

SVENSK STANDARD

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Slam, behandlat bioavfall och mark – Bestämning av dioxiner och furaner och dioxin-liknande polyklorerade bifenyler med högupplösande masspektrometri kopplat med gaskromatografi (HR GC-MS)

Soil, treated biowaste and sludge – Determination of dioxins and furans and dioxin-like polychlorinated biphenyls by gas chromatography with high resolution mass selective detection (HR GC-MS)

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EUROPEAN STANDARD

EN 16190

NORME EUROPÉENNE

EUROPÄISCHE NORM

December 2018

ICS 13.030.01; 13.080.10

Supersedes CEN/TS 16190:2012

English Version

Soil, treated biowaste and sludge - Determination of dioxins and furans and dioxin-like polychlorinated biphenyls by gas chromatography with high resolution mass selective detection (HR GC-MS)

Sols, bio-déchets traités et boues - Dosage des dioxines et furanes et polychlorobiphényles de type dioxine par chromatographie en phase gazeuse avec spectrométrie de masse à haute résolution (HR GC-SM)

Boden, behandelter Bioabfall und Schlamm - Bestimmung von Dioxinen und Furanen sowie Dioxin-vergleichbaren polychlorierten Biphenylen mittels Gaschromatographie und hochauflösender massenspektrometrischer Detektion (HR GC-MS)

This European Standard was approved by CEN on 23 October 2018.

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This European Standard was corrected and reissued by the CEN-CENELEC Management Centre on 9 January 2019.



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

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

Contents	Page
European foreword.....	4
Introduction	5
1 Scope.....	6
2 Normative references.....	7
3 Terms and definitions	7
4 Abbreviations	7
5 Principle	8
6 Reagents	8
7 Apparatus and materials.....	9
8 Sample storage and sample pretreatment.....	10
8.1 Sample storage.....	10
8.2 Sample pretreatment.....	10
9 Extraction and clean-up.....	11
9.1 General.....	11
9.2 Extraction.....	11
9.3 Clean-up.....	13
9.3.1 General.....	13
9.3.2 Gel permeation chromatography	13
9.3.3 Multilayer column.....	13
9.3.4 Sulphuric acid treatment.....	13
9.3.5 Activated carbon column.....	13
9.3.6 Aluminium oxide column	13
9.3.7 Removal of sulphur	13
9.4 Final concentration of cleaned sample extract	13
9.5 Addition of recovery standard	14
10 HRGC/HRMS analysis.....	14
10.1 General.....	14
10.2 Gas chromatographic analysis	15
10.3 Mass spectrometric detection	15
10.4 Minimum requirements for identification of PCDF/PCDD and PCB.....	16
10.5 Minimum requirements for quantification of PCDF/PCDD and PCB.....	17
10.6 Calibration of the HRGC/HRMS system	18
10.6.1 General.....	18
10.6.2 Calibration for 2,3,7,8-congeners.....	18
10.6.3 Calibration for sum of homologue groups	19
10.7 Quantification of HRGC/HRMS results.....	20
10.7.1 Quantification of concentrations of 2,3,7,8-congeners	20
10.7.2 Quantification of recovery rates of ¹³ C-labelled standards	20
10.7.3 Quantification of sum of homologue groups	21
10.7.4 Calculation of the toxic equivalent.....	21
10.7.5 Calculation of the limit of detection and the limit of quantification.....	22
11 Expression of results.....	22

12	Precision	22
13	Test report	23
	Annex A (informative) Toxic equivalency factor (TEF)	24
	Annex B (informative) Examples of extraction and clean-up methods	26
B.1	Example A	26
B.1.1	General	26
B.1.2	Chemicals	26
B.1.3	Procedure	27
B.2	Example B: Approved clean-up methods	32
	Annex C (informative) Examples of operation of GC/HRMS determination — Example	34
C.1	General	34
C.2	Gas chromatographic analysis	34
C.3	Mass spectrometric detection	35
	Annex D (informative) Repeatability and reproducibility data	38
D.1	Materials used in the interlaboratory comparison study	38
D.2	Interlaboratory comparison results	38
D.3	Calculation of toxicity factors on the basis of interlaboratory data	43
	Bibliography	45

SS-EN 16190:2019 (E)

European foreword

This document (EN 16190:2018) has been prepared by Technical Committee CEN/TC 444 “Test methods for environmental characterization of solid matrices”, the secretariat of which is held by NEN.

This document shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by June 2019, and conflicting national standards shall be withdrawn at the latest by June 2019.

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 CEN/TS 16190:2012.

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

The preparation of this document by CEN is based on a mandate by the European Commission (Mandate M/330), which assigned the development of standards on sampling and analytical methods for hygienic and biological parameters as well as inorganic and organic determinants, aiming to make these standards applicable to sludge, treated biowaste and soil as far as this is technically feasible.

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Introduction

Two groups of related chlorinated aromatic ethers are known as polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs); they consist of a total of 210 individual substances (congeners): 75 PCDD and 135 PCDF.

A group of chlorinated aromatic compounds similar to polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) is known as polychlorinated biphenyls (PCBs) which consist of 209 individual substances.

PCDD and PCDF can form in the combustion of organic materials; they also occur as undesirable by-products in the manufacture or further processing of chlorinated organic chemicals. PCDD/PCDF enter the environment via these emission paths and through the use of contaminated materials. In fact, they are universally present at very small concentrations. The 2,3,7,8-substituted congeners are toxicologically significant. Toxicologically much less significant than the tetrachlorinated to octachlorinated dibenzo-p-dioxins/dibenzofurans are the 74 monochlorinated to trichlorinated dibenzo-p-dioxins/dibenzofurans.

PCB have been produced over a period of approximately 50 years until the end of the 1990s for the purpose of different use in open and closed systems, e.g. as electrical insulators or dielectric fluids in capacitors and transformers, as specialized hydraulic fluids, as a plasticizer in sealing material. Worldwide more than one million tons of PCB were produced.

PCDD/PCDF as well as PCB are emitted during thermal processes as e.g. waste incineration. In 1997 a group of experts of the World Health Organization (WHO) fixed toxicity equivalent factors (TEF) for PCDD and twelve PCB, known as dioxin-like PCB (see Annex A). These twelve dioxin-like PCB consist of four non-ortho PCB and eight mono-ortho PCB (no or only one chlorine atoms in 2-, 2'-, 6- and 6'-position), having a planar or mostly planar structure. Dioxin-like PCB can contribute considerably to the total WHO-TEQ.

Only skilled operators who are trained in handling highly toxic compounds should apply the method described in this document.

This document is applicable for several types of matrices and validated for municipal sludge (see also Annex A for the results of the validation).

WARNING — Persons using this document should be familiar with normal laboratory practice. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

IMPORTANT — It is absolutely essential that tests conducted according to this document be carried out by suitably trained staff.

SS-EN 16190:2019 (E)

1 Scope

This document specifies a method for quantitative determination of 17 2,3,7,8-chlorine substituted dibenzo-p-dioxins and dibenzofurans and dioxin-like polychlorinated biphenyls in sludge, treated biowaste and soil using liquid column chromatographic clean-up methods and GC/HRMS.

The analytes to be determined with this document are listed in Table 1.

Table 1 — Analytes and their abbreviations

Substance	Abbreviation
Tetrachlorodibenzo-p-dioxin	TCDD
Pentachlorodibenzo-p-dioxin	PeCDD
Hexachlorodibenzo-p-dioxin	HxCDD
Heptachlorodibenzo-p-dioxin	HpCDD
Octachlorodibenzo-p-dioxin	OCDD
Tetrachlorodibenzofuran	TCDF
Pentachlorodibenzofuran	PeCDF
Hexachlorodibenzofuran	HxCDF
Heptachlorodibenzofuran	HpCDF
Octachlorodibenzofuran	OCDF
Polychlorinated biphenyl	PCB
Trichlorobiphenyl	TCB
Tetrachlorobiphenyl	TeCB
Pentachlorobiphenyl	PeCB
Hexachlorobiphenyl	HxCB
Heptachlorobiphenyl	HpCB
Decachlorobiphenyl	DecaCB

The limit of detection depends on the kind of sample, the congener, the equipment used and the quality of chemicals used for extraction and clean-up. Under the conditions specified in this document, limits of detection better than 1 ng/kg (expressed as dry matter) can be achieved.

This method is “performance based”. It is allowed to modify the method if all performance criteria given in this method are met.

NOTE In principle this method can also be applied for sediments, mineral wastes and for vegetation. It is the responsibility of the user of this document to validate the application for these matrices. For measurement in complex matrices like fly ashes adsorbed on vegetation it can be necessary to further improve the clean-up. This can also apply to sediments and mineral wastes.

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 15934, *Sludge, treated biowaste, soil and waste — Calculation of dry matter fraction after determination of dry residue or water content*

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

internal standard

¹³C₁₂-labelled 2,3,7,8-PCDD/PCDF analogue added to samples prior to extraction against which the concentrations of native PCDD and PCDF are calculated

[SOURCE: ISO 18073:2004, 3.1.5]

3.2

recovery standard

¹³C₁₂-labelled 2,3,7,8-chloro-substituted PCDD/PCDF, added before injection into the GC

[SOURCE: ISO 18073:2004, 3.1.12]

4 Abbreviations

I-TEF NATO/CCMS	International toxic equivalent factor proposed by NATO-CCMS in 1988 (for detailed description, see Annex A)
I-TEQ	International toxic equivalent obtained by multiplying the mass determined with the corresponding I-TEF including PCDD and PCDF (for detailed description, see Annex A). Should only be used for comparison with older data
PCDD/PCDF or PCDD/F	Polychlorinated dibenzo-p-dioxins/dibenzofurans
WHO-TEF	Toxic equivalent factor proposed by WHO in 2005 (for detailed description, see Annex A)
WHO-TEQ	Toxic equivalent obtained by multiplying the mass determined with the corresponding WHO-TEF including PCDD, PCDF and PCB (for detailed description, see Annex D). WHO-TEQ _{PCB} , WHO-TEQ _{PCDD/PCDF} should be used to distinguish different compound classes