

Foods of plant origin - Determination of pesticide residues using GC-MS and/or LC-MS/MS following acetonitrile extraction/partitioning and cleanup by dispersive SPE - QuEChERS-method

EESTI STANDARDI EESSÕNA

NATIONAL FOREWORD

Käesolev Eesti standard EVS-EN 15662:2008 sisaldab Euroopa standardi EN 15662:2008 ingliskeelset teksti.

Standard on kinnitatud Eesti Standardikeskuse 15.12.2008 käskkirjaga ja jõustub sellekohase teate avaldamisel EVS Teatajas.

Euroopa standardimisorganisatsioonide poolt rahvuslikele liikmetele Euroopa standardi teksti kättesaadavaks tegemise kuupäev on 12.11.2008.

Standard on kättesaadav Eesti standardiorganisatsioonist.

This Estonian standard EVS-EN 15662:2008 consists of the English text of the European standard EN 15662:2008.

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.

Date of Availability of the European standard text 12.11.2008.

The standard is available from Estonian standardisation organisation.

ICS 67.050

Võtmesõnad:

Standardite reprodutseerimis- ja levitamiseõigus 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.

Kui Teil on küsimusi standardite autorikaitse kohta, palun võtke ühendust Eesti Standardikeskusega:
Aru 10 Tallinn 10317 Eesti; www.evs.ee; Telefon: 605 5050; E-post: info@evs.ee

English Version

Foods of plant origin - Determination of pesticide residues using
GC-MS and/or LC-MS/MS following acetonitrile
extraction/partitioning and clean-up by dispersive SPE -
QuEChERS-method

Aliments d'origine végétale - Méthode polyvalente de
détermination des résidus des pesticides par GC-SM et
SL/SM(SM) avec extraction/partition avec de l'acétonitrile et
nettoyage par SPE dispersés - Méthode QuEChERS

Pflanzliche Lebensmittel - Bestimmung von
Pestizidrückständen mit GC-MS und/oder LC-MS/MS nach
Acetonitril-Extraktion/Verteilung und Reinigung mit
dispersiver SPE - QuEChERS-Verfahren

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

Management Centre: rue de Stassart, 36 B-1050 Brussels

Contents

Page

Foreword.....	3
1 Scope	4
2 Principle	4
3 Reagents	4
4 Apparatus	8
5 Procedure	10
6 Evaluation of results	16
7 Confirmatory tests	21
8 Precision	21
9 Test report	21
Annex A (informative) Examples of experimental conditions	22
Annex B (informative) Precision data	25
Annex C (informative) Procedure schematically (for 10 g sample)	75
Annex D (informative) Complementary information	76
Bibliography	81

Foreword

This document (EN 15662:2008) 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.

WARNING — The application of this standard may involve hazardous materials, operations and equipment. This standard does not claim to address all the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

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.

1 Scope

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

2 Principle

The homogeneous sample is extracted with the help of acetonitrile. Samples with low water content (< 80 %) require the addition of water before the initial extraction to get a total of approximately 10 g of water. After addition of magnesium sulfate, sodium chloride and buffering citrate salts, the mixture is shaken intensively and centrifuged for phase separation. An aliquot of the organic phase is cleaned-up by dispersive solid phase extraction (D-SPE) employing bulk sorbents as well as magnesium sulfate for the removal of residual water. Following clean-up with amino-sorbents (e.g. primary secondary amin sorbent, PSA) extracts are acidified by adding a small amount of formic acid, to improve the storage stability of certain base-sensitive pesticides. The final extract can be directly employed for GC- and LC-based determinative analysis. Quantification is performed using an internal standard, which is added to the extract after the initial addition of acetonitrile. A brief overview of the method is shown in the flowchart in Annex C.

3 Reagents

3.1 General and safety aspects

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

DISCLAIMER — This standard refers to several trade names products and instruments which are commercially available and suitable for the described procedure. This information is given for the convenience of users of this European Standard and does not constitute an endorsement by CEN of the products named. Equivalent products may be used if they can be shown to lead to equivalent results.

3.2 Water, HPLC quality

3.3 Acetonitrile, HPLC quality

3.4 Methanol, HPLC quality

3.5 Ammonium formate

3.6 Magnesium sulfate, anhydrous, grit, e.g. Fluka No. 63135

Phthalates may be removed in a muffle furnace by heating to 550 °C (e.g. overnight).

3.7 Magnesium sulfate, anhydrous, fine powder

Phthalates may be removed in a muffle furnace by heating to 550 °C (e.g. overnight).

3.8 Sodium chloride

3.9 Disodium hydrogencitrate sesquihydrate