

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)

EESTI STANDARDI EESSÕNA

NATIONAL FOREWORD

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Standard on jõustunud sellekohase teate avaldamisega EVS Teatajas.	This standard has been endorsed with a notification published in the official bulletin of the Estonian Centre for Standardisation.
Euroopa standardimisorganisatsioonid on teinud Euroopa standardi rahvuslikele liikmetele kättesaadavaks 19.12.2018.	Date of Availability of the European standard is 19.12.2018.
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ICS 13.030.01, 13.080.10

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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

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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of an identical text or by endorsement, at the latest by June 2019, and conflicting national standards shall be withdrawn at the latest by June 2019.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.

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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.

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