

Animal feeding stuffs: Methods of sampling and analysis  
- Determination of OCPs and PCBs by GC-MS

## EESTI STANDARDI EESSÕNA

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ICS 65.120

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English Version

## Animal feeding stuffs: Methods of sampling and analysis - Determination of OCPs and PCBs by GC/MS

Aliments des animaux : Méthodes d'échantillonnage et  
d'analyse - Détermination des pesticides organochlorés  
(POC) et des polychlorobiphényles (PCB) par GC/MS

Futtermittel: Probenahme- und  
Untersuchungsverfahren - Bestimmung von OCP und  
PCB mittels GC/MS

This European Standard was approved by CEN on 6 January 2020.

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## European foreword

This document (EN 15741:2020) 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 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of an identical text or by endorsement, at the latest by September 2020, and conflicting national standards shall be withdrawn at the latest by September 2020.

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

This document supersedes EN 15741:2009.

In comparison with the previous edition, the following technical modification has been made:

Regarding non dioxin-like PCBs (ndl-PCBs), this document contains two approaches that can be followed. Method 1 concerns the original extraction and clean-up methods of the previous edition of this standard, but combined with more sensitive detection approaches. In method 2, the extraction and clean-up methods have been modified in order to increase the test portion. The detection of method 2 concerns the original detection method of the previous edition of this standard.

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

According to the CEN-CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom

## Introduction

**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 European Standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

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## 1 Scope

This document specifies a gas chromatographic mass spectrometric (GC-MS) method for the determination of organochlorine pesticides (OCPs) and polychlorinated biphenyls (PCBs) in compound feeds and feed materials (oils and fats, fish meal).

The method is applicable to compound feeds consisting of less than 20 % water by mass and oil/fatty samples containing residues of one or more of the following OCPs and PCBs 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 o,p'-DDT, p,p'-DDT, p,p'-TDE (p,p'-DDD), and p,p'-DDE;
- endosulfan, as the sum of  $\alpha$ -/ $\beta$ -isomers and endosulfan-sulphate;
- endrin, as the sum of endrin and delta-keto-endrin;
- heptachlor, as the sum of heptachlor and heptachlor epoxide;
- hexachlorobenzene (HCB);
- hexachlorocyclohexane isomers  $\alpha$ -HCH ( $\alpha$ -BHC),  $\beta$ -HCH ( $\beta$ -BHC),  $\gamma$ -HCH ( $\gamma$ -BHC or lindane);
- photo heptachlor;
- *cis*- and *trans*-nonachlor;
- non dioxin-like PCBs (ndl-PCBs), as the sum of PCB 28, 52, 101, 138, 153 and 180.

The method has been fully validated by a collaborative trial for the substances and corresponding ranges ( $\mu\text{g/kg}$ ) noted in Table 1.

**Table 1 — Residue compound and range of (µg/kg) collaborative trial**

Compound	Range (µg/kg)
all ndl-PCBs	0,7 to 39
aldrin	10 to 34
dieldrin	12 to 97
endrin	13 to 88
<i>cis</i> -chlordane <sup>a</sup>	16 to 24 <sup>a</sup>
<i>trans</i> -chlordane	7 to 25
p,p'-DDT <sup>b</sup>	19 to 200 <sup>b</sup>
o,p'-DDT	8 to 87
pp'-TDE <sup>c</sup>	9 to 103 <sup>c</sup>
pp'-DDE	21 to 264
α-endosulfan	15 to 165
β-endosulfan	26 to 331
endosulfan sulphate <sup>d</sup>	29 to 61 <sup>d</sup>
heptachlor	15 to 365
heptachlor epoxide	15 to 382
HCB <sup>e</sup>	8 to 170 <sup>e</sup>
α-HCH	21 to 247
β-HCH	6 to 84
γ-HCH <sup>f</sup>	17 to 186 <sup>f</sup>
NOTE The following information is to be taken into consideration: a) <i>Cis</i> -chlordane has not been fully validated for chicken feed, pig feed and fish oil. b) p,p'-DDT has not been fully validated for pig feed and vegetable oil. c) pp'-TDE has not been fully validated for pig feed and fish meal. d) Endosulfan sulphate has not been fully validated for pig feed and vegetable oil. e) HCB has not been fully validated for fish oil. f) γ-HCH has not been fully validated for fish oil.	

The method has not been fully validated for oxychlordane, endrin ketone, *cis*- and *trans*-nonachlor and photo heptachlor in all matrices.

For those matrix-analyte combinations where the validation data were regarded insufficient, the results obtained with this method can only be regarded as screening results, unless the laboratory performs an in-house validation to show that satisfactory results can be obtained.

The method is not applicable to chlorocamphene (toxaphene), a complex mixture of polychlorinated camphenes. Chlorocamphene has a very distinctive chromatographic profile and is easily recognizable by GC/ECD. Positive identification of the toxaphene isomers can be performed by negative chemical ionization mass spectrometry (NCI-MS), electron impact tandem mass spectrometry (EI MS × MS) or electron impact high resolution mass spectrometry (EI-HRMS), which is not within the scope of this method.



A limit of quantification (LOQ) for the mentioned organochlorine pesticides of 6 to 29 µg/kg should normally be obtained (see Table 1). For the ndl-PCBs an LOQ of 0,5 to 1,0 µg/kg should be obtained. The LOQs mentioned apply to the individual compounds (i.e. not the sum of two or more compounds). Individual laboratories are responsible for ensuring that the equipment that they used will achieve these LOQs. On customers' demand the standard may be applied to solely the analysis of PCBs or 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 3 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 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 blank determinations ( $n > 10$ ).

### 3.3

#### feed additives

substances that are added to animal nutrition

Note 1 to entry: see Regulation (EC) No.1831/2003 of the European Parliament and of the Council on additives for use in animal nutrition for more information.

## 4 Principle

### 4.1 General

In order to check for the presence of organochlorine pesticides (OCPs), a test portion of animal feeding stuff is fortified with internal standard ( $^{13}\text{C}_{12}$ -PCB mix) and extracted with ethyl acetate. The extract is concentrated and subsequently purified by:

- gel permeation chromatography (GPC), with a mixture of cyclohexane/ethyl acetate as eluting solvent;