

SLOVENSKI STANDARD SIST EN 12393-3:2009

01-februar-2009

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Foods of plant origin - Multiresidue methods for the gas chromatographic determination of pesticide residues - Part 3: Determination and confirmatory tests

Pflanzliche Lebensmittel - Multiverfahren zur gaschromatographischen Bestimmung von Pestizidrückständen - Teil 3: Verfahren zur Bestimmung und Absicherung

Aliments d'origine végétale - Méthod<u>es multirésidus de</u> détermination par chromatographie en phase gazeuse de résidus de pesticides - Partie 3 : Détermination et essais de confirmation 3079aea71a48/sist-en-12393-3-2009

Ta slovenski standard je istoveten z: EN 12393-3:2008

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 12393-3:2009

en,fr,de



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SIST EN 12393-3:2009

EUROPEAN STANDARD NORME EUROPÉENNE EUROPÄISCHE NORM

EN 12393-3

November 2008

ICS 67.080.01

Supersedes EN 12393-3:1998

English Version

Foods of plant origin - Multiresidue methods for the gas chromatographic determination of pesticide residues - Part 3: Determination and confirmatory tests

Aliments d'origine végétale - Méthodes multirésidus de détermination par chromatographie en phase gazeuse de résidus de pesticides - Partie 3: Détermination et essais de confirmation Pflanzliche Lebensmittel - Multiverfahren zur gaschromatographischen Bestimmung von Pestizidrückständen - Teil 3: Verfahren zur Bestimmung und Absicherung

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.

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Ref. No. EN 12393-3:2008: E

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Foreword

This document (EN 12393-3:2008) 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 12393-3:1998 with following significant technical changes:

- a) revision of Clause 4 "Determination": outdated techniques are deleted;
- b) revision of Clause 5 "Confirmatory tests": outdated techniques are deleted.

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., 3079aea71a48/sist-en-12393-3-2009
- 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 Clause 3.

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1 Scope

This European Standard gives guidance on some recommended techniques for the determination of pesticide residues in foods of plant origin and on confirmatory tests.

The identity of any observed pesticide residue is confirmed, particularly in those cases in which it would appear that the maximum residue limit has been exceeded.

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

3 General

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The methods specified in this European Standard permit identification and quantification of pesticide residues by gas chromatography using selective detectors: **OS.iteh.ai**)

All relevant results require confirmation of identity and quantity.

The procedures listed for confirmation such as alternative GC columns, alternative GC detectors, highperformance liquid chromatography (HPLC), column fractionation, derivatization, spectral measurements, etc. are all of value.

Results obtained using mass spectrometry (MS) present the most definitive evidence for confirmation/identification purpose.

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.

4 Determination

4.1 Gas chromatography (GC)

4.1.1 General

A suitable GC system, preferably equipped with separate heaters for injector, detector and column ovens, shall be used. The facility to inject directly on the GC column is generally of advantage. Although the choice of the different parts of the GC system is a matter for the experience of the analyst, the following general recommendations are made.

The detectors should be properly adjusted, according to the manufacturers' instructions. Variations in detector sensitivity should be checked periodically by verifying the linearity of the calibration curves using standard solutions of pesticides.

The quantification unit of the gas chromatographic apparatus should include an integration system which permits the calculation not only of peak heights but also peak areas.

It has been found in practice that equivalent results can be achieved despite the adoption of different GC conditions, and different makes of instruments. On the other hand, specifying standard GC parameters does not guarantee that the quality of the results generated will be identical.

For typical GC conditions, see Annex A.

4.1.2 GC columns

Columns should be conditioned for at least 24 h at a temperature near the maximum recommended operating temperature with the type of stationary phase used and should then be tested for their efficiency and selectivity at the required operating temperature using standard mixtures of pesticides. The end of the column should always be disconnected from the detector during conditioning.

Pure (oxygen-free) and dry (water-free) nitrogen, hydrogen or helium should be used as carrier gas. The flow rate depends on the size and type of column used. Generally, ensure that gas flow rates are controlled as accurately as possible. Gas purification filters should be installed for all gas supplies and replaced regularly.

Finally, make sure that the GC conditions (column length, stationary phase type, injector, detector and column temperatures, gas flow rates, etc.) are such that the separation of the pesticides likely to be present is as complete as possible.

Fused silica columns having an internal diameter of 0,20 mm to 0,35 mm and a length of between 10 m and 60 m have proved particularly satisfactory because of their separation efficiency, service life and mechanical properties. Wide bore columns having an internal diameter of 0,5 mm to 0,8 mm may also be useful in some cases.

The following stationary phases are frequently used as coatings; 2009

_	SE-30 ¹⁾	https://standards.iteh.ai/catalog/standards/sist/80e16788-ab43-499c-9fe9-(equivalent to OV-1, DB_{0}
	SE-54	(equivalent to DB-5, CP Sil 8, BP-5, SPB-5, etc.)
	OV-17	(equivalent to OV-11, OV-22, SP-2250, DC-710, DB-608, etc.)
	DB-1301	(equivalent to DB-624, etc.)
	DB-1701	(equivalent to OV-1701, CP-SIL-19-CB, BP-10, SPB-7, etc.)
	OV-225	(equivalent to DB-225, SIL-43-CB, SPB-2330, etc.)
	Wax	(equivalent to DB-Wax, Wax-52-CB, Carbowax 20M ¹⁾ , etc.)

4.1.3 Injection techniques

Various injection techniques are useful such as split/splitless injection or programmed temperature vaporization (PTV) injection.

The applicability of these techniques depends on the apparatus used and on special requirements.

¹⁾ SE-30... Carbowax 20M are examples of suitable products available commercially. This information is given for the convenience of users of this standard and does not constitute an endorsement by CEN of these products.

4.1.4 Detectors

See EN 12393-1:2008, 3.4.

4.2 Preliminary tests

Determine the linear dynamic range of detector response under the actual GC conditions used by injecting diluted standard solutions.

Inject into the gas chromatograph an appropriate volume (between 1,0 µl and 10,0 µl, depending on the system) of the purified extracts obtained according to the analytical method used in EN 12393-2. The chromatogram so obtained should enable both the identity and the approximate concentration of the compounds present in the extracts to be established.

4.3 Determination

Make sure that all the measurements are performed within the linear dynamic range of the system. Prepare at least two standard solutions of the identified pesticide in the same solvent as used in the final extract. Their concentrations should encompass the probable concentration expected in the final extract. Then inject equal volumes of the final extracts obtained and of the two or more standard solutions into the gas chromatograph. It is essential that the injections of the purified portions of the sample extracts are preceded and followed by injection of the standard solutions.

Measure the peak areas or peak heights. The results obtained from any two successive injections of the same standard solution should not differ more than approximately 10 % from each other. Inclusion of an internal standard is useful standard is useful.

It is necessary to ensure that the standard materials and samples are dissolved in the same solvent, otherwise varying evaporation profiles will result which could lead to changes in the retention times and peak areas or heights. SIST EN 12393-3:2009

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A quantitative determination is possible if the mean of recoveries from replicate determinations falls within the range of 70 % up to 110 %, with a relative standard deviation less or equal to 20 %. Compliance with this condition has to be checked periodically by repeated measurements of recovery from samples containing known additions of the relevant standard material.

5 Confirmatory tests

5.1 General

When analyses are performed for regulatory purposes, it is especially important that confirmatory tests for identification and quantification of the residues are carried out before reporting adversely on samples containing residues of pesticides not usually associated with that commodity or where MRLs appear to have been exceeded [1]. Contamination of samples with non pesticidal chemicals occurs from time to time and in some chromatographic methods these compounds can have similar properties to pesticides and could therefore be misidentified as such. Examples in gas chromatography include the responses of electroncapture detectors to phthalate esters and of phosphorus specific detectors to compounds containing sulfur.

Confirmatory tests can be divided into two types: quantitative tests are necessary when MRLs appear to be exceeded whilst qualitative confirmation of identity is also needed in these cases, and when atypical residues are encountered. Qualitative tests can involve chemical reactions or separations where some loss of the residue occurs. Particular problems occur in confirmation when MRLs are set at or about the limit of determination.

The need for confirmatory tests can depend upon the type of sample or its known history. In many substrates, certain residues are nearly always found. For a series of samples of similar origin it may only be necessary to