatography and tandem mass spectrometry (LC-MS/MS)

EESTI STANDARDI EESSÕNA

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English Version

**Foodstuffs - Determination of melamine and cyanuric acid
in foodstuffs by liquid chromatography and tandem mass
spectrometry (LC-MS/MS)**

Produits alimentaires - Détermination de la teneur en
mélatrine et en acide cyanurique dans les produits
alimentaires par chromatographie en phase liquide
couplée à la spectrométrie de masse en tandem (CL-
SM/SM)

Lebensmittel - Bestimmung von Melamin und
Cyanursäure in Lebensmitteln mit
Flüssigchromatographie und Tandem-
Massenspektrometrie (LC-MS/MS)

This European Standard was approved by CEN on 10 April 2017.

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European foreword

This document (EN 16858:2017) 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of an identical text or by endorsement, at the latest by November 2017, and conflicting national standards shall be withdrawn at the latest by November 2017.

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1 Scope

This European Standard specifies a method for the determination of melamine and cyanuric acid in foodstuffs with liquid chromatography in combination with tandem mass spectrometry. The method has been validated in an interlaboratory study via the analysis of spiked samples of milk based infant formula, soy based infant formula, milk powder, whole milk, soy drink and milk chocolate ranging from 0,71 mg/kg to 1,43 mg/kg for melamine and 0,57 mg/kg to 1,45 mg/kg for cyanuric acid. The limits of quantification (LOQ) for melamine and cyanuric acid in food are 0,05 mg/kg and 0,25 mg/kg, respectively. The upper limit of the working range is up to 10 mg/kg for melamine and up to 25 mg/kg for cyanuric acid.

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

A test portion of the homogenous food sample is fortified with ^{13}C labelled internal standards (melamine and cyanuric acid). After incubation for at least one hour, water is added to the sample and after shaking, the slurry is dissolved in a mixture of acetonitrile and water. The sample is shaken and centrifuged. After separation of supernatant from sediments benzoguanamine is added as a recovery standard. An aliquot of the aqueous supernatant is injected into a LC-MS/MS system. The triple quadrupole mass spectrometer is coupled either to high performance liquid chromatography (HPLC) or to ultra performance liquid chromatography (UHPLC). Chromatography is based on hydrophilic interaction liquid chromatography (HILIC). Ionization is achieved by electrospray ionization (ESI) in multiple reaction monitoring (MRM).

4 Reagents

Use only reagents of recognized analytical grade and water complying with grade 1 of EN ISO 3696:1995, unless otherwise specified. Use only reagents with purity suitable for melamine and cyanuric acid analysis. Check the purity of the reagents and reference materials (e.g. standard solutions) by performing a blank test under the same conditions as used in the method. The chromatogram shall not show any interfering impurity at the retention time of compounds of interest.