

Foodstuffs - Determination of citrinin in food by liquid chromatography tandem mass spectrometry (LC-MS/MS)

EESTI STANDARDI EESSÕNA

NATIONAL FOREWORD

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ICS 67.050

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English Version

Foodstuffs - Determination of citrinin in food by liquid chromatography tandem mass spectrometry (LC-MS/MS)

Produits alimentaires - Dosage de la citrinine dans les produits alimentaires par chromatographie liquide couplée à une spectrométrie de masse en tandem (CL-SM/SM)

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

This European Standard was approved by CEN on 9 November 2018.

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European foreword

This document (EN 17203:2018) 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of an identical text or by endorsement, at the latest by June 2019, and conflicting national standards shall be withdrawn at the latest by June 2019.

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Introduction

The mycotoxin citrinin is a polyketide secondary metabolite produced mainly post-harvest in food and feed by several fungi of the genera *Penicillium* (e.g. *P. citrinum*), *Aspergillus* (e.g. *A. candidus*), and *Monascus* (e.g. *M. purpureus*). Citrinin occurs mainly in stored grains like rice, maize, wheat, barley, oats, and rye. Citrinin can be found as a contaminant in red fermented rice with *Monascus purpureus* and its formulated dietary supplements.

WARNING 1 — Suitable precaution and protection measures need to be taken when carrying out working steps with harmful chemicals. The latest version of the hazardous substances ordinance, Regulation (EC) No 1907/2006 [5] should be taken into account as well as appropriate National statements.

WARNING 2 — The use of this document 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 3 — Citrinin is known to have nephrotoxic properties, damaging the proximal tubules of the kidney [6].

1 Scope

This document describes a procedure for the determination of the citrinin content in food (cereals, red yeast rice (RYR)), herbs and food supplements by liquid chromatography tandem mass spectrometry (LC-MS/MS).

This method has been validated for citrinin in red yeast rice and in the formulated food supplements in the range of 2,5 µg/kg to 3000 µg/kg and in wheat flour in the range of 2,5 µg/kg to 100 µg/kg.

Laboratory experiences have shown that this method is also applicable to white rice, herbs such as a powder of *ginkgo biloba* leaves and the formulated food supplements in the range of 2,5 µg/kg to 50 µg/kg.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 ISO 3696, *Water for analytical laboratory use — Specification and test methods (ISO 3696)*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <http://www.iso.org/obp>

4 Principle

A test portion is humidified with a hydrochloric acid aqueous solution and extracted with ethyl acetate/acetonitrile/glacial acetic acid mixture for 60 min. Magnesium sulfate and sodium chloride are added to the extract, agitated and centrifuged in order to expel water and allow phase separation from the mixture. An aliquot of supernatant is collected, filtered, isotopic labelled internal standard (ISTD) solution is added and analysed by reversed phase LC-MS/MS. Quantification is based on matching citrinin/citrinin-¹³C ratios and citrinin concentrations.

5 Reagents

Use only reagents of recognized analytical grade and water complying with grade 1 of EN ISO 3696, unless otherwise specified. Commercially available solutions with equivalent properties to those listed may also be used.

5.1 Ethyl acetate, analytical grade or higher.

5.2 Acetonitrile, LC-MS grade. .

5.3 Glacial acetic acid (CH₃COOH), analytical grade or higher.

5.4 Glacial acetic acid (CH₃COOH), LC-MS grade.

5.5 Magnesium sulfate; anhydrous (MgSO₄), analytical grade or higher.