SLOVENSKI STANDARD
SIST EN 12393-1:2009

01-februar-2009

Nadomeġ ļa:
SIST EN 12393-1:1999

Ģivila rastlinskega izvora - Multirezidualne metode za doloļ evanje ostankov pesticidov s plinsko kromatografijo - 1. del: Sploġna navodila

Foods of plant origin - Multiresidue methods for the gas chromatographic determination of pesticide residues - Part 1: General considerations

Pflanzliche Lebensmittel - Multiverfahren zur gaschromatographischen Bestimmung von Pestizidrückständen - Teil 1: Allgemeines

Aliments d'origine végétale - Méthodes multirésidus de détermination par chromatographie en phase gazeuse de pesticides - Partie 1: Généralités

Ta slovenski standard je istoveten z: **EN 12393-1:2008**

ICS:

67.050	Splošne preskusne in analizne metode za živilske proizvode	General methods of tests and analysis for food products
67.080.01	Sadje, zelenjava in njuni proizvodi na splošno	Fruits, vegetables and derived products in general

SIST EN 12393-1:2009 **en,fr,de**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 12393-1

November 2008

ICS 67.080.01

Supersedes EN 12393-1:1998

English Version

Foods of plant origin - Multiresidue methods for the gas chromatographic determination of pesticide residues - Part 1: General considerations

Aliments d'origine végétale - Méthodes multirésidus de détermination par chromatographie en phase gazeuse de résidus de pesticides - Partie 1: Généralités

Pflanzliche Lebensmittel - Multiverfahren zur gaschromatographischen Bestimmung von Pestizidrückständen - Teil 1: Allgemeines

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

This document (EN 12393-1:2008) 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 12393-1:1998 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) complete revision of subclause 6.3: Preparation and storage of the samples.

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 selected analytical 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.

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, 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 3.1.

1 Scope

This European Standard gives general considerations for the determination of pesticide residues in foods of plant origin.

Each method specified in this European Standard is suitable for identifying and quantifying a definite range of those organohalogen, and/or organophosphorus and/or organonitrogen pesticides which occur as residues in foodstuffs of plant origin.

This European Standard contains the following 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® [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 with gel permeation chromatography [7].

The applicability of the four methods L to P for residue analysis of organohalogen, organophosphorus and organonitrogen pesticides, respectively, is given for each method.

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 12393-2, *Foods of plant origin – Multiresidue methods for the gas chromatographic determination of pesticide residues – Part 2: Methods for extraction and clean-up*

EN 12393-3:2008, *Foods of plant origin – Multiresidue methods for the gas chromatographic determination of pesticide residues – Part 3: Determination and confirmatory tests*

3 Principle

3.1 General

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.

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

The methods described in this European Standard are based on a four-stage process (in some cases two stages may be combined, in whole or in part), as given in 3.2 to 3.5.

Quality control procedures for pesticide residue analysis, e.g. published by the European Commission [8], should be followed in its updated versions.

3.2 Extraction

Extraction of the residues from the sample matrix by the use of appropriate solvents, so as to obtain the maximum efficiency of extraction of the residues and minimum co-extraction of any substances which can give rise to interferences in the determination.

3.3 Clean-up

Removal of interfering materials from the sample extract to obtain a solution of the extracted residue in a solvent which is suitable for determination by the selected method of determination.

3.4 Determination

Gas chromatography (GC) with selective detectors may be used: electron-capture detection (ECD) for organohalogen, thermionic detector (NPD, P-mode or N/P mode) for organophosphorus and organonitrogen compounds and flame-photometric detector (FPD) for organophosphorus and organosulfurous pesticides. Hall detector (ECHD), atomic emission detector (AED) and mass spectrometry (MS) may also be used for a large class of pesticides.

3.5 Confirmation

Procedures to confirm the identity and quantity of observed residues, particularly in those cases where it would appear that the maximum residue limit (MRL) has been exceeded.

4 Reagents

4.1 General

Use reagents of purity suitable for pesticide residue analysis and check their purity (see 4.2). If required, purify water and solvents used, e.g. as described in Annex A, and check their purity (see 4.2). Purify and periodically activate adsorbents according to the requirements of the different analytical methods; check their purity (see 4.2).

Take every precaution to avoid possible contamination of water, solvents, adsorbents, etc. from plastics and rubber materials.

4.2 Check for purity of reagents

4.2.1 Solvents

Concentrate solvents by the factor involved in the respective method to be used. Test for purity by GC under the same conditions as used in the method. The chromatogram should not show any interfering impurity. Extract or concentrate acetonitrile, acetone, ethyl acetate, hexane, light petroleum and dichloromethane in the same volume as used in the method and examine the resulting solution as above by GC.

4.2.2 Water

Extract 10 parts by volume of water with one part by volume of *n*-hexane or light petroleum, dichloromethane or any other non water miscible solvent used in the method. Separate the organic phase, concentrate by the

factor involved in the respective method and test for purity by GC under the same conditions as used in the method. The chromatogram should not show any interfering impurity.

4.2.3 Inorganic salts

Extract inorganic salts, for example sodium chloride, after purification according to Annex A or the requirements of the different analytical methods. Extract the salts and any aqueous solution used, with *n*-hexane or light petroleum, dichloromethane or any other non water miscible solvent used in the method. Concentrate the extract by the factor involved in the respective method and test the purity by GC under the same conditions as used in the method. The chromatogram should not show any interfering impurity.

4.2.4 Adsorbents

Elute an amount of adsorbent equal to that used in the analytical method with the corresponding type and volume of solvent or solvent mixture. Concentrate the eluate as indicated in the analytical method and test for purity by GC. The chromatogram should not show any interfering impurity. Check the activity of adsorbents regularly as described in the methods L to P (see EN 12393-2).

4.2.5 Standard materials and solutions

Use standard materials of at least 95 % purity and traceable quality as standards for residue analysis.

Ensure dilute solutions are prepared and checked frequently, and that standard solutions are stored in glass bottles in a refrigerator and every precaution is taken to avoid possible contamination from plastics or rubber materials. Ensure that the standard solutions are not directly exposed to sunlight or ultraviolet light for prolonged periods of time. Examine analytical standards for impurities.

NOTE 1 When stored at -20 °C, standard materials are generally stable for at least a year. To allow equilibration, it is recommended to allow the standards to come up to room temperature before the containers are opened. Stock solutions of concentration 1 mg/ml, if kept in a freezer (at about -20 °C), are usually stable for 6 months.

NOTE 2 Changes in volume due to solvent evaporation, for example through the space between a glass stopper and the neck of a flask, can be a source of error. Therefore, the use of poly tetra fluoro ethylene (PTFE) screw-cap flasks is recommended for the storage of stock and standard solutions.

NOTE 3 Experience has shown that errors introduced in the preparation, handling and storage of standards and standard solutions are major sources of inaccuracies. Experiences obtained by other national, European and international bodies should be observed [8], [9].

4.3 Safety aspects associated with reagents

4.3.1 General

The analysis of pesticide residues in a food matrix includes the use of several hazardous chemicals. Safety precautions as given in 4.3.2 and 4.3.3 shall be observed at all times.

4.3.2 Pesticides

Many pesticides are extremely toxic by various routes of exposure, especially in their concentrated forms. As an example, the family of organophosphorus pesticides is consistently highly toxic, not only by oral ingestion, but dermally and by inhalation as well. When working with standard materials, standard solutions, etc. observe the following minimal precautions at all times (consult safety data sheets or labels for additional information):

a) Perform all laboratory sampling, mixing, weighing, etc., under an effective fume removal device in an area having a good forced ventilation of non-recirculated air; or wear a gas mask of the proper type. If the mask is used, replace cartridges as recommended, since using a contaminated mask could be worse than wearing no mask at all;