

Non-fatty food — Determination of N-methylcarbamate residues —

Part 1: HPLC-method with SPE clean-up

The European Standard EN 14185-1:2003 has the status of a
British Standard

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National foreword

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The UK participation in its preparation was entrusted to Technical Committee AW/-/3, Horizontal analysis, which has the responsibility to:

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- present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
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Part 1: HPLC-method with SPE clean-up

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méthylcarbamates - Partie 1: Méthode par CLHP avec
purification SPE

Fettarme Lebensmittel - Bestimmung von N-
Methylcarbamat-Rückständen - Teil 1: HPLC-Verfahren mit
Reinigung durch Festphasenextraktion

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Foreword

This document (EN 14185-1:2003) 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of an identical text or by endorsement, at the latest by October 2003, and conflicting national standards shall be withdrawn at the latest by October 2003.

Annexes A and B are informative.

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, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This European Standard specifies a high performance liquid chromatographic (HPLC) method for the determination of N-methylcarbamate pesticides in cereals, fruits and vegetables.

The method has been validated by collaborative study for carbaryl, carbofuran, methiocarb, methomyl, oxamyl and propoxur parent compounds and for methiocarb sulfoxide in green peppers and apples at levels between 0,08 mg/kg and 0,9 mg/kg.

No collaborative data are available for the performance of the method in the determination of other significant metabolites although it is known that the method will not work for oxamyl and methomyl oximes.

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 (including amendments).

EN ISO 3696, *Water for analytical laboratory use — Specification and test methods (ISO 3696:1987)*.

3 Principle

The sample is homogenized with acetone, dichloromethane and light petroleum and the homogenate is centrifuged to yield two layers of the supernatant. An aliquot portion of the upper layer is evaporated to dryness. Optionally, this extract may be cleaned up by solid phase extraction (SPE) using a cartridge packed with aminopropyl-bonded silica. In the extract solution, the N-methylcarbamates are determined by reversed-phase high performance liquid chromatography (HPLC) with post-column hydrolysis. The methylamine formed is allowed to react with o-phthalaldehyde and 2-mercaptoethanol and the derivatives are detected with a fluorescence detector. For further information on this method, see [1] to [4].

4 Reagents

4.1 General

Unless otherwise specified, use reagents of recognized analytical grade, preferably for HPLC and pesticide residue analysis, and distilled water for cleaning of glassware or water of at least grade 1 as defined in EN ISO 3696.

Label all standard containers with name and purity of the pesticides. For the full chemical names and structures, see ISO 1750.

4.2 Safety aspects associated with reagents

WARNING — Many pesticides are toxic by various routes of exposure, especially in concentrated form. When working with these pesticides consult safety data sheets of the manufacturer for information.

Vapours from some volatile solvents are toxic. Several of these solvents are readily absorbed through skin. Use an effective fume hood to remove vapours of these solvents as they are set free.

4.3 Acetone

4.4 Acetic acid

4.5 Dichloromethane

4.6 Light petroleum, boiling range 40 °C to 60 °C

4.7 Methanol

4.8 Acetonitrile

4.9 Solvent mixture

Acetonitrile (4.8) / acetic acid (4.4) 99,9 + 0,1 (V/V)

4.10 Water, purified by a LC-grade water purification system

4.11 Sodium acetate

4.12 o-Phthaldialdehyde

4.13 Sodium tetraborate, anhydrous

4.14 2-Mercaptoethanol

4.15 Trimethacarb

4.16 Mobile phase A

Acetonitrile (4.8) / water (4.10) 20 + 80 (V/V), containing 2,5 mmol/l sodium acetate (4.11). Prior to use, filter the mobile phase A with gentle suction through a membrane filter (5.10).

4.17 Mobile phase B

Methanol (4.7) / water (4.10) 20 + 80 (V/V), containing 2,5 mmol/l sodium acetate (4.11). Prior to use, filter the mobile phase B with gentle suction through a membrane filter (5.10).