Foods of plant origin - Determination of pesticide residues using LC-MS/MS following methanol extraction and clean-up using diatomaceous earth

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NATIONAL FOREWORD

Käesolev Eesti standard EVS-EN 15637:2008 sisaldab Euroopa standardi EN 15637:2008 ingliskeelset teksti.	This Estonian standard EVS-EN 15637:2008 consists of the English text of the European standard EN 15637:2008.
Standard on kinnitatud Eesti Standardikeskuse 15.12.2008 käskkirjaga ja jõustub sellekohase teate avaldamisel EVS Teatajas.	This standard is ratified with the order of Estonian Centre for Standardisation dated 15.12.2008 and is endorsed with the notification published in the official bulletin of the Estonian national standardisation organisation.
Euroopa standardimisorganisatsioonide poolt rahvuslikele liikmetele Euroopa standardi teksti kättesaadavaks tegemise kuupäev on 05.11.2008.	Date of Availability of the European standard text 05.11.2008.
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Foods of plant origin - Determination of pesticide residues using LC-MS/MS following methanol extraction and clean-up using diatomaceous earth

Aliments d'origine végétale - Détermination des résidus des pesticides par LC-MS/MS après extraction méthanolique et purification sur terre de diatomées

Pflanzliche Lebensmittel - LC-MS/MS-Verfahren zur Bestimmung von Pestizidrückständen mit Methanolextraktion und Reinigung an Diatomeenerde

This European Standard was approved by CEN on 13 September 2008.

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Foreword

This document (EN 15637:2008) 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of an identical text or by endorsement, at the latest by May 2009, and conflicting national standards shall be withdrawn at the latest by May 2009.

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1 Scope

This European Standard describes a method for the analysis of pesticide residues in foods of plant origin, such as fruits, vegetables, cereals, nuts as well as processed products including dried fruits. The method has been collaboratively studied on a large number of commodity/pesticide combinations.

2 Principle

The sample is extracted with methanol after addition of some water. After partition into dichloromethane the organic phase is evaporated and the residue is reconstituted with methanol. Quantification of pesticide residues is performed by liquid chromatography with tandem mass spectrometric detection, using electrospray ionisation. To achieve the required selectivity the mass spectrometer is operated in the selected reaction monitoring mode (SRM).

3 Reagents

3.1 General and safety considerations

Unless otherwise specified, use reagents of recognised analytical grade. Take every precaution to avoid possible contamination of water, solvents, inorganic salts, etc.

3.2 Ammonium formate

3.3 Sodium chloride

3.4 Water, HPLC quality

3.5 Dichloromethane, for residue analysis

3.6 Methanol, HPLC quality

3.7 Internal Standard (ISTD) solutions in methanol, ρ = 10 µg/ml to 50 µg/ml[1)]

Table 1 shows a list of potential internal standards that may be used in this method. The concentrations listed refer to the ISTD solutions that should be added at the first extraction step (5.2) and to standard solutions.

Table 1: Potential internal standards (ISTDs) or quality control (QC) standards

Name of the compound	Log P (octanol-water partion coefficient)	Chlorine atoms	Concentration C_{ISTD} µg/ml
Triphenyl phosphate	4,59	-	20
Tris-(1,3-dichlorisopropyl)-phosphate	3,65	6	50
Bis-nitrophenyl urea (nicarbazin)	3,76	-	10

1) ρ = mass concentration