International Standard

ISO 13914

Soil, treated biowaste and sludge — Determination of dioxins and furans and dioxin-like polychlorinated biphenyls by gas chromatography with mass selective detection (high resolution mass spectrometry, HRMS, and tandem mass spectrometry, MS/MS)

Sols, biodéchets traités et boues — Dosage des dioxines, des furanes et des polychlorobiphényles de type dioxine par chromatographie en phase gazeuse avec détection sélective de masse (spectrométrie de masse à haute résolution ou SMHR, et spectrométrie de masse en tandem ou SM/SM)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 190, *Soil quality,* Subcommittee SC 3, *Chemical and physical characterization.*

This third edition cancels and replaces the second edition (ISO 13914:2023), which has been technically revised.

The main changes are as follows:

— tandem mass spectrometry (MS/MS) has been added as an alternative gas chromatographic analysis to high resolution mass spectrometry (HRMS);

— validation results for MS/MS have been added.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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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 of which 17 have chlorine substitution in the 2,3,7,8-positions.

A group of chlorinated aromatic compounds similar to polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) is known as polychlorinated biphenyls (PCBs) which consists 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 mainly 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 uses in open and closed systems, e.g. as electrical insulators or dielectric fluids in capacitors and transformers, as specialized hydraulic fluids and 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, for example 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 (dl-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.

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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.

IMPORTANT — Tests conducted according to this document shall 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 gas chromatography/high resolution mass spectrometry (GC/HRMS). Detection by tandem mass spectrometry (MS/MS) can be used in an equivalent way.

The analytes that can be determined with the method specified in 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 [a]	TCB
Tetrachlorobiphenyl	TeCB
Pentachlorobiphenyl	PeCB
Hexachlorobiphenyl	HxCB
Heptachlorobiphenyl	HpCB
Decachlorobiphenyl [a]	DecaCB
[a] Groups containing no dl-PCB, given for informative purpose only.	

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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". The method can be modified if all performance criteria given in this method are met.

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

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 such as 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.

ISO 11465, *Sludge and solid environmental matrices — Determination of dry residue or water content and calculation of the dry matter fraction on a mass basis*

3 Terms and definitions

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

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

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
internal standard
$^{13}C_{12}$-labelled analyte analogue added to samples prior to extraction against which the concentrations of native analytes are calculated

[SOURCE: ISO 18073:2004, 3.1.5, modified — "2,3,7,8-PCDD/PCDF" and "PCDDs and PCDFs" were changed to "analytes".]

3.2
recovery standard
$^{13}C_{12}$-labelled dl-polychlorinated biphenyl (dl-PCB) and polychlorinated dibenzo-p-dioxin/polychlorinated dibenzofuran (PCDD/PCDF), added before injection into the GC

[SOURCE: ISO 18073:2004, 3.1.12, modified — "2,3,7,8-chloro-substituted PCDD/PCDF" was changed to "dl-PCB and PCDD/PCDF".]

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