

Non-fatty foods — Multiresidue methods for the gas chromatographic determination of pesticide residues —

Part 1: General considerations

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ICS 67.050

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Summary of pages

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Aliments non gras — Méthodes multirésidues de détermination par chromatographie en phase gazeuse de résidus de pesticides —
Partie 1: Généralités

Fettarme Lebensmittel — Multiverfahren zur gaschromatographischen Bestimmung von Pestizidrückständen — Teil 1: Allgemeines

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CEN

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Foreword

This European Standard 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of an identical text or by endorsement, at the latest by April 1999, and conflicting national standards shall be withdrawn at the latest by April 1999.

This EN 12393, *Non-fatty foods — Multiresidue methods for the gas chromatographic determination of pesticide residues*, consists of 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 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, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, 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 a 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 non-fatty foods.

Each method described 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 inter-laboratory 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 Florisil¹⁾ [2, 3, 4];
- method N: extraction with acetone, liquid–liquid partition with dichloromethane and clean-up with gel permeation and silica gel chromatography [5];
- method O: extraction with acetonitrile, liquid–liquid partition with light petroleum and clean-up on a Florisil[®] column [6];
- method P: extraction of organophosphorus compounds with ethyl acetate and, if necessary, clean-up with gel permeation chromatography [7].

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

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent

amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

EN 12393-2:1998, *Non-fatty foods — Multiresidue methods for the gas chromatographic determination of pesticide residues — Part 2: Methods for extraction and clean-up*.

EN 12393-3:1998, *Non-fatty foods — 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 always be clearly documented and demonstrated to give valid results.

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.

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.

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.