

Animal feeding stuffs: Methods of sampling and analysis
- Determination of dioxins and dioxin-like PCBs by
GC/HRMS and of indicator PCBs by GC/HRMS

EESTI STANDARDI EESSÕNA

NATIONAL FOREWORD

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English Version

Animal feeding stuffs: Methods of sampling and analysis - Determination of dioxins and dioxin-like PCBs by GC/HRMS and of indicator PCBs by GC/HRMS

Aliments des animaux : Méthodes d'échantillonnage et
d'analyse - Dosage des dioxines, des PCB de type
dioxine et des PCB indicateurs par GC/HRMS

Futtermittel - Probenahme- und
Untersuchungsverfahren - Bestimmung von Dioxinen
und dioxin-ähnlichen PCBs mittels GC/HRMS und von
Indikator-PCBs mittels GC/HRMS

This European Standard was approved by CEN on 11 November 2019.

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

CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

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European foreword

This document (EN 16215:2020) 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of an identical text or by endorsement, at the latest by July 2020, and conflicting national standards shall be withdrawn at the latest by July 2020.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 16215:2012.

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

- incorrect technical details have been corrected,
- references have been updated,
- inconsistencies were removed, and
- editorial adaptations have been made.

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

The previous version of this document was developed in response to Directive 2002/32/EC of the European Parliament and of the Council of 7 May 2002 on undesirable substances in animal feed. The document provides analytical laboratories active in the field of feed analysis with guidance for the analysis of dioxins and PCBs and meets criteria as set in Commission Regulation (EC) No 152/2009 of 27 January 2009 laying down the methods of sampling and analysis for the official control of feed.

In this updated version, obvious mistakes were corrected.

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.

1 Scope

This document is applicable to the determination of polychlorinated dibenzo-p-dioxins (PCDDs), polychlorinated dibenzofurans (PCDFs), (together termed 'dioxins' (PCDD/Fs)) and dioxin-like PCBs and non-dioxin-like PCBs (dl-PCBs and ndl-PCBs) in animal feeding stuffs. Collaborative studies have been carried out. The method is suitable for the determination of dioxins, dl-PCBs and ndl-PCBs at the appropriate MRL in compound feed and ingredients e.g. oil, mineral clay. The method is applicable to samples containing trace level amounts of one or more dioxins, dioxin-like PCBs and non-dioxin-like PCBs. The limit of quantification (LOQ) is

- 0,05 pg/g (OCDD/F = 0,1 pg/g) for the relevant individual congeners of dioxins/furans,
- 0,05 pg/g for non-ortho PCBs,
- 10 pg/g for mono-ortho PCBs, and
- 100 pg/g for non-dioxin-like-PCBs.

For determination of dioxins and dioxin-like PCBs, the procedure can be used as confirmatory method as defined by Commission Regulation (EC) No 152/2009 for dioxins and dl-PCB in feed [1]. Confirmatory methods as described in this standard are high-resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS) methods. If only the analysis of non-dioxin-like PCBs is required, a GC-LRMS method can be used (e.g. EN 15741 [2]) provided that appropriate analytical performance criteria are met in the relevant range for the matrix of interest.

This document is split into four modules. Each module describes a part of the whole procedure (see Figure 1 and Figure 2) to be followed:

- a) Module A: Description of standards which might be used;
- b) Module B: Description of extraction procedures;
- c) Module C: Description of clean-up procedures;
- d) Module D: GC/HRMS determination.

Each module describes a part of the whole method as well as, when applicable, alternatives which should be equivalent. Each module has to be regarded as an example. Combining modules and/or alternatives gives a highly flexible, "performance based" procedure. It is permitted to modify the method if all performance criteria laid down in Commission Regulation (EC) No 152/2009 [1] are met.

Any deviation of the described method, combination of modules needs to be recorded as part of the QA/QC procedures of accredited laboratories and should be available on request.

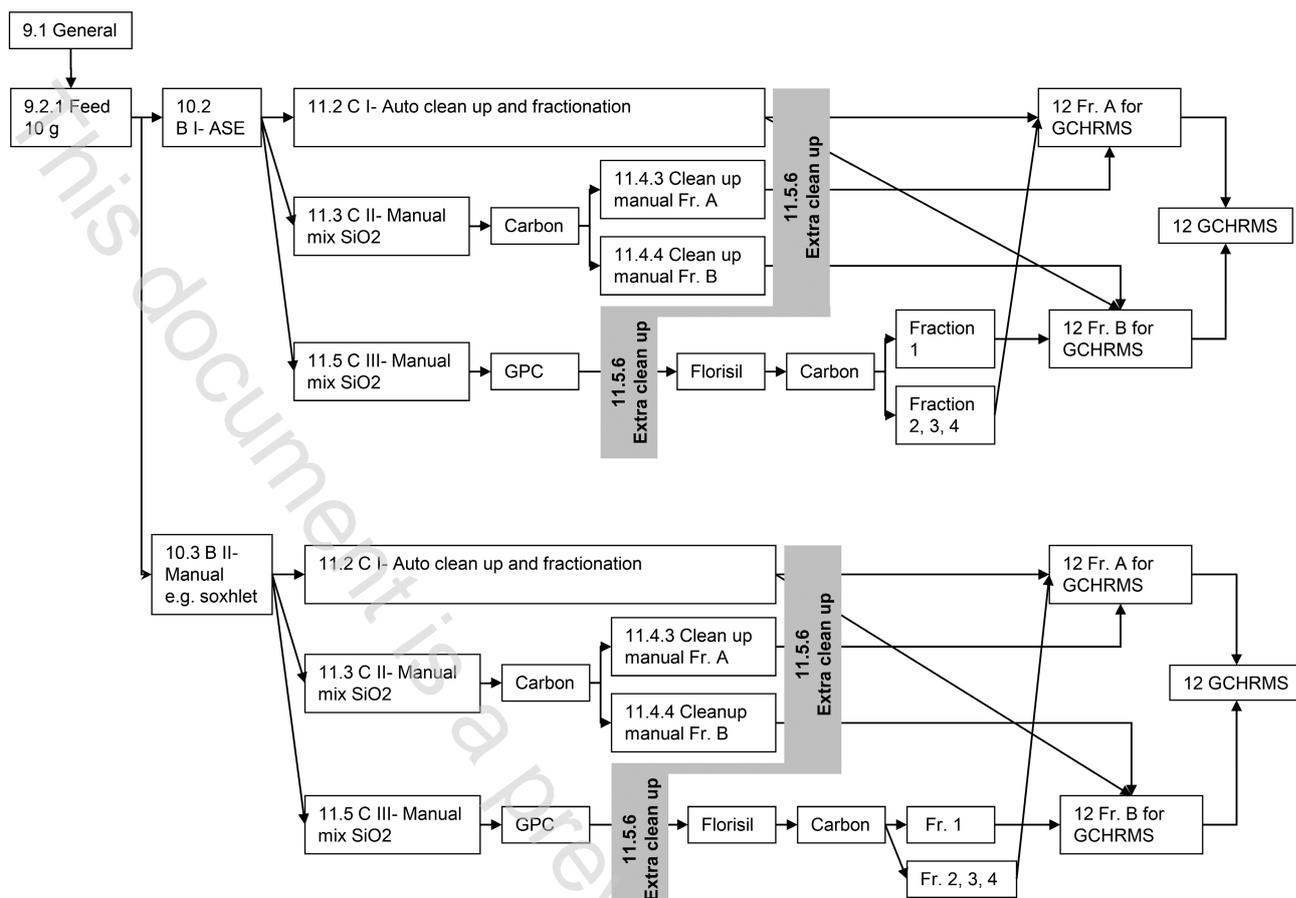


Figure 1 — Flow scheme for the determination of dioxins, dl-PCBs and non-dioxin-like-PCBs in feed

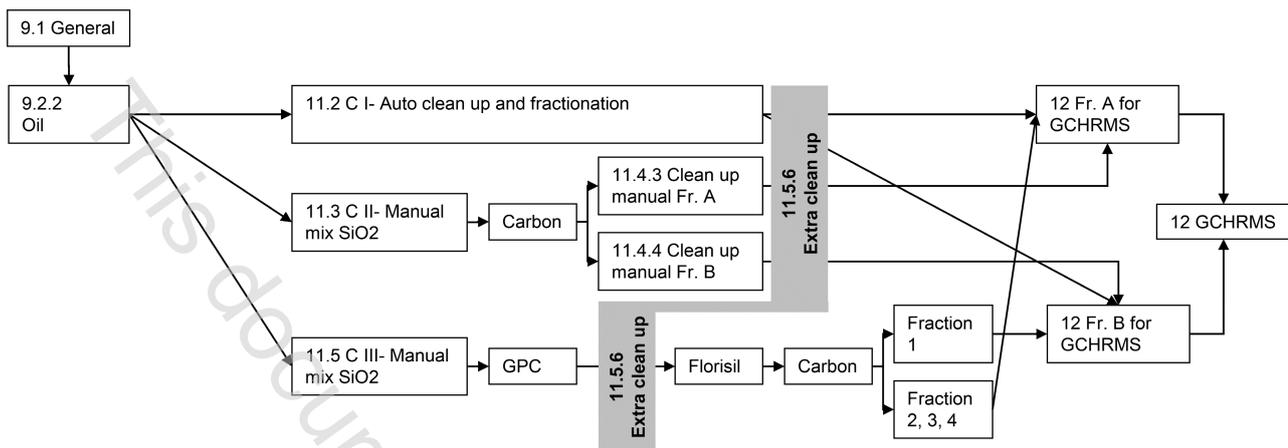


Figure 2 — Flow scheme for the determination of dioxins, dl-PCBs and non-dioxin-like-PCBs in oil and fat

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 blank determinations ($n > 10$).