SLOVENSKI STANDARD
SIST EN 15742:2009

01-maj-2009

Krma - Določ evanje OC-pesticidov in PCB z GC/ECD

Animal feeding stuffs - Determination of OC-pesticides and PCB’s by GC/ECD

Futtermittel - Bestimmung der OC-Pestizide und PCB's mittels GC/ECD-Verfahren

Aliments des animaux - Détermination des pesticides organochlorés (OC) et des polychlorobiphényles (PCB) par GC/ECD

Ta slovenski standard je istoveten z: **EN 15742:2009**

ICS:

65.120	Krmila	Animal feeding stuffs

SIST EN 15742:2009 **en,fr,de**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 15742

February 2009

ICS 65.120

English Version

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COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Ref. No. EN 15742:2009: E

Contents

Page

Foreword

This document (EN 15742:2009) has been prepared by Technical Committee CEN/TC 327 “Animal feeding stuffs”, 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of an identical text or by endorsement, at the latest by August 2009, and conflicting national standards shall be withdrawn at the latest by August 2009.

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1 Scope

This European Standard specifies a gas chromatographic method with electron capture detection (ECD) for the determination of organochlorine pesticides (OC's) and polychlorinated biphenyls (PCBs) in animal feeding stuffs.

The method is applicable to animal feeding stuffs with a water content up to about 20 wt% and oil/fatty samples containing residues of one or more of the following OC's, PCBs, toxaphene and some of their isomers and degradation products:

— Aldrin;

— Dieldrin;

— Chlorocamphene (Toxaphene);

— Chlordane (= sum of Chlordane isomers and Oxychlordane);

— DDT (= sum of isomers op'-DDT, pp'-DDT, pp'-TDE (pp'-DDD), and pp'-DDE);

— Endosulfan (sum of α-/β-isomers and Endosulfan-sulphate);

— Endrin;

— Heptachlor (= sum of Heptachlor and β-Heptachlorepoxide);

— Hexachlorobenzene (HCB);

— Hexachlorocyclohexane isomers α-HCH (α-BHC), β-HCH (β-BHC), γ-HCH (γ-BHC or lindane);

— PCB 28, 52, 101, 138, 153 and 180 ("Indicator PCBs") and PCB 198, 209.

The limit of quantification for the mentioned organochlorine pesticides and PCBs is 5 ng/g in general. However, 10 ng/g applies for Heptachlor, Aldrin, Endrin, Dieldrin, and Endosulfan (α-, β- and sulphate). Individual laboratories are responsible to ensure that the equipment they used will achieve these limits of quantifications.

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.

ISO 6498, *Animal feeding stuffs – Preparation of test samples*

3 Terms and definitions

For the purpose of this document, the following terms and definitions apply.

3.1
limit of detection
smallest measured content, from which it is possible to deduce the presence of the analyte with reasonable statistical certainty

NOTE The limit of detection is numerically equal to three times the standard deviation of the mean of blank determinations (n>10).

3.2
limit of quantification
lowest content of the analyte which can be measured with reasonable statistical certainty

NOTE If both accuracy and precision are constant over a concentration range around the limit of detection, then the limit of quantification is numerically equal to 6 times the standard deviation of the mean of blank determinations (n>10).

3.3
feed additives
substances are feed additives when they comply with the definition of feed additives given in the Regulation 1831/2003

4 Principle

A test portion of animal feeding stuff is fortified with internal standard (PCB 198), and is extracted with ethylacetate. The extract is concentrated and subsequently purified by:

— Gel permeation chromatography (GPC), with cyclohexane/ethylacetate as eluting solvent

— chromatography on partially deactivated silica gel.

The collected fraction containing the compounds of interest is concentrated and re-dissolved in a solution containing another internal standard (PCB 209) as a reference standard. After cleanup the analytes are measured using GC-ECD. Indentification is done on the basis of comparing retention times on capillary columns of different polarity. Quantification is done using the internal standard method.

5 Reagents and materials

Use only reagents of recognized analytical grade and with a purity suitable for OC and PCB 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 at the retention time of compounds of interest.

WARNING — The use of this European Standard can involve hazardous materials, operations and equipment. This standard does not purport to address all the safety problems associated with its use. It is the responsibility of the user of this European Standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

5.1 Cyclohexane

5.2 Ethylacetate

5.3 Hexane

5.4 Dichloromethane

5.5 Iso-octane

5.6 Toluene

5.7 Hexane/toluene = 3+7, parts by volume

Mix 30 ml of hexan (5.3) with 70 ml of toluene (5.6) thoroughly. Store at room temperature in a tightly closed glass bottle.

5.8 Sodium Sulphate, anhydrous

Heated at 160°C during at least 24 h.

5.9 Ethylacetate/Cyclohexane = 1+1, parts by volume

Mix 500 ml of ethylacetate (5.2) with 500 ml of cyclohexane (5.1) thoroughly. Store at room temperature in a tightly closed glass bottle.

5.10 Silica gel, deactivated with 3,5% water

Heat silica gel 60 (63µm to 200µm = 70 mesh to 230 mesh), at 130ºC for at least 5 h, allow to cool in a desiccator, and store in a tightly stopped container in the desiccator. To 96,5 g dried silica gel in a 300 ml Erlenmeyer flask with a ground joint, add 3,5 ml water dropwise from a burette, with continuous swirling. Immediately stopper the flask with a ground stopper and shake vigorously for 5 min until all lumps have disappeared. Next shake for 2 h on a mechanical shaker, and then store in a tightly stoppered container. Deactivated silica gel is tenable during approximately 2 weeks if carefully stored.

5.11 Internal standard (PCB 198)

5.12 Internal Standard (PCB 209)

5.13 OC-pesticide reference standards

Each with a purity not less than 99%.

Aldrin
((1R,4S,4aS,5S,8R,8aR)-1,2,3,4,10,10-hexachloro-1,4,4a,5,8,8a-hexahydro-1,4:5,8-dimethanonaphthalene)
CAS Number: 309-00-2

Dieldrin
((1R,4S,4aS,5R,6R,7S,8S,8aR)-1,2,3,4,10,10-hexachloro-1,4,4a,5,6,7,8,8a-octahydro-6,7epoxy-1,4:5,8-dimethanonaphthalene)
CAS Number: 60-57-1

Chlordane
(1,2,4,5,6,7,8,8-octachloro-2,3,3a,4,7,7a-hexahydro-4,7-ethano-1*H*-indene); α and β isomer
CAS Numbers: 5103-71-9 and 5103-74-2

Oxychlordane
(4,7-Methanoindan, 1,2,4,5,6,7,8,8-octachloro-2,3-epoxy-3a,4,7,7a-tetrahydro-, exo,endo-)
CAS Number: 27304-13-8

op'-DDT
[o,p'-(1,1,1-trichloro-2,2-bis(4-chlorophenyl)ethane)]
CAS Number: 789-02-6

pp'-DDT
[p,p'-(1,1,1-trichloro-2,2-bis(4-chlorophenyl) ethane)]
CAS Number: 50-29-3

pp'-TDE
(p,p'-DDD) [p,p'-1,1-dichloro-2,2-bis(4-chlorophenyl) ethane]
CAS Number: 72-54-8

pp'-DDE
[p,p'-(1,1-dichloro-2,2-bis(4-chlorophenyl) ethylene)]
CAS Number: 72-55-9

Endosulfan
(6,7,8,9,10,10-hexachloro-1,5,5a,6,9,9a-hexahydro-6,9-methano-2,4,3-benzodioxathiepin 3-oxide)
two stereoisomers, α, (I), CAS Number: 959-98-8 and β, (II), CAS Number: 33213-65-9.

Endosulfan-sulphate;
CAS Number: 1031-07-8

Endrin
[(1R,4S,4aS,5S,6S,7R,8R,8aR)-1,2,3,4,10,10-hexachloro-1,4,4a,5,6,7,8,8a-octahydro-6,7-epoxy-1,4:5,8-dimethanonaphthalene]
CAS Number: 72-20-8

Heptachlor
(1,4,5,6,7,8,8-heptachloro-3a,4,7,7a-tetrahydro-4,7-methanoindene)
CAS Number: 76-44-8

β-Heptachlorepoxide
(1,4,5,6,7,8,8-heptachloro-3a,4,7,7a-tetrahydro-4,7-methanoindene(exo))
CAS Number: 1024-57-3

HCB
(hexachlorobenzene)
CAS Number: 118-74-1

α-HCH (α-BHC)
(α-1,2,3,4,5,6-hexachlorocyclohexane)
CAS Number: 319-84-6

β-HCH (β-BHC)
(β-1,2,3,4,5,6-hexachlorocyclohexane)
CAS Number: 319-85-7

γ-HCH (γ-BHC; lindane)
(1,2,3,4,5,6-hexachlorocyclohexane)
CAS Number: 58-89-9

Or a Certified Mixture at a concentration of 10 µg/ml.

5.14 PCBs reference standards

Each with a purity not less than 99%.

PCB 28 (2,4,4' trichlorobiphenyl) ; CAS Number: 7012-37-5

PCB 52 (2,2',5,5' tetrachlorobiphenyl) ; CAS Number: 35693-99-3

PCB 101 (2,2',4,5,5' pentachlorobiphenyl) ; CAS Number: 37680-73-2

PCB 138 (2,2',3',4,4',5 hexachlorobiphenyl) ; CAS Number: 35065-28-2

PCB 153 (2,2',4,4',5,5' hexachlorobiphenyl) ; CAS Number: 35065-27-1

PCB 180 (2,2',3,4,4',5,5' heptachlorobiphenyl) ; CAS Number: 35065-29-3

Or a Certified Mixture at a concentration of 10 µg/ml.

5.15 Chlorocamphene (Toxaphene)

Technical mixture.

5.16 Stock solutions, 100 µg/ml

Weigh 5 -10 mg (± 0,01 mg) of each compound (5.11, 5.12, 5.13, 5.14 and 5.15) in seperate brown medicine glass bottles of 100 ml and add iso-octane (5.5) to achieve a concentration of 100 µg/ml. Store the solutions in a refrigerator at 4°C (± 3°C). The solution is tenable under these conditions during at least 5 years if the weight is carefully controled.

Dissolve ß-HCH in 10 ml toluene (5.6), to achieve complete solvability and dilute further with iso-octane (5.5) to achieve a concentration of 100 µg/ml.

5.17 Mixed stock solutions

5.17.1 Mixed stock solution OC (without Endosulfan and Toxaphene)

Pipet of each OC-stock solution (5.16) the indicated volume (Table 1) in a volumetric flask of 100 ml. Fill up to 100 ml with iso-octane (5.5) and mix. The achieved concentration is given in Table 1. Transport this solution to a brown medicine glass bottle of 100 ml and store it in a refrigerator at 4°C (± 3°C). The solution is tenable under these conditions during at least 5 years if the weight is carefully controled.

5.17.2 Mixed stock solution Endosulfan

Pipet of each Endosulfan-stock solution (5.16) the indicated volume (Table 1) in a volumetric flask of 100 ml. Fill up to 100 ml with iso-octane (5.5) and mix. The achieved concentration is given in Table 1. Transport this solution to a brown medicine glass bottle of 100 ml and store it in a refrigerator at 4°C (± 3°C). The solution is tenable under these conditions during at least 5 years if the weight is carefully controled.

Table 1 — Concentration of OCs in Mixed stock solution (5.17) and mixed standard solution (5.18)

Compound	Pipet volume (ml)	Mixed stock solution (5.17 1&2) (µg/ml)	Mixed standard solution (5.18.1 1&2) (µg/ml)
Aldrin	2,0	2,0	0,10
Dieldrin	2,0	2,0	0,10
α-Chlordane	1,0	1,0	0,05
γ-Chlordane	1,0	1,0	0,05
Oxychlordane	1,0	1,0	0,05
o,p'-DDT	4,0	4,0	0,20
p,p'-DDT	4,0	4,0	0,20
p,p'-TDE	4,0	4,0	0,20
p,p'-DDE	4,0	4,0	0,20
α-Endosulfan	2,0	2,0	0,10
β-Endosulfan	2,0	2,0	0,10
Endosulfan-sulphate	1,0	1,0	0,05
Endrin	1,0	1,0	0,05
Heptachlor	2,0	2,0	0,10
Heptachlor epoxide	2,0	2,0	0,10
HCB	1,0	1,0	0,05
α-HCH	2,0	2,0	0,10
ß-HCH	1,0	1,0	0,05
γ-HCH (Lindane)	1,0	1,0	0,05