

Stationary source emissions — Determination of the mass concentration of PCDDs/PCDFs

Part 2. Extraction and clean-up

The European Standard EN 1948-2 : 1996 has the status of a
British Standard

ICS 13.040.40

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- Electricity Association
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National foreword

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This British Standard is published under the direction of the Health and Environment Sector Board whose Technical Committee EH/2 has the responsibility to:

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, the EN title page, pages 2 to 24, an inside back cover and a back cover.

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English version

Stationary source emissions — Determination of the mass concentration of PCDDs/PCDFs — Part 2: Extraction and clean-up

Emissions de sources fixes — Détermination de la concentration massique en PCDDs/PCDFs —
Partie 2: Extraction et purification

Emissionen aus stationären Quellen — Bestimmung der Massenkonzentrationen von PCDDs/PCDFs
Teil 2: Extraktion und Reinigung

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

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Foreword

This European Standard has been prepared by Technical Committee CEN/TC 264, Air quality, the secretariat of which is held by DIN.

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of an identical text or by endorsement, at the latest by June 1997, and conflicting national standards shall be withdrawn at the latest by June 1997.

This European Standard has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association, and supports essential requirements of EU Directive(s).

For relationship with EU Directive(s), see informative annex C, which is an integral part of this standard.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

This European Standard was elaborated by

AENOR (Spain)

AFNOR (France)

BSI (United Kingdom)

DIN (Germany)

DS (Denmark)

NNI (Netherlands)

NSF (Norway)

ON (Austria)

SFS (Finland)

SIS (Sweden)

SNV (Switzerland)

UNI (Italy)

The precision and the performance characteristics were determined in four comparative and validation trials at waste incinerators sponsored by the Commission of the European Communities, the European Free Trade Association and the German Federal Environment Agency.

This European Standard EN 1948 : 1996 consists of:

Part 1. Sampling

Part 2. Extraction and clean-up

Part 3. Identification and quantification

All three parts are necessary for the performance of the dioxin measurements.

The European Standard was developed on the basis of the following national standards or guidelines:

NFX 43-313 : 1991	<i>Air Quality — Stationary Source Emissions — Determination of PCDD/PCDF</i>
Nordic : 1987	<i>Recommended method for dioxin measurements in flue gases from waste incineration, Swedish Environmental Protection Agency</i>
Unichim Method N° 825 : 1989	<i>Stationary source emission measurements</i> <i>Conveyed gas flows</i> <i>Sampling and determination of organic micropollutants</i> – Sampling – PAH determination – PCDD and PCDF determination – PCB determination
VDI 3499 Part 1 : 1990 (Draft)	<i>Emission measurement — Measurement of residual materials — Determination of polychlorinated dibenzodioxins and dibenzofurans in flue and stack gas of incineration and firing plants — Dilution method — Determination in filter dust, potash and slag</i>
VDI 3499 Part 2 : 1993 (Draft)	<i>Emission measurement — Determination of polychlorinated dibenzo-p-dioxins (PCDD) and dibenzofurans (PCDF) — Filter/condenser method</i>
VDI 3499 Part 3 : 1996 (Draft)	<i>Emission measurement — Determination of polychlorinated dibenzo-p-dioxins (PCDD) and dibenzofurans (PCDF) — Cooled probe method</i>

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Introduction

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

PCDDs and PCDFs can form in the combustion of organic materials; they also may occur as undesirable by-products in the manufacture or further processing of chlorinated organic chemicals. PCDDs/PCDFs enter the environment via these emission paths and through the use of contaminated materials. In fact, they are universally present in very small concentrations.

The 2,3,7,8-chlorine substituted congeners are toxicologically significant. Toxicologically much less significant than the tetrachlorinated to octachlorinated dibenzodioxins/dibenzofurans are the 74 monochlorinated to trichlorinated dibenzodioxins/dibenzofurans (for toxicity equivalent factors, see annex A of EN 1948-1 : 1996).

Only skilled operators who are trained in handling highly toxic compounds should apply this standard.

1 Scope

The present Part of this standard specifies the extraction and clean-up procedures of the sampled PCDDs/PCDFs. It is an integral part of the complete measurement procedure. The use of the other two Parts EN 1948-1 : 1996 and EN 1948-3 : 1996 describing sampling or identification and quantification, respectively, is necessary for the determination of the PCDDs/PCDFs.

This standard has been designed to measure PCDD/PCDF concentrations at about 0,1 ng I-TEQ/m³ in stationary source emissions.

This standard specifies both method validation and a framework of quality control requirements which have to be fulfilled by any PCDD/PCDF extraction and clean-up methods to be applied. Some methods are described in detail in annex A as examples of proven procedures.

Each of the three sampling methods (Part 1) can be combined with the extraction and clean-up (Part 2) and the identification and quantification (Part 3) to complete the measurement procedure.

During comparison measurements on a municipal waste incinerator at the level of about 0,1 ng I-TEQ/m³ these three methods have been deemed comparable within the expected range of uncertainty. Validation trials were performed on the flue gas of municipal waste incinerators at the level of about 0,1 ng I-TEQ/m³ and a dust loading of from 1 mg/m³ to 15 mg/m³.

In principle it is not possible to evaluate the accuracy (trueness and precision) of emission measurements. Following the validation trials the internal and external variabilities were calculated for the process considered and are given in clause 13 of EN 1948-3 : 1996. These variabilities give an indication of the variabilities which have been observed when using this standard and should be taken into account when expressing results.

The procedure described in the three Parts of standard EN 1948 : 1996 lays down requirements in order to measure the 17 congeners required to calculate the total I-TEQ (see table A.1 of EN 1948-1 : 1996).

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

- EN 1948-1 : 1996 *Stationary source emissions — Determination of the mass concentration of PCDDs/PCDFs — Part 1. Sampling*
- EN 1948-3 : 1996 *Stationary source emissions — Determination of the mass concentration of PCDDs/PCDFs — Part 3: Identification and quantification*

A bibliography is shown in annex B (informative).

3 Definitions and abbreviations

3.1 Definitions

For the purposes of this standard, the following definitions apply:

3.1.1 spiking

Addition of $^{13}\text{C}_{12}$ -labelled PCDD/PCDF standards.

3.1.2 isokinetic sampling

Sampling at a rate such that the velocity including the direction of the gas entering the sampling nozzle is the same as that of the gas in the duct at the sampling point. [ISO 9096 : 1992]

3.1.3 operational performance characteristics

Measures which deal with the influence of the physical and chemical environment and maintenance problems, for example; mains voltage, temperature, supply of certain substances, set-up time, period of unattended operation.

3.1.4 statistical performance characteristics

Measures which quantify, for measured values, the possible deviations resulting from the random part of the measuring process; these are, for example, repeatability or instability.

3.1.5 control blank

A sample taken at the plant site in an identical manner to the normal samples including the spiking, but without introducing the probe into the flue gas and without introducing air into the sampling train. All the compartments up to and including the last collecting stage are rinsed or extracted in the normal manner.

3.1.6 extraction blank

A blank sample covering the complete analytical procedure including extraction, clean-up, identification and quantification including all the relevant reagents and materials.

3.1.7 sampling standard

$^{13}\text{C}_{12}$ -labelled 2,3,7,8-chlorine substituted PCDD/PCDF, added before sampling.

3.1.8 extraction standard

$^{13}\text{C}_{12}$ -labelled 2,3,7,8-chlorine substituted PCDD/PCDF, added before extraction and used for calculation of results.

3.1.9 syringe standard

$^{13}\text{C}_{12}$ -labelled 2,3,7,8-chlorine substituted PCDD/PCDF, added before injection into the GC.

3.1.10 keeper

High boiling point solvent added to the sampling standard solution.

3.1.11 congener

Any one of the 210 individual PCDDs/PCDFs.

3.1.12 PCDD/PCDF isomers

PCDDs or PCDFs with identical chemical composition but different structure.

3.1.13 pattern

Defined as a chromatographic print of any series of PCDD/PCDF isomers.

3.1.14 profile

Graphic representation of the sums of the isomer concentrations of the PCDDs and the PCDFs.

3.2 Abbreviations

3.2.1 I-TEQ

International toxic equivalent obtained by weighting the mass determined with the corresponding I-TEF. (For detailed description, see annex A of EN 1948-1 : 1996.)

3.2.2 I-TEF

International toxic equivalent factor. (For detailed description, see annex A of EN 1948-1 : 1996.)

3.2.3 GC/MS

Gas chromatography/mass spectrometry.

3.2.4 TCDD

Tetrachlorodibenzo-p-dioxin.

3.2.5 PeCDD

Pentachlorodibenzo-p-dioxin.

3.2.6 HxCDD

Hexachlorodibenzo-p-dioxin.

3.2.7 HpCDD

Heptachlorodibenzo-p-dioxin.

3.2.8 OCDD

Octachlorodibenzo-p-dioxin.

3.2.9 PCDD

Polychlorodibenzo-p-dioxin.

3.2.10 TCDF

Tetrachlorodibenzofuran.

3.2.11 PeCDF

Pentachlorodibenzofuran.

3.2.12 HxCDF

Hexachlorodibenzofuran.

3.2.13 HpCDF

Heptachlorodibenzofuran.

3.2.14 OCDF

Octachlorodibenzofuran.

3.2.15 PCDF

Polychlorodibenzofuran.

3.2.16 PCDDs/PCDFs

Polychlorinated dibenzo-p-dioxins/dibenzofurans.

3.2.17 PTFE

Polytetrafluoroethylene



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