SLOVENSKI STANDARD
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Krma: metode vzorčenja in analize - Določevanje OC-pesticidov z GC/ECD

Animal feeding stuffs: Methods of sampling and analysis - Determination of OCPs by GC/ECD

Futtermittel - Probenahme- und Untersuchungsverfahren - Bestimmung von OCPs mittels GC/ECD

Aliments des animaux: Méthodes d'échantillonnage et d'analyse

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Animal feeding stuffs: Methods of sampling and analysis - Determination of OCPs by GC/ECD

Aliments des animaux: Méthodes d'échantillonnage et d'analyse

Futtermittel - Probenahme- und Untersuchungsverfahren - Bestimmung von OC-Pesticiden mittels GC/ECD

This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 327.

If this draft becomes a European Standard, 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.

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Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

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

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European foreword

This document (prEN 15742:2018) has been prepared by Technical Committee CEN/TC 327 “Animal feeding stuffs: Methods of sampling and analysis”, the secretariat of which is held by NEN.

This document is currently submitted to the CEN Enquiry.

This document will supersede EN 15742:2009.

In comparison with the previous edition, the following technical modifications have been made:

The analysis of polychlorinated biphenyls (PCBs) has been removed from this standard as current legislation on maximum limits requires sensitivity that cannot be provided by GC/ECD. Additionally, editorial changes were made.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

Introduction

This document was developed in response to Directive 2002/32/EC of the European Parliament and the Council of 7 May 2002 on undesirable substances in animal feed.

The previous edition of this document (EN 15742:2009) was fully validated by means of a collaborative study for aldrin, dieldrin, endrin, p,p'-DDT, o,p'-DDT, p,p'-TDE, pp-DDE, alpha-endosulfan, beta-endosulfan, HCB, alpha-HCH, beta-HCH and gamma-HCH. Attempts in the framework of the third Mandate from the European Commission to CEN/TC 327 to perform additional validation of the method through a full collaborative study (2017) for photo heptachlor, cis/trans nonachlor and keto-endrin were unsuccessful as no more than three laboratories volunteered to send in results [1].

WARNING — The use of this document 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 document to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This document specifies a gas chromatographic method with electron capture detection (ECD) for the determination of organochlorine pesticides (OCP's) in animal feeding stuffs.

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

— aldrin;

— dieldrin;

— chlordane (as the sum of chlordane isomers and oxychlordane);

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

— endosulfan (as the sum of α-/β-isomers and endosulfan-sulphate);

— endrin (sum of endrin and delta-keto-endrin);

— heptachlor (as the sum of heptachlor and heptachlor epoxide);

— hexachlorobenzene (HCB);

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

— photo heptachlor;

— cis- and trans-nonachlor;

A limit of quantification (LOQ) for the mentioned OCPs of 5 ng/g is intended to be obtained. However, 10 ng/g applies for heptachlor, aldrin, endrin, dieldrin, and endosulfan (α-/β– and sulphate). Individual laboratories are responsible for ensuring that the equipment that they use, achieves these limits of quantifications. The LOQs apply to the individual OCPs.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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.

EN ISO 6498, *Animal feeding stuffs - Guidelines for sample preparation (ISO 6498)*

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at http://www.electropedia.org/
- ISO Online browsing platform: available at http://www.iso.org/obp

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 1 to entry: The limit of detection is numerically equal to three times the standard deviation of the mean of blanc determinations (n > 10).

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

Note 1 to entry 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 blanc determinations (n > 10).

3.3
feed additives
substances are feed additives when they comply with the definition of feed additives given in the Regulation (EC) No.1831/2003 of the European Parliament and of the Council on additives for use in animal nutrition

4 Principle

In order to check for the presence of OCPs, a test portion of animal feeding stuff is fortified with internal standard (PCB 198), and is extracted with ethyl acetate. The extract is concentrated and subsequently purified by:

— gel permeation chromatography (GPC), with cyclohexane or ethyl acetate 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 clean up, the analytes are measured using GC-ECD. Identification 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

5.1 General

Use only reagents of recognized analytical grade and with a purity suitable for OCP and PCB residue analysis. Check the purity of the reagents by performing a blanc 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.

5.2 Chemicals

5.2.1 Cyclohexane

5.2.2 Ethyl acetate

5.2.3 Hexane

5.2.4 Dichloromethane

5.2.5 Iso-octane

5.2.6 Toluene

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

Mix 30 ml of hexane (5.2.3) with 70 ml of toluene (5.2.6) thoroughly. Store at room temperature in a tightly closed glass bottle.

5.2.8 Sodium sulphate, anhydrous

Heated to 160-200 °C during at least 24 h.

5.2.9 Ethyl acetate/cyclohexane = 1+1, parts by volume

Mix 500 ml of ethyl acetate (5.2.2) with 500 ml of cyclohexane (5.2.1) thoroughly. Store at room temperature in a tightly closed glass bottle.

5.2.10 Silica gel, deactivated with 3,5 % water

Heat silica gel 60 (63 µm to 200 µm = 70 mesh to 230 mesh), at 130 °C for at least five hours, allow to cool in a desiccator, and store in a tightly stopped container in the desiccator.

Add 3,5 ml water dropwise from a burette, with continuous swirling, to 96,5 g dried silica gel in a 300 ml Erlenmeyer flask with a ground joint.

Immediately stopper the flask with a ground stopper and shake vigorously for five minutes until all lumps have disappeared.

Next, shake for two hours on a mechanical shaker, and then store in a tightly stoppered container.

Deactivated silica gel is tenable during approximately two weeks if carefully stored.

5.2.11 Internal standard (PCB 198)

5.2.12 Internal Standard (PCB 209)

5.2.13 OCP 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.

Delta-keto-endrin

CAS Number 53494-70-5.