

**Heitmed püsiallikatest. PCDD/PCDF  
massikontsentratsiooni määramine. Osa  
1: Proovivõtmine**

Stationary source emissions - Determination of the  
mass concentration of PCDDs/PCDFs - Part 1:  
Sampling

## EESTI STANDARDI EESSÕNA

## NATIONAL FOREWORD

<p>Käesolev Eesti standard EVS-EN 1948-1:1999 sisaldab Euroopa standardi EN 1948-1:1996 ingliskeelset teksti.</p> <p>Käesolev dokument on jõustatud 12.12.1999 ja selle kohta on avaldatud teade Eesti standardiorganisatsiooni ametlikus väljaandes.</p> <p>Standard on kättesaadav Eesti standardiorganisatsioonist.</p>	<p>This Estonian standard EVS-EN 1948-1:1999 consists of the English text of the European standard EN 1948-1:1996.</p> <p>This document is endorsed on 12.12.1999 with the notification being published in the official publication of the Estonian national standardisation organisation.</p> <p>The standard is available from Estonian standardisation organisation.</p>
--	---

<p><b>Käsitlusala:</b> Standardi käesolev osa määrab kindlaks PCDD/PCDF proovi võtmise. See on tervikliku mõõtmisprotseduuri lahutamatu osa. PCDD/PCDF määramiseks on vaja kasutada ka standardi kahte ülejäänud osa EN 1948-2:1996 ja EN 1948-3:1996, mis vastavalt kirjeldavad järgnevat ekstraheerimist, puhastamist ning kvalitatiivset ja kvantitatiivset analüüsimist.</p>	<p><b>Scope:</b></p>
--	----------------------

**ICS** 13.040.40

**Võtmesõnad:** ekstraheerimine, emissioon, heitgaasid, kontsentratsioon, kvaliteet, määramine, pcdd, pcdf, proovivõtmine, tuhastusseadmed, õhk, õhu saastumine

ICS 13.040.40

Descriptors: Air quality, PCDD, PCDF, emission, measurements.

**English version**

Stationary source emissions

**Determination of the mass concentration  
of PCDDs/PCDFs**

Part 1: Sampling

Emissions de sources fixes –  
Détermination de la concentration  
massique en PCDDs/PCDFs – Partie 1:  
Prélèvement

Emissionen aus stationären Quellen –  
Bestimmung der Massenkonzentration  
von PCDD/PCDF – Teil 1: Probenahme

This European Standard was approved by CEN on 1996-12-27.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

The European Standards exist in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, the Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, the Netherlands, Norway, Portugal, Spain, Sweden, Switzerland, and the United Kingdom.

**CEN**

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

**Central Secretariat: rue de Stassart 36, B-1050 Brussels**

## Contents

	Page
Foreword	4
Introduction	6
1 Scope	7
2 Normative references	8
3 Definitions and abbreviations	8
3.1 Definitions	8
3.2 Abbreviations	10
4 Principle of the complete PCDD/PCDF measurement procedure	11
4.1 Sampling	11
4.1.1 General	11
4.1.2 Filter/condenser method	11
4.1.3 Dilution method	12
4.1.4 Cooled probe method	13
4.2 Extraction and clean-up	14
4.3 Identification and quantification	14
5 Sampling device and materials	15
5.1 Sampling device	15
5.1.1 General device	15
5.1.2 Sampling device for the filter/condenser method	15
5.1.2.1 Without division of sample gas	15
5.1.2.2 With division of sample gas	16
5.1.3 Sampling device for the dilution method	17
5.1.4 Sampling device for the cooled probe method	18
5.2 Materials	19
6 Minimum requirements for sampling	20
6.1 Method validation criteria	20
6.2 Minimum requirements for sampling	20
6.3 Additional sampling minimum requirements for the filter/condenser method	22
6.4 Additional sampling minimum requirements for the dilution method	22
6.5 Additional minimum requirements for the cooled probe/adsorber method	23
7 Safety measures and transport	23
8 Preparations and sampling	23
8.1 Advance preparations at the sampling site	23
8.1.1 Selection of suitable sampling location	23
8.2 Advance preparations at the laboratory	24
8.2.1 Preparation of equipment	24

8.2.2	Preparation of parts of the sampling train in contact with the sample	24
8.3	Sampling at the plant	25
8.3.1	Initial checks	25
8.3.2	Preliminary survey	25
8.3.3	Assembly of apparatus	25
8.3.4	Sampling	26
8.3.4.1	Record keeping	26
8.3.4.2	Changing sampling line	26
8.3.5	After sampling	26
8.3.5.1	Sample recovery from the sampling train	26
8.3.5.2	Sample storage	27
8.4	Calculation of the flue gas volume	27
8.5	Sampling report	28
Annex A (informative)	Toxic equivalency	31
Annex B (informative)	Examples of operation	33
B.1	Filter/condenser method	34
B.2	Filter/condenser method - Variant with flow division	37
B.3	Filter/condenser method - Variant with solid adsorbent upstream of the condensate flask	43
B.4	Dilution method - Automated variant	46
B.5	Dilution method - Manual variant	51
B.6	Cooled probe method - Variant 1	54
B.7	Cooled probe method - Variant 2	58
Annex C (informative)	Bibliography	65
Annex D (informative)	Examples of adsorbents and their preparations and cleaning	67
Annex E (informative)	Moisture content determination	68
Annex F (informative)	Oxygen content determination	69
Annex G (informative)	Sampling measurement record	70
Annex H (informative)	Clauses of this European Standard addressing essential requirements or other provisions of EU Directives	74

## **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 H, 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 European Commission, the European Free Trade Association and the German Federal Environment Agency.

This European Standard EN 1948:1996 consists of three parts dealing with the determination of the mass concentration of PCDDs and PCDFs in stationary source emissions:

- 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:

- NF X 43-313:1991 Air quality - Stationary source emissions - Determination of PCDD/PCDF
- Nordic:1987 Recommended method for dioxin measurements in flue gases from waste incineration, Swedish Environmental Protection Agency
- Unichim Method N° 825:1989 Stationary source emission measurements - Conveyed gas flows - Sampling and determination of organic micropollutants
  - Sampling
  - PAH determination
  - PCDD and PCDF determination
  - PCB determination
- VDI 3499 Part 1:1990 (draft) 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
- VDI 3499 Part 2:1993 (draft) Emission measurement - Determination of polychlorinated dibenzo-p-dioxins (PCDD) and dibenzofurans (PCDF) - Filter/condenser method
- VDI 3499 Part 3:1996 (draft) Emission measurement - Determination of polychlorinated dibenzo-p-dioxins (PCDD) and dibenzofurans (PCDF) - Cooled probe method

## **Introduction**

Two groups of related chlorinated aromatic 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 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 tetrachlorinated to octachlorinated dibenzodioxins/dibenzofurans are the 74 monochlorinated to trichlorinated dibenzodioxins/dibenzofurans (for toxicity equivalent factors, see Annex A).

## 1 Scope

This part of the Standard specifies the sampling of PCDDs/PCDFs. It is an integral part of the complete measurement procedure. The use of the other two parts EN 1948-2:1996 and EN 1948-3:1996 describing extraction and clean-up and identification and quantification, respectively, is necessary for the determination of the PCDDs/PCDFs.

This Standard has been developed to measure concentrations at about 0,1 ng I-TEQ/m<sup>3</sup> 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 sampling.

The user has the possibility to choose between three different methods:

"Filter/Condenser Method"

"Dilution Method"

"Cooled Probe Method"

Each sampling method is illustrated by some sampling systems described in detail in Annex B as examples of proven procedures.

During comparison measurements on municipal waste incinerators at the level of about 0,1 ng I-TEQ/m<sup>3</sup> 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/m<sup>3</sup> and a dust loading of from 1 mg/m<sup>3</sup> to 15 mg/m<sup>3</sup>.

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 need to be taken into account when expressing results.

The procedure described in the three parts of EN 1948:1996 specifies requirements which shall be met in order to measure the 17 congeners necessary to calculate the total I-TEQ (see Table A.1).

## 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 1948-2:1996 Stationary source emissions - Determination of the mass concentration of PCDDs/PCDFs - Part 2: Extraction and clean-up
- EN 1948-3:1996 Stationary source emissions - Determination of the mass concentration of PCDDs/PCDFs - Part 3: Identification and quantification
- ISO 4793:1980 Laboratory sintered (fritted) filters - Porosity grading, classification and designation
- ISO 845:1988 Cellular plastics and rubbers - Determination of apparent (bulk) density
- ISO 9096:1992 Stationary source emissions - Determination of concentration and mass flow rate of particulate material in gas-carrying ducts - Manual gravimetric method

A bibliography is shown in Annex C (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 PCDDs and PCDFs standards
- 3.1.2 isokinetic sampling** Sampling at a rate such that the velocity 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