Foodstuffs - Determination of zearalenone in edible vegetable oils by LC-FLD or LC-MS/MS

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NATIONAL FOREWORD

See Eesti standard EVS-EN 16924:2017 sisaldab Euroopa standardi EN 16924:2017 ingliskeelset teksti.	This Estonian standard EVS-EN 16924:2017 consists of the English text of the European standard EN 16924:2017.
Standard on jõustunud sellekohase teate avaldamisega EVS Teatajas.	This standard has been endorsed with a notification published in the official bulletin of the Estonian Centre for Standardisation.
Euroopa standardimisorganisatsioonid on teinud Euroopa standardi rahvuslikele liikmetele kättesaadavaks 24.05.2017.	Date of Availability of the European standard is 24.05.2017.
Standard on kättesaadav Eesti Standardikeskusest.	The standard is available from the Estonian Centre for Standardisation.

Tagasisidet standardi sisu kohta on võimalik edastada, kasutades EVS-i veebilehel asuvat tagasiside vormi või saates e-kirja meiliaadressile standardiosakond@evs.ee.

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Foodstuffs - Determination of zearalenone in edible vegetable oils by LC-FLD or LC-MS/MS

Produits alimentaires - Dosage de la zéaralénone dans les huiles végétales alimentaires par CL-FLD ou CL-SM/SM

Lebensmittel - Bestimmung von Zearalenon in pflanzlichen Speiseölen mit LC-FLD oder LC-MS/MS

This European Standard was approved by CEN on 27 February 2017.

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European foreword

This document (EN 16924:2017) 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of an identical text or by endorsement, at the latest by November 2017, and conflicting national standards shall be withdrawn at the latest by November 2017.

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Introduction

The mycotoxin zearalenone is a resorcylic acid derivative, which is produced by several species of the fungi genus *Fusarium*, in particular by *Fusarium roseum var. graminearum*. Especially cereals like maize and wheat are affected, so that zearalenone can also be detected in the oils produced from them.

WARNING 1 — Suitable precaution and protection measures need to be taken when carrying out working steps with harmful chemicals. The hazardous substances ordinance, Regulation (EC) No 1907/2006 [3], should be taken into account as well as appropriate National statements e.g. such as in [4].

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 — Zearalenone is known to have strong oestrogenic effects.

1 Scope

This European Standard describes a procedure for the determination of the zearalenone content in edible vegetable oils specifically maize germ oil by either of the following techniques: High performance liquid chromatography with fluorescence detection (LC-FLD) or high performance liquid chromatography with tandem mass spectrometry (LC-MS/MS) after basic extraction of the diluted oil.

The method has been validated for zearalenone in naturally contaminated maize germ oil at levels of 61,2 µg/kg to 515 µg/kg [5].

Laboratory experiences [6] have shown that this method is also applicable to other vegetable oils such as wheat germ oil (n = 4), sunflower oil (n = 5), pumpkin seed oil (n = 1), soybean oil (n = 5), hemp seed oil (n = 5), rape seed oil (n = 11), and mixed oils including maize germ oil (n = 3). However occasionally, samples can result in interferences in the FLD-chromatograms. In this case, the detection with MS/MS is recommended.

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, *Water for analytical laboratory use — Specification and test methods (ISO 3696)*

3 Principle

After diluting the edible vegetable oil, zearalenone is extracted by shaking with an alkaline methanol - ammonium hydrogen carbonate mixture.

For the determination by LC-FLD, an aliquot of the centrifuged methanolic-alkaline extract is evaporated to dryness, then the residue is diluted in acidified LC-eluent and the zearalenone content is determined by LC-FLD.

For the determination by LC-MS/MS, an aliquot of the centrifuged methanolic-alkaline extract is used directly for analysis.

4 Reagents

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

4.1 Methanol, p. a. (pro analysis) for extraction.

4.2 Dilution solvent, (defatting solvent), *n*-hexane or, alternatively, *n*-heptane, p. a.

n-Heptane may be used instead of *n*-hexane, however, only *n*-hexane was used in the interlaboratory test.

4.3 Acetonitrile, LC quality.

4.4 Ammonium hydrogen carbonate (NH_4HCO_3).

4.5 Ammonium hydrogen carbonate solution, mass concentration ρ = 10 g/l.

Weigh in 1 g of ammonium hydrogen carbonate (4.4) into a 100 ml volumetric flask and fill up to the mark with water. Prepare a fresh solution each day of analysis.