

**Foods of plant origin - Multiresidue methods for the  
determination of pesticide residues by GC or LC-MS/MS -  
Part 2: Methods for extraction and clean-up**

## EESTI STANDARDI EESSÕNA

## NATIONAL FOREWORD

See Eesti standard EVS-EN 12393-2:2013 sisaldab Euroopa standardi EN 12393-2:2013 inglisekeelset teksti.	This Estonian standard EVS-EN 12393-2:2013 consists of the English text of the European standard EN 12393-2:2013.
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](mailto:standardiosakond@evs.ee).

ICS 67.050

### Standardite reprodutseerimise ja levitamise õigus kuulub Eesti Standardikeskusele

Andmete paljundamine, taastekitamine, kopeerimine, salvestamine elektroonsesse süsteemi või edastamine ükskõik millises vormis või millisel teel ilma Eesti Standardikeskuse kirjaliku loata on keelatud.

Kui Teil on küsimusi standardite autorikaitse kohta, võtke palun ühendust Eesti Standardikeskusega:  
Aru 10, 10317 Tallinn, Eesti; [www.evs.ee](http://www.evs.ee); telefon 605 5050; e-post [info@evs.ee](mailto:info@evs.ee)

### The right to reproduce and distribute standards belongs to the Estonian Centre for Standardisation

No part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying, without a written permission from the Estonian Centre for Standardisation.

If you have any questions about copyright, please contact Estonian Centre for Standardisation:  
Aru 10, 10317 Tallinn, Estonia; [www.evs.ee](http://www.evs.ee); phone 605 5050; e-mail [info@evs.ee](mailto:info@evs.ee)

## English Version

## Foods of plant origin - Multiresidue methods for the determination of pesticide residues by GC or LC-MS/MS - Part 2: Methods for extraction and clean-up

Aliments d'origine végétale - Méthodes multirésidus de détermination de résidus de pesticides par CPG ou CL-SM/SM - Partie 2: Méthodes d'extraction et de purification

Pflanzliche Lebensmittel - Multiverfahren zur Bestimmung von Pestizidrückständen mit GC oder LC-MS/MS - Teil 2: Verfahren zur Extraktion und Reinigung

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

## Contents

Page

Foreword.....	3
Introduction .....	4
1     Scope .....	5
2     Normative references .....	5
3     Principles .....	5
4     General: Summary of procedures .....	6
5     Method M: Extraction with acetone and liquid-liquid partition with dichloromethane/light petroleum, if necessary clean-up on Florisil® .....	7
6     Method N: Extraction with acetone, liquid-liquid partition with dichloromethane or cyclohexane/ethyl acetate, clean-up with gel permeation and silica gel chromatography .....	10
7     Method P: Extraction with ethyl acetate, and if necessary, clean-up by gel permeation chromatography.....	35
Annex A (informative) Average water content of crops and foods .....	41
Bibliography .....	42

## Foreword

This document (EN 12393-2:2013) 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 12393-2:2008.

In comparison with EN 12393-2:2008, the following significant technical changes have been made:

- a) implementation of liquid chromatography in combination with tandem mass spectrometry (LC-MS/MS) for the quantification and/or confirmation of pesticide residues;
- b) incorporation of information on GC-MS/MS;
- c) deletion of method L as no longer in use;
- d) editorial updating of the document according to references, etc;
- e) enlargement of scope of method N concerning number of pesticides and validation data.

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 specifies methods for the extraction and clean-up of food samples of plant origin for quantitative determination of pesticide residues.

Different solvents can be used for this purpose. These pesticide residues are generally associated with other co-extracted compounds which would interfere in the analysis. To purify the crude extracts to be analysed, several techniques can be used.

This European Standard contains the following extraction and clean-up methods that have been subjected to interlaboratory studies and/or are adopted throughout Europe:

- method M: Extraction with acetone and liquid-liquid partition with dichloromethane/light petroleum, if necessary clean-up on Florisil® <sup>1)</sup> [1], [2], [3];
- method N: Extraction with acetone, liquid-liquid partition with dichloromethane or cyclohexane/ethyl acetate and clean-up with gel permeation and silica gel chromatography [4], [5];
- method P: Extraction with ethyl acetate, and if necessary, clean-up by gel permeation chromatography [6].

This European Standard specifies the details of methods M, N and P for the extraction and the clean-up of food samples of plant origin. Several solvents at different volumes are used for extraction. Techniques of clean-up are listed such as liquid-liquid partition, liquid chromatography on various adsorbents and gel permeation chromatography.

A table providing the couples (matrix/pesticide) which have been submitted to collaborative studies and a list of indicative applicability of the method to different pesticides are given for each method, wherever possible.

## 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 12393-1:2013, *Foods of plant origin — Multiresidue methods for the determination of pesticide residues by GC or LC-MS/MS — Part 1: General considerations*

EN 12393-3:2013, *Foods of plant origin — Multiresidue methods for the determination of pesticide residues by GC or LC-MS/MS — Part 3: Determination and confirmatory tests*

## 3 Principles

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.

The pesticide residues are extracted from the sample by the use of appropriate solvents, so as to obtain the maximum efficiency of extraction of the pesticide residues and minimum co-extracted substances which can give rise to interferences in the determination. Any interfering materials are removed from the sample extract to obtain a solution of the extracted pesticide residues in a solvent which is suitable for quantitative examination by the selected method of determination.

---

1) Florisil® is an example of a suitable product available commercially from U.S. Silica company. This information is given for convenience of users of this standard and does not constitute an endorsement by CEN of this product.