Mitterasvased toiduained. Mitme jäägi tekkimisega meetodid pestitsiidijääkide määramiseks gaasikromatograafia abil. Osa 2: Ekstraheerimise ja puhastamise meetodid

Foods of plant origin - Multiresidue methods for the gas chromatographic determination of pesticide residues - Part 2: Methods for extraction and clean-up



#### **EESTI STANDARDI EESSÕNA**

#### **NATIONAL FOREWORD**

This Estonian standard EVS-EN 12393-2:2008 consists of the English text of the European standard EN 12393-2:2008.

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.

Standard on kättesaadav Eesti standardiorganisatsioonist.

The standard is available from Estonian standardisation organisation.

ICS 67.080.01

**Võtmesõnad:** ekstraheerimismeetodid, gaasikromatograafia, keemiline analüüs, kromatograafiline analüüs, pestitsiidid, pestitsiidijäägid, puhtus, sisalduse määramine, toiduainetooted

#### Standardite reprodutseerimis- ja levitamisõigus kuulub Eesti Standardikeskusele

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

## EUROPEAN STANDARD NORME EUROPÉENNE

**EUROPÄISCHE NORM** 

EN 12393-2

November 2008

ICS 67.080.01

Supersedes EN 12393-2:1998

#### **English Version**

# Foods of plant origin - Multiresidue methods for the gas chromatographic determination of pesticide residues - Part 2: Methods for extraction and cleanup

Aliments d'origine végétale - Méthodes multirésidus de détermination par chromatographie en phase gazeuse de résidus de pesticides - Partie 2: Méthodes d'extraction et de purification Pflanzliche Lebensmittel - Multiverfahren zur gaschromatographischen Bestimmung von Pestizidrückständen - Teil 2: Verfahren zur Extraktion und Reinigung

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

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 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 Management Centre has the same status as the official versions.

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



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

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#### **Foreword**

This document (EN 12393-2:2008) 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.

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 12393-2:1998 with following significant technical changes:

- a) deletion of method O: Extraction with acetonitrile, liquid-liquid partition with light petroleum and clean-up on a Florisil® column:
- b) addition of an alternative partition step and expansion of the pesticide spectrum in method N: Extraction with acetone, liquid-liquid partition with dichloromethane or cyclohexane/ethyl acetate, clean-up with gel permeation and silica gel chromatography.

This European Standard EN 12393 "Foods of plant origin - Multiresidue methods for the gas chromatographic determination of pesticide residues" is divided in 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 L to 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, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and the United Kingdom.

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### 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 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, ap, nina. ondition. 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 standard contains the following extraction and clean-up methods that have been subjected to interlaboratory studies and /or are adopted throughout Europe:

- method L: Extraction with acetone, liquid-liquid partition with dichloromethane and clean-up on a silica gel/charcoal column [1];
- method M: Extraction with acetone and liquid-liquid partition with dichloromethane/light petroleum, if necessary clean-up on Florisil<sup>®</sup> 1) [2], [3], [4];
- method N: Extraction with acetone, liquid-liquid partition with dichloromethane or cyclohexane/ethyl acetate and clean-up with gel permeation and silica gel chromatography [5], [6];
- method P: Extraction with ethyl acetate, and if necessary, clean-up by gel permeation chromatography [7].

This European Standard specifies the details of methods L to 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 referenced documents are indispensable for the application 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 12393-1:2008, Foods of plant origin – Multiresidue methods for the gas chromatographic determination of pesticide residues – Part 1: General considerations

EN 12393-3, Foods of plant origin – Multiresidue methods for the gas chromatographic determination of pesticide residues – 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.

<sup>1)</sup> Florisil<sup>®</sup> is an example of a suitable product available commercially. This information is given for convenience of users of this standard and does not constitute an endorsement by CEN of this product.