Foods of plant origin - Multimethod for the determination of pesticide residues using GC- and LC-based analysis following acetonitrile extraction/partitioning and clean-up by dispersive SPE - Modular QuEChERS-method

EESTI STANDARDIKESKUS
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EESTI STANDARDI EESSÕNA

NATIONAL FOREWORD

See Eesti standard EVS-EN 15662:2018 sisaldab Euroopa standardi EN 15662:2018 ingliskeelset teksti.	This Estonian standard EVS-EN 15662:2018 consists of the English text of the European standard EN 15662:2018.
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 23.05.2018.	Date of Availability of the European standard is 23.05.2018.
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 - Multimethod for the determination of pesticide residues using GC- and LC-based analysis following acetonitrile extraction/partitioning and clean-up by dispersive SPE - Modular QuEChERS-method

Aliments d'origine végétale - Multiméthode de détermination des résidus de pesticides par analyse CG et CL après extraction/partition avec de l'acétonitrile et purification par SPE dispersive - Méthode modulaire QuEChERS

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

This European Standard was approved by CEN on 27 December 2017.

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, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and United Kingdom.

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

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European foreword

This document (EN 15662:2018) 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 November 2018, and conflicting national standards shall be withdrawn at the latest by November 2018.

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

This document supersedes EN 15662:2008.

With the revised version, some amendments and improvements have been taken into consideration, notably:

— the more precise differentiation between feasible modes of operation (Table 1 to Table 5);

— the opportunity to report the applied modes of operation (e.g. extraction or clean-up modules) in a simple way;

— clear indications of approved modes of operation for particular commodities (Table 6);

— the optimization of extraction efficiency by longer extraction time;

— the specification of suitable parameters for the detection with UPLC-MS/MS and GC-MS/MS;

— new approaches for the quantitation of pesticide residues including a simplified procedure for the calculation of residue levels;

— references to the improved validation data for the method (see Table 7 and CEN/TR 17063);

— a list of abbreviations has been added in Annex C.

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 organisations 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, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

1 Scope

This European Standard stipulates a method for the analysis of pesticide residues in foods of plant origin, such as fruits (including dried fruits), vegetables (including dried vegetables), cereals and many processed products thereof by using GC, GC-MS(/MS), and/or LC-MS(/MS). The method has been collaboratively studied on a large number of commodity/pesticide combinations. Precision data are summarized in CEN/TR 17063. Guidelines for calibration are outlined in CEN/TS 17061.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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.

CEN/TS 17061:2017, *Foodstuffs - Guidelines for the calibration and quantitative determination of pesticide residues and organic contaminants using chromatographic methods*

3 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 amine sorbent, PSA) and if necessary graphitized carbon black (GCB) or octadecylsilane (ODS), 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 analysis. Suitable detectors for GC analysis are mass-selective detectors (MS or MS/MS) with unit or high mass resolution or other GC detectors, such as flame photometric detector, FPD, and electron capture detector, ECD. For the analysis with LC hyphenations with tandem mass-spectrometry (LC-MS/MS) or high resolution mass-spectrometry are particularly suitable. Quantification may be performed using an internal standard, which is added to the test portion before the first extraction, but this is not mandatory. Details for calibration, see CEN/TS 17061.

4 Preparation and storage of the samples

4.1 General

Sample processing and storage procedures should be demonstrated to have no significant effect on the residues present in the test sample (sometimes also called “analytical sample”). Processing should also ensure that the test sample is homogeneous enough so that portion to portion (sub-sampling) variability is acceptable. If a single analytical portion is unlikely to be representative of the test sample, larger or replicate portions shall be analysed, to provide a better estimate of the true value. The degree of comminution should support a quantitative residue extraction.

4.2 Laboratory sample

A laboratory sample that is wholly or extensively spoiled or degraded should not be analysed. When possible, prepare laboratory samples immediately after arrival and in any event, before any significant physical or chemical changes have taken place. If a laboratory sample cannot be prepared without delay, it should be stored under appropriate conditions to keep it fresh and to avoid deterioration. Dried or similarly processed samples should be analysed within their stated shelf life.