SLOVENSKI STANDARD
SIST EN ISO 14181:2001

01-februar-2001

Krma - Dolo̧evanje ostankov organoklornih pesticidov - Plinska kromatografska metoda (ISO 14181:2000)

Animal feeding stuffs - Determination of residues of organochlorine pesticides - Gas chromatographic method (ISO 14181:2000)

Futtermittel - Bestimmung des Gehalts an Organochlorpestizid-Rückständen - Gaschromatographisches Verfahren (ISO 14181:2000)

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Aliments des animaux - Détermination des résidus de pesticides organochlorés - Méthode par chromatographie en phase gazeuse (ISO 14181:2000)

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Ta slovenski standard je istoveten z: EN ISO 14181:2000

ICS:

65.120 Krmila Animal feeding stuffs

SIST EN ISO 14181:2001 en

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EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN ISO 14181

September 2000

ICS 65.120

English version

Animal feeding stuffs - Determination of residues of organochlorine pesticides - Gas chromatographic method (ISO 14181:2000)

Aliments des animaux - Détermination des résidus de pesticides organochlorés - Méthode par chromatographie en phase gazeuse (ISO 14181:2000)

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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Ref. No. EN ISO 14181:2000 E

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CORRECTED 2001-08-30

Foreword

The text of the International Standard ISO 14181:2000 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 NEN.

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of an identical text or by endorsement, at the latest by March 2001, and conflicting national standards shall be withdrawn at the latest by March 2001.

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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NOTE: Normative references to International Standards are listed in annex ZA (normative).

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Annex ZA (normative)
Normative references to international publications with their relevant European publications

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).

NOTE Where an International Publication has been modified by common modifications, indicated by (mod.), the relevant EN/HD applies.

Publication	Year	Title	EN	Year
ISO 3696	1987	Water for analytical laboratory use - Specification and test methods	EN ISO 3696	1995

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INTERNATIONAL STANDARD

ISO 14181

First edition
2000-09-15

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

Aliments des animaux — Détermination des résidus de pesticides organochlorés — Méthode par chromatographie en phase gazeuse

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Reference number
ISO 14181:2000(E)

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SIST EN ISO 14181:2001

Contents

Page

Foreword iv

1 Scope 1

2 Normative references 1

3 Principle 1

4 Reagents and materials 1

5 Apparatus 3
5.1 Grinder 5

6 Sampling 5

7 Preparation of test sample 5

8 Procedure 5
8.1 General 5
8.2 Test portion 5
8.3 Extraction 5
8.4 Column clean-up 6
8.5 Gas chromatography 6

9 Expression of results 7
9.1 Calculation 7
9.2 Recovery 8

10 Confirmation of identity 8

11 Precision 8
11.1 Interlaboratory test 8
11.2 Repeatability 8
11.3 Reproducibility 8

12 Test report 9

Annex A (informative) **Examples of GC operating conditions for organochlorine pesticides** 10

Annex B (informative) **Results of interlaboratory test** 11

Bibliography 20

iTeh STANDARD PREVIEW
(standards.iteh.ai)
SIST EN ISO 14181:2001
https://standards.iteh.ai/catalog/standards/sist/cd16ddb7-dac2-41e1-bdee-0ec06c6382ce/sist-en-iso-14181-2001

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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 14181 was prepared by Technical Committee ISO/TC 34, *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 organochlorine pesticides — Gas chromatographic method

1 Scope

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

The method is applicable to animal feeding stuffs containing residues of one or more of the following organochlorine pesticides and some of their isomers and degradation products: aldrin, *op'*-DDE, *pp'*-DDE, *op'*-DDT, *pp'*-DDT, dieldrin, endosulfan, endrin, HCB, α-HCH (α-BHC), β-HCH (β-BHC), γ-HCH (γ-BHC), δ-HCH (δ-BHC), heptachlor, heptachlor epoxide, *op'*-TDE (*op'*-DDD), *pp'*-TDE (*pp'*-DDD) and methoxychlor.

The lower determination limit for these organochlorine pesticides is 0,005 μg/g. However, the lower determination limit is 0,01 μg/g for *op'*-DDT and *pp'*-DDT, and 0,05 μg/g for methoxychlor.

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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 edition of the normative document 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.

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ISO 3696:1987, *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 saturated sodium chloride solution. The pesticides are partitioned into dichloromethane. The concentrated extract is purified on a chromatographic column of 10 % water-deactivated silica gel. The pesticide residues are determined by gas chromatography using an electron capture 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.

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

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4.2 Hexane.

4.3 Acetone.

4.4 Dichloromethane.

4.5 Silica gel 60, with a mass fraction of water of 10 %.

Activate silica gel 60, particle size 63 μm to 200 μm, at 130 °C overnight, then cool in a desiccator. After cooling to room temperature, pour the silica gel into an airtight 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 should not be stored for more than 6 h.

4.6 Eluting solvent: dichloromethane in hexane, 20 % (by volume).

Mix 1 volume of dichloromethane with 4 volumes of hexane.

4.7 Inert gas, for example nitrogen.

4.8 Sodium sulfate, anhydrous.

4.9 Sodium chloride, saturated solution.

4.10 Pesticide reference standards, as follows:

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— aldrin [(1*R*,4*S*,4*aS*,5*S*,8*R*,8*aR*)-1,2,3,4,10,10-hexachloro-1,4,4*a*,5,8,8*a*-hexahydro-1,4:5,8-dimethano-naphthalene];

— *op'*-DDE [*o,p'*-(1,1-dichloro-2,2-bis(4-chlorophenyl) ethylene)];

— *pp'*-DDE [*p,p'*-(1,1-dichloro-2,2-bis(4-chlorophenyl) ethylene)];

— *op'*-DDT [*o,p'*-(1,1,1-trichloro-2,2-bis(4-chlorophenyl) ethane)];

— *pp'*-DDT [*p,p'*-(1,1,1-trichloro-2,2-bis(4-chlorophenyl) ethane)];

— dieldrin [(1*R*,4*S*,4*aS*,5*R*,6*R*,7*S*,8*S*,8*aR*)-1,2,3,4,10,10-hexachloro-1,4,4*a*,5,6,7,8,8*a*-octahydro-6,7epoxy-1,4:5,8-dimethanonaphthalene];

— endosulfan (6,7,8,9,10,10-hexachloro-1,5,5*a*,6,9,9*a*-hexahydro-6,9-methano-2,4,3-benzodioxathiepin 3-oxide);

— endrin [(1*R*,4*S*,4*aS*,5*S*,6*S*,7*R*,8*R*,8*aR*)-1,2,3,4,10,10-hexachloro-1,4,4*a*,5,6,7,8,8*a*-octahydro-6,7-epoxy-1,4:5,8-dimethanonaphthalene];

— HCB (hexachlorobenzene);

— α-HCH (α-BHC) (α-1,2,3,4,5,6-hexachlorocyclohexane);

— β-HCH (β-BHC) (β-1,2,3,4,5,6-hexachlorocyclohexane);

— γ-HCH (γ-BHC; lindane) (γ-1,2,3,4,5,6-hexachlorocyclohexane);

— δ-HCH (δ-BHC) (δ-1,2,3,4,5,6-hexachlorocyclohexane);

— heptachlor (1,4,5,6,7,8,8-heptachloro-3*a*,4,7,7*a*-tetrahydro-4,7-methanoindene);

— heptachlor epoxide (heptachloroepoxy-tetrahydromethanoindene);

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