Foods of plant origin - Multiresidue methods for the determination of pesticide residues by GC or LC-MS/MS - Part 3: Determination and confirmatory tests

EESTI STANDARDIKESKUS
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NATIONAL FOREWORD

See Eesti standard EVS-EN 12393-3:2013 sisaldab Euroopa standardi EN 12393-3:2013 inglisekeelset teksti.	This Estonian standard EVS-EN 12393-3:2013 consists of the English text of the European standard EN 12393-3:2013.
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 13.11.2013.	Date of Availability of the European standard is 13.11.2013.
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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English Version

Foods of plant origin - Multiresidue methods for the determination of pesticide residues by GC or LC-MS/MS - Part 3: Determination and confirmatory tests

Aliments d'origine végétale - Méthodes multirésidus de détermination de résidus de pesticides par CPG ou CL-SM/SM - Partie 3: Détermination et essais de confirmation

Pflanzliche Lebensmittel - Multiverfahren zur Bestimmung von Pestizidrückständen mit GC oder LC-MS/MS - Teil 3: Verfahren zur Bestimmung und Absicherung

This European Standard was approved by CEN on 21 September 2013.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN-CENELEC Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and United Kingdom.

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels

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Foreword

This document (EN 12393-3:2013) 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 2014, and conflicting national standards shall be withdrawn at the latest by May 2014.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 12393-3:2008.

This document will supersede EN 12393-3:2008 with the following significant technical changes:

a) introduction of the LC-MS/MS as a recommended technique for the determination of pesticide residues;

b) deletion of method L as no longer in use;

c) deletion of old Annex B with considerations concerning MS confirmation;

d) addition of a new Annex B with suitable GC-MS/MS operating conditions;

e) addition of new Annex C with typical LC-MS/MS operating conditions.

EN 12393, *Foods of plant origin — Multiresidue methods for the determination of pesticide residues by GC or LC-MS/MS*" is divided into three parts:

— Part 1 "*General considerations*" provides general considerations with regard to reagents, apparatus, gas chromatography, etc., applying to each of the analytical selected methods;

— Part 2 "*Methods for extraction and clean-up*" presents methods M, N and P for the extraction and clean-up using techniques such as liquid-liquid partition, adsorption column chromatography or gel permeation column chromatography, etc.;

— Part 3 "*Determination and confirmatory tests*" gives some recommended techniques for the qualitative and the quantitative measurements of residues and the confirmation of the results.

According to the CEN-CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Introduction

This European Standard comprises a range of multi-residue methods of equal status: no single method can be identified as the prime method because, in this field, methods are continuously developing. The selected methods included in this European Standard have been validated and/or are widely used throughout Europe.

Because these methods can be applied to the very wide range of food commodities/pesticide combinations, using different systems for determination, there are occasions when variations in equipment used, extraction, clean-up and chromatographic conditions are appropriate to improve method performance, see Clause 3.

1 Scope

This European Standard gives guidance on some recommended techniques for the determination of pesticide residues in foods of plant origin and on confirmatory tests.

The identity of any observed pesticide residue is confirmed, particularly in those cases in which it would appear that the maximum residue limit has been exceeded.

2 Normative references

Not applicable.

3 General

The methods specified in this European Standard permit identification and quantification of pesticide residues by gas chromatography using selective detectors or liquid chromatography with tandem-mass spectrometric detector (LC-MS/MS).

All relevant results require confirmation of identity and quantity.

The procedures listed for confirmation such as alternative GC columns, alternative GC detectors, high-performance liquid chromatography (HPLC), column fractionation, derivatisation, spectral measurements, etc. are all of value.

Results obtained using mass spectrometry (MS) present the most definitive evidence for confirmation/identification purpose.

As already described in the introduction, in certain occasions it is possible to improve the method performance by variations in equipment used, extraction, clean-up and chromatographic conditions. Such variations shall be always clearly documented and demonstrated to give valid results.

4 Determination

4.1 General

4.1.1 Identification

A number of parameters can be employed to determine the identity of an analyte present in the sample extract. This includes:

a) retention time of the analyte in question (RT) or, even better, the retention time ratio against the ISTD (Rt(A)/Rt(ISTD)) obtained from the same run (the simultaneous use of columns of different polarity improves this type of identification);

b) in case of MS or MS/MS detection, the relative abundance of simultaneously recorded signals (in general 3 ions are required in MS applications and 2 SRM transitions in MS/MS);

c) the application of high resolution mass spectrometry;

d) in case of MS with electron impact ionisation the comparison of the full scan mass spectrum of a suspected peak (when indicated after subtraction of background) with spectral libraries;