
Krma - Dolol' evanje ostankov organofosfornih pesticidov - Plinska kromatografska metoda (ISO 14182:1999)

Animal feeding stuffs - Determination of residues of organophosphorus pesticides - Gas chromatographic method (ISO 14182:1999)

Futtermittel - Bestimmung von Organophosphorpestizid-Rückständen - Gaschromatographisches Verfahren (ISO 14182:1999)

Aliments des animaux - Détermination des résidus de pesticides organo-phosphorés - Méthode par chromatographie en phase gazeuse (ISO 14182:1999)

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EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN ISO 14182

December 1999

ICS 65.120

English version

Animal feeding stuffs - Determination of residues of
organophosphorus pesticides - Gas chromatographic method
(ISO 14182:1999)

Aliments des animaux - Détermination des résidus de
pesticides organo-phosphorés - Méthode par
chromatographie en phase gazeuse (ISO 14182:1999)

Futtermittel - Bestimmung von Organophosphorpestizid-
Rückständen - Gaschromatographisches Verfahren (ISO
14182:1999)

This European Standard was approved by CEN on 15 December 1999.

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 Central Secretariat 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 Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

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

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

Foreword

The text of the International Standard ISO 14182:1999 has been prepared by Technical Committee ISO/TC 34 "Agricultural food products" in collaboration with Technical Committee CEN/TC 327 "Animal feeding stuffs - Methods of sampling and analysis", the secretariat of which is held by NNI.

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 June 2000, and conflicting national standards shall be withdrawn at the latest by June 2000.

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.

NOTE FROM CEN/CS: The foreword is susceptible to be amended on reception of the German language version. The confirmed or amended foreword, and when appropriate, the normative annex ZA for the references to international publications with their relevant European publications will be circulated with the German version.

Endorsement notice

The text of the International Standard ISO 14182:1999 was approved by CEN as a European Standard without any modification.

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INTERNATIONAL STANDARD

**ISO
14182**

First edition
1999-12-15

Animal feeding stuffs — Determination of residues of organophosphorus pesticides — Gas chromatographic method

*Aliments des animaux — Détermination des résidus de pesticides organo-
phosphorés — Méthode par chromatographie en phase gazeuse*

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ISO 14182:1999(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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

International Standard ISO 14182 was prepared by Technical Committee ISO/TC 34, *Agricultural food products*, Subcommittee SC 10, *Animal feeding stuffs*.

Annexes A and B of this International Standard are for information only.

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Animal feeding stuffs — Determination of residues of organophosphorus pesticides — Gas chromatographic method

1 Scope

This International Standard specifies a gas chromatographic method for the determination of the residues of organophosphorus pesticides in animal feeding stuffs.

The method is applicable to animal feeding stuffs containing residues of one or more of the following organophosphorus pesticides: azinphos-ethyl, azinphos-methyl, bromophos, carbophenothion, chlorpyrifos, chlorpyrifos-methyl, diazinon, dimethoate, ethion, fonofos, malathion, methidathion, parathion, parathion-methyl, pirimiphos-ethyl and pirimiphos-methyl.

The lower limit of determination for these organophosphorus pesticides is 0,01 µg/g.

NOTE The method is probably equally applicable to other organophosphorus pesticides such as methacrifos and fenitrothion, but it has not been validated for these pesticides.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative documents referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 3696, *Water for analytical laboratory use — Specification and test methods*.

ISO 6498, *Animal feeding stuffs — Preparation of test samples*.

3 Principle

A test portion is extracted with acetone. The filtered extract is diluted with water and a saturated sodium chloride solution. The pesticides are partitioned in dichloromethane. The concentrated extract is purified on a chromatographic column of 10 % water-deactivated silica gel. Gas chromatographic determination is carried out with a phosphorus-selective detector or a mass-selective detector.

4 Reagents and materials

Use only reagents of recognized analytical grade and with a purity suitable for pesticide residue analysis.

Check the purity of the reagents by performing a blank test under the same conditions as used in the method. The chromatogram should not show any interfering impurity.

WARNING — Some of the organic solvents are suspected carcinogens. Use with care.

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4.1 Water, complying with at least grade 3 in accordance with ISO 3696.

4.2 Hexane

4.3 Acetone

4.4 Dichloromethane

4.5 Ethyl acetate

4.6 Silica gel, with a mass fraction of water of 10 %.

Activate silica gel 60, particle size 63 µm to 200 µm, at 130 °C overnight and cool in a desiccator. After cooling to room temperature, pour the silica gel into an air-tight glass container and add sufficient distilled water to bring the final mass fraction of water to 10 %. Shake the container mechanically or by hand vigorously for 30 s and allow to stand for 30 min with occasional shaking. After 30 min the silica gel is ready for use. It may not be stored for more than 6 h.

4.7 Eluting solvent, dichloromethane in hexane (50 % volume fraction).

Mix equal volumes of dichloromethane (4.4) and hexane(4.2).

4.8 Inert gas, e.g. nitrogen.

4.9 Sodium sulfate, anhydrous.

4.10 Sodium chloride saturated solution

4.11 Pesticide reference standards, as follows:

- azinphos-ethyl [S-(3,4-dihydro-4-oxobenzo[d][1,2,3]triazin-3-ylmethyl) O,O-diethyl phosphorodithioate];
- azinphos-methyl [S-(3,4-dihydro-4-oxobenzo[d][1,2,3]triazin-3-ylmethyl) O,O-dimethyl phosphorodithioate];
- bromophos [O-4-bromo-2,5-dichlorophenyl O,O-dimethyl phosphorothioate];
- carbophenothion [S-4-chlorophenylthiomethyl O,O-diethyl phosphorothioate];
- chlorpyrifos [O,O-diethyl O-3,5,6-trichloro-2-pyridyl phosphorothioate];
- chlorpyrifos-methyl [O,O-dimethyl O-3,5,6-trichloro-2-pyridyl phosphorothioate];
- diazinon [O,O-diethyl O-2-isopropyl-6-methylpyrimidin-4-yl phosphorothioate];
- dimethoate [O,O-dimethyl S-methylcarbamoylmethyl phosphorodithioate];
- ethion [O,O,O',O'-tetraethyl S,S'-methylene di(phosphorodithioate)];
- fonofos [O-ethyl S-phenyl ethylphosphonodithioate];
- malathion [diethyl (dimethoxythiophosphorylthio)succinate];
- methidathion [S-2,3-dihydro-5-methoxy-2-oxo-1,3,4-thiadiazol-3-ylmethyl O,O-dimethyl phosphorodithioate];
- parathion [O,O-diethyl O-4-nitrophenyl phosphorothioate];
- parathion-methyl [O,O-dimethyl O-4-nitrophenyl phosphorothioate];
- pirimiphos-ethyl [O-2-diethylamino-6-methylpyrimidin-4-yl O,O-diethyl phosphorothioate];
- pirimiphos-methyl [O-2-diethylamino-6-methylpyrimidin-4-yl O,O-dimethyl phosphorothioate];

NOTE The common names and the chemical names (between square brackets) according to IUPAC nomenclature, are in accordance with ISO 1750 [1].

4.12 Internal standard: tributylphosphate.

4.13 Pesticide standard solutions

4.13.1 Stock solutions, of concentration 1 000 µg/ml.

Prepare a stock solution of each pesticide reference standard (4.11) and of the internal standard (4.12) as follows.

Weigh, to the nearest 0,1 mg, a mass of a pesticide reference standard (4.11) or the internal standard (4.12) which will result in a solution with a content of reference standard or internal standard of 1 000 µg/ml. While weighing, observe the cleanness of the standard material. Transfer the weighed mass into a volumetric flask, dissolve in ethyl acetate (4.5) and dilute to volume with ethyl acetate.

These solutions are stable for 6 months when stored at 4 °C in the dark.

4.13.2 Intermediate solutions, of concentration 10 µg/ml.

Pipette 1 ml of each stock solution (4.13.1) into individual 100 ml volumetric flasks. Dilute to volume with ethyl acetate (4.5). The solutions are stable for 1 month when stored at 4 °C in the dark.

NOTE The stability of properly stored pesticide standards is very widely known. Investigations have shown that all neat pesticide standards tested are stable for 15 years when stored at –18 °C and that stock solutions of pesticide standards in toluene of 1 mg/kg are stable for at least 3 years when stored at –18 °C.

A recommended practice for longer storage is as follows. Transfer portions of the prepared standard solutions to amber vials with PTFE-lined screwcaps. Weigh the vials and store at –20 °C. When needed, remove a vial from the freezer, bring to room temperature and weigh. If accumulated loss in mass (due to evaporation) is 10 % or more of the prefrozen net mass, discard the vial. Weigh and refreeze stock standards and intermediate solutions that are in use for more than 1 month (usually in 25 ml vials). Otherwise, the prepared standard solutions (usually in 2 ml vials) may be stored at 4 °C and shall be discarded after 1 month.

4.13.3 Working solutions, of concentration 0,5 µg/ml.

Pipette 5 ml of each intermediate solution (4.13.2) into 100 ml volumetric flasks and dilute to volume with ethyl acetate (4.5). The solutions are stable for 1 month when stored at 4 °C in the dark (see 4.13.2).

4.14 Blank sample solutions, of the same type as the samples being analysed, but free of positives, resulting from previous determinations.

5 Apparatus

Before use, wash all glassware thoroughly with detergent free of interfering substances, rinse with water, then with acetone and dry.

Avoid the use of plastics containers and do not lubricate the stopcocks with grease, otherwise impurities could be introduced into the solvents.

Usual laboratory equipment and in particular the following.

5.1 Separating funnels, of capacities 500 ml and 1000 ml, with polytetrafluoroethylene (PTFE) stopcocks and stoppers.

5.2 Filtering flasks, of capacity 500 ml.

5.3 Büchner funnel, made of porcelain, with internal diameter 90 mm.

5.4 Graduated tubes, of capacity 10 ml, with polytetrafluoroethylene (PTFE) stoppers.