
**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, biodéchets traités et boues — Dosage des dioxines, des furanes
et des polychlorobiphényles de type dioxine par chromatographie en
phase gazeuse avec spectrométrie de masse à haute résolution (HR
CG-SM)*

[ISO 13914:2023](https://standards.iteh.ai/catalog/standards/sist/d5ee3b88-0baa-4f32-9da1-03123aacc63a1/iso-13914-2023)

<https://standards.iteh.ai/catalog/standards/sist/d5ee3b88-0baa-4f32-9da1-03123aacc63a1/iso-13914-2023>



iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 13914:2023

<https://standards.iteh.ai/catalog/standards/sist/d5ee3b88-0baa-4f32-9da1-03123aec63a1/iso-13914-2023>



COPYRIGHT PROTECTED DOCUMENT

© ISO 2023

All rights reserved. Unless otherwise specified, or required in the context of its implementation, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

	Page
Foreword.....	v
Introduction.....	vi
1 Scope.....	1
2 Normative references.....	2
3 Terms and definitions.....	2
4 Abbreviated terms.....	2
5 Principle.....	2
6 Reagents.....	3
6.1 Chemicals.....	3
6.2 Standards.....	3
7 Apparatus and materials.....	3
7.1 Equipment for sample preparation.....	3
7.2 Soxhlet extractor.....	4
7.3 Clean-up apparatus.....	4
7.4 Concentration apparatus.....	4
7.5 Other equipment.....	5
8 Sample storage and sample pretreatment.....	5
8.1 Sample storage.....	5
8.2 Sample pretreatment.....	5
9 Extraction and clean-up.....	5
9.1 General.....	5
9.2 Extraction.....	6
9.3 Clean-up.....	7
9.3.1 General.....	7
9.3.2 Gel permeation chromatography.....	7
9.3.3 Multilayer column.....	7
9.3.4 Sulphuric acid treatment.....	7
9.3.5 Activated carbon column.....	7
9.3.6 Aluminium oxide column.....	7
9.3.7 Removal of sulphur.....	7
9.4 Final concentration of cleaned sample extract.....	7
9.5 Addition of recovery standard.....	8
10 HRGC/HRMS analysis.....	8
10.1 General.....	8
10.2 Gas chromatographic analysis.....	8
10.3 Mass spectrometric detection.....	9
10.4 Minimum requirements for identification of PCDF/PCDD and PCB.....	10
10.5 Minimum requirements for quantification of PCDF/PCDD and PCB.....	10
10.6 Calibration of the HRGC/HRMS system.....	11
10.6.1 General.....	11
10.6.2 Calibration for 2,3,7,8-congeners.....	12
10.6.3 Calibration for sum of homologue groups.....	12
10.7 Quantification of HRGC/HRMS results.....	13
10.7.1 Quantification of concentrations of 2,3,7,8-congeners.....	13
10.7.2 Quantification of recovery rates of ¹³ C-labelled standards.....	13
10.7.3 Quantification of sum of homologue groups.....	13
10.7.4 Calculation of the toxic equivalent.....	14
10.7.5 Calculation of the limit of detection and the limit of quantification.....	14
11 Expression of results.....	15

12	Precision	15
13	Test report	15
	Annex A (informative) Toxic equivalency factor (TEF)	16
	Annex B (informative) Examples of extraction and clean-up methods	18
	Annex C (informative) Examples of operation of GC/HRMS determination — Example	25
	Annex D (informative) Repeatability and reproducibility data	29
	Bibliography	36

iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 13914:2023

<https://standards.iteh.ai/catalog/standards/sist/d5ee3b88-0baa-4f32-9da1-03123aec63a1/iso-13914-2023>

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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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 CEN/TC 444, *Environmental characterization of solid matrices* (as EN 16190:2018) and drafted in accordance with its editorial rules. It was assigned to Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical and physical characterization* and adopted under the "fast-track procedure".

This second edition cancels and replaces the first edition (ISO 13914:2013), which has been technically revised.

The main changes are as follows:

- technical content of EN 16190:2018 has been adopted;
- comprehensive validation results for soil, treated biowaste and sludge have been included;
- calculation of toxicity factors on the basis of interlaboratory data has 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.

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.

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

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)

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”. The method can be modified 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:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

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 Abbreviated terms

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

5 Principle

This document is based on the use of gas chromatography/mass spectrometry combined with the isotope dilution technique to enable the separation, detection and quantification of PCDD/PCDF and dioxin-like PCB in sludge, biowaste and soil. For the isotope dilution, method 17 labelled PCDD/PCDF and 12 labelled PCB internal standards are used. The extracts for the GC-MS measurements contain one or two recovery standards. The gas chromatographic parameters offer information which enables

the identification of congeners (position of chlorine substitutes) whereas the mass spectrometric parameters enable the differentiation between isomers with different numbers of chlorine substitutes and between dibenzo-p-dioxins, furans and PCB.

$^{13}\text{C}_{12}$ -labelled PCDD/PCDF and PCB congeners are added to the sample prior to extraction and HRGC/HRMS measurement. Losses during extraction and clean-up are detected and compensated by using these added congeners as internal standards for quantification together with recovery standards which are added just before the HRGC/HRMS analysis. For the determination of these substances it is necessary to separate PCB from PCDD/PCDF and vice versa.

The main purpose of the clean-up procedure of the raw sample extract is the removal of sample matrix components, which may overload the separation method, disturb the quantification or otherwise severely impact the performance of the identification and quantification method and the separation of PCDD/PCDF from dioxin-like PCB. Furthermore, the enrichment of the analytes in the final sample extract is achieved. Extraction procedures are usually based on Soxhlet or equivalent extraction methods of dried, preferably freeze-dried, samples. Sample clean-up is usually carried out by multi-column liquid chromatographic techniques using different adsorbents. The determination of PCDD/PCDF and PCB is based on quantification by the isotope-dilution technique using HRGC/HRMS.

6 Reagents

6.1 Chemicals

Solvents used for extraction and clean-up shall be of pesticide grade or equivalent quality and checked for blanks. Adsorbents like aluminium oxide, silica gel, diatomaceous earth and others used for clean-up shall be of analytical grade quality or better and pre-cleaned and activated if necessary.

NOTE See [Annex B](#) for a specific list of solvents and chemicals.

6.2 Standards

- ^{13}C -spiking solution for PCDD/PCDF (internal standard);
- ^{13}C -spiking solution for PCB (internal standard);
- calibration solutions PCDD/PCDF;
- calibration solutions PCB;
- recovery standard PCDD/PCDF;
- recovery standard PCB.

NOTE See [Annex B](#) for examples of concentration of the standard solutions.

7 Apparatus and materials

The apparatus and materials listed below are meant as minimum requirements for “conventional” sample treatment with Soxhlet extraction and column chromatographic clean-up. Additional apparatus and materials may be necessary due to different methods of sample extraction and clean-up methods.

7.1 Equipment for sample preparation

7.1.1 Laboratory fume hood, of sufficient size to contain the sample preparation equipment listed below.

7.1.2 Desiccator.

7.1.3 Balances, consisting of an analytical type capable of weighing 0,1 mg and a top-loading type capable of weighing 10 mg.

7.2 Soxhlet extractor

7.2.1 Soxhlet, 50 mm internal diameter, 150 ml or 250 ml capacity with 500 ml round bottom flask.

7.2.2 Thimble, 43 mm × 123 mm, to fit Soxhlet.

7.2.3 Hemispherical heating mantle, to fit 500 ml round-bottom flask.

7.3 Clean-up apparatus

7.3.1 Disposable pipettes, either disposable Pasteur pipettes, or disposable serological pipettes.

7.3.2 Glass chromatographic columns of the following sizes:

- 150 mm length × 8 mm internal diameter, with coarse-glass frit or glass-wool plug, 250 ml reservoir and glass or polytetrafluoroethylene (PTFE) stopcock;
- 200 mm length × 15 mm internal diameter, with coarse-glass frit or glass-wool plug, 250 ml reservoir and glass or PTFE stopcock;
- 300 mm length × 25 mm internal diameter, with coarse-glass frit or glass-wool plug, 300 ml reservoir and glass or PTFE stopcock.

7.3.3 Oven, capable of maintaining a constant temperature (± 5 °C) in the range of 105 °C to 450 °C for baking and storage of adsorbents.

7.4 Concentration apparatus

7.4.1 Rotary evaporator, equipped with a variable temperature water bath and:

- vacuum source for rotary evaporator equipped with shutoff valve at the evaporator and vacuum gauge;
- recirculating water pump and chiller, providing cooling water of (9 ± 4) °C (use of tap water for cooling the evaporator wastes large volumes of water and can lead to inconsistent performance as water temperatures and pressures vary);
- round-bottom flask, 100 ml and 500 ml or larger, with ground-glass fitting compatible with the rotary evaporator.

7.4.2 Nitrogen blowdown apparatus, equipped with either a water bath controlled in the range of 30 °C to 60 °C or a heated stream of nitrogen or of another suitable inert gas, installed in a fume hood.

7.4.3 Kuderna-Danish¹⁾ concentrator.

7.4.4 Sample vials, of the following types:

- amber glass, nominated volume 2 ml to 5 ml, with PTFE-lined screw cap;
- glass, 0,3 ml, conical, with PTFE-lined screw or crimp cap.

1) Kuderna Danish is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

7.5 Other equipment

7.5.1 Gas chromatograph, equipped with a splitless or on-column or temperature programmed injection port for the use with capillary columns, and an oven temperature programme which enables isothermal hold.

7.5.2 GC column for PCDD/PCDF and for isomer specificity for 2,3,7,8-TCDD (e.g. 60 m length × 0,32 mm internal diameter; 0,25 µm; 5 % phenyl, 94 % methyl, 1 % vinyl silicone bonded-phase fused-silica capillary column).

7.5.3 Mass spectrometer, 28 eV to 80 eV electron impact ionization, capable of repetitively selectively monitoring of twelve exact masses minimum at high resolution (>10 000) during a period of approximately 1 s.

7.5.4 Data system, capable of collecting, recording, and storing mass spectrometric data.

8 Sample storage and sample pretreatment

8.1 Sample storage

Samples should be stored in suitable containers with an appropriate closure material such as polytetrafluoroethylene (PTFE). Samples to be frozen may be stored in aluminium containers pre-cleaned by heating to 450 °C for minimum 4 h or by rinsing with a non-chlorinated solvent.

Samples should be kept cold (<8 °C) and in the dark. The sample pretreatment should take place within three days of sampling. If not achievable, samples may be frozen (-18 °C) directly after sampling and kept frozen before sample pretreatment.

8.2 Sample pretreatment

Drying and homogenization should be carried out according to EN 16179, if not otherwise specified. Store the ground material in a desiccator or a tightly closed glass container.

Determination of water content shall be carried out according to EN 15934.

9 Extraction and clean-up

9.1 General

In this document, the minimum requirements for extraction and clean-up to be met are described as well as examples of operation. The analyst may use any of the procedures given below and in [Annex C](#) or any suitable alternative procedures.

The determination of PCDD/PCDF is based on quantification by the isotope-dilution technique using HRGC/HRMS. ¹³C₁₂-labelled 2,3,7,8-chlorine substituted PCDD/PCDF congeners are added at different stages of the whole method. Losses during extraction and clean-up can be detected and compensated by using these added congeners as internal standards for quantification together with recovery standards which are added just before the HRGC/HRMS analysis. However, due to possible differences in the binding and adsorption characteristics between the native PCDD/PCDF and the ¹³C₁₂-labelled congeners, which are added during analysis, complete substantiation of the extraction efficiency and compensation of losses during clean-up is not ensured. Therefore, in addition the applied methods shall be validated thoroughly. Examples of well-proven extraction and clean-up methods are given in [Annex C](#).

The main purpose of the clean-up procedure of the raw sample extract is the removal of sample matrix components, which may overload the separation method, disturb the quantification or otherwise

severely impact the performance of the identification and quantification method and to separate dioxin-like PCB from PCDD/PCDF. Furthermore, an enrichment of the analytes in the final sample extract is achieved. Extraction procedures are normally based on Soxhlet extraction of the < 2 mm fraction of the dry and ground or sieved solid sample. Sample clean-up is usually carried out by multi-column liquid chromatographic techniques using different adsorbents.

In principle any clean-up method can be used which recovers the analytes in sufficient quantities. Furthermore, the final sample extract shall not affect adversely the performance of the analytical system or the quantification step. However, all applied methods shall be tested thoroughly and shall pass a set of method validation requirements before they can be employed (see Annex D). In addition, the verification of the method performance for each single sample shall be part of the applied quality assurance protocol.

9.2 Extraction

The sample amount used for extraction can vary from 5 g to 50 g depending on the expected level of contamination.

The internal standard consisting of ¹³C₁₂-labelled congeners listed in Table 2 shall be added directly onto the sample before extraction.

The extraction procedure is carried out using Soxhlet extraction with toluene. Duration of extraction should be adjusted according to kind and amount of sample used. The minimum requirement is 50 extraction cycles or approximately 12 h.

Other solvents or other methods like pressurized liquid extraction can also be used but shall be of proven equal performance.

Table 2 — ¹³C labelled congeners included in the internal standard

¹³ C-spiking solution – Internal standard	
PCDD/PCDF congeners	PCB congeners
2,3,7,8- ¹³ C ₁₂ -TCDD	¹³ C ₁₂ -PCB 77
1,2,3,7,8- ¹³ C ₁₂ -PeCDD	¹³ C ₁₂ -PCB 81
1,2,3,4,7,8- ¹³ C ₁₂ -HxCDD	¹³ C ₁₂ -PCB 126
1,2,3,6,7,8- ¹³ C ₁₂ -HxCDD	¹³ C ₁₂ -PCB 169
1,2,3,7,8,9- ¹³ C ₁₂ -HxCDD	
1,2,3,4,6,7,8- ¹³ C ₁₂ -HpCDD	¹³ C ₁₂ -PCB 105
¹³ C ₁₂ -OCDD	¹³ C ₁₂ -PCB 114
	¹³ C ₁₂ -PCB 118
2,3,7,8- ¹³ C ₁₂ -TCDF	¹³ C ₁₂ -PCB 123
1,2,3,7,8- ¹³ C ₁₂ -PeCDF	¹³ C ₁₂ -PCB 156
2,3,4,7,8- ¹³ C ₁₂ -PeCDF	¹³ C ₁₂ -PCB 157
1,2,3,4,7,8- ¹³ C ₁₂ -HxCDF	¹³ C ₁₂ -PCB 167
1,2,3,6,7,8- ¹³ C ₁₂ -HxCDF	¹³ C ₁₂ -PCB 189
2,3,4,6,7,8- ¹³ C ₁₂ -HxCDF	
1,2,3,7,8,9- ¹³ C ₁₂ -HxCDF	
1,2,3,4,6,7,8- ¹³ C ₁₂ -HpCDF	
1,2,3,4,7,8,9- ¹³ C ₁₂ -HpCDF	
¹³ C ₁₂ -OCDF	

9.3 Clean-up

9.3.1 General

Clean-up methods shall prepare the sample extract in an appropriate manner for the subsequent quantitative determination. Clean-up procedures shall concentrate PCDD/PCDF and dioxin-like PCB in the extracts and to remove interfering matrix components present in the raw extract.

Proven clean-up procedures shall be used including usually two or more of the following techniques which can be combined in different orders. A detailed description of some of the procedures is given in [Annex D](#).

Other methods can also be used but shall be of proven equal performance as the techniques described below.

9.3.2 Gel permeation chromatography

The interesting molecular weight range for PCDD/PCDF and dioxin-like PCB of 200 g/mol to 500 g/mol can be isolated from larger molecules and polymers which might overload other clean-up methods. This method can also be used for the removal of sulphur.

9.3.3 Multilayer column

Multilayer column liquid chromatography using silica with different activity grades and surface modifications is used. Compounds with different chemical properties than PCDD/PCDF and dioxin-like PCB can be removed.

9.3.4 Sulphuric acid treatment

A direct treatment of the sample extract with sulphuric acid is possible but is not recommended due to risk of accident. Furthermore, this shall be carried out very carefully to avoid losses of PCDD/PCDF and dioxin-like PCB on the formed carboniferous surfaces.

9.3.5 Activated carbon column

Column adsorption chromatography using activated carbon can be used to separate planar PCDD/PCDF and coplanar PCB molecules from mono-ortho PCB and other interfering non-planar molecules.

9.3.6 Aluminium oxide column

Column liquid chromatography on aluminium oxide of different activity grade and acidity/basicity is used. Interfering compounds with small differences in polarity or structure compared to PCDD/PCDF and dioxin-like PCB can be removed.

Additionally, aluminium oxide columns can be used to separate PCDD/PCDF from dioxin-like PCB.

9.3.7 Removal of sulphur

The removal of sulphur can be achieved by refluxing the extract with powdered copper or by gel permeation chromatography.

9.4 Final concentration of cleaned sample extract

To achieve sufficient detection limits, the cleaned sample extract shall be concentrated to a volume in the order of 25 µl to 100 µl before quantification. The final solvent shall be nonane, toluene or another high boiling solvent.