

Foodstuffs - Determination of ergot alkaloids in cereals and cereal products by dSPE clean-up and HPLC-MS/MS

EESTI STANDARDI EESSÕNA

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English Version

**Foodstuffs - Determination of ergot alkaloids in cereals
and cereal products by dSPE clean-up and HPLC-MS/MS**

Produits alimentaires - Dosage des alcaloïdes de l'ergot
dans les céréales et les produits céréaliers par
purification par dSPE et CL-SM/SM

Lebensmittel - Bestimmung von Ergotalkaloiden in
Getreiden und Getreideerzeugnissen mit dSPE-
Reinigung und LC-MS/MS

This European Standard was approved by CEN on 21 September 2020.

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European foreword

This document (EN 17425:2021) 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of an identical text or by endorsement, at the latest by October 2021, and conflicting national standards shall be withdrawn at the latest by October 2021.

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Introduction

Ergot alkaloids are a group of mycotoxins produced by several species of *Claviceps* fungi growing on cereals and forage grass. These toxins are a risk for consumers as they can enter the food chain. All ergot alkaloids share a common structure, the ergoline system, and are divided into several classes, based on the presence of functional groups. The chiral carbon atom C-8 is responsible for the epimerization.

The isomers of each of these compounds are nominally known as 'ines' and the 'inines'. And besides, ergocryptine and ergocryptinine can both occur as α - and β -forms.

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 [3], 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 — Ergot alkaloids can cause vasoconstrictive, neurotoxic, reproductive and developmental adverse effects, and can be acutely and chronically toxic [4].

1 Scope

This document describes a method for the determination of the sum of six ergot alkaloids (ergocornine, ergometrine, ergocristine, ergotamine, ergosine and ergocryptine) and their -inine epimer pairs by liquid chromatography coupled with tandem mass spectrometry (LC-MS/MS) after clean-up by dispersive solid phase extraction (dSPE).

The method has been validated in the range 13,2 µg/kg to 168 µg/kg for the sum of the twelve ergot alkaloids, in rye flour, rye bread and cereal products (breakfast cereal, infant breakfast cereal, and crispbread) that contained rye as an ingredient, as well as seeded wholemeal flour and a barley and rye flour mixture.

Method performance was satisfactory in the range 24,1 µg/kg to 168 µg/kg, however at lower concentrations RSD_R values were greater than 44 %, and HorRat values exceeded 2,0, indicating the method may not be fully suitable at concentrations below 24 µg/kg for sum of ergot alkaloids, although it is suitable for screening at these concentrations.

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 <https://www.iso.org/obp>

4 Principle

Ergot alkaloids are extracted from cereals and cereal-based foods with a buffer at pH 8,9 and cleaned up with a dispersive solid phase material prior to filtering. Ergot alkaloids are detected and quantified by liquid chromatography coupled to tandem mass spectrometry (LC-MS/MS).

5 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.

NOTE Ergometrine and ergotamine are listed as Category 1 scheduled substances in Regulation (EC) No 273/2004 [5] on drug precursors. It is a requirement to have an appropriate licence in order to purchase and store these compounds (and their related -inine epimers).

5.1 Ergocornine, e.g. crystalline, as a film or as certified standard solution.

5.2 Ergocorninine, e.g. crystalline, as a film or as certified standard solution.

5.3 Ergocristine, e.g. crystalline, as a film or as certified standard solution.