



BSI Standards Publication

Stationary source emissions — Determination of the mass concentration of PCDDs/PCDFs and dioxin-like PCBs

Part 5: Long-term sampling of PCDDs/
PCDFs and PCBs

National foreword

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**Stationary source emissions - Determination of the mass
concentration of PCDDs/PCDFs and dioxin-like PCBs - Part 5:
Long-term sampling of PCDDs/PCDFs and PCBs**

Emissions de sources fixes - Détermination de la
concentration massique en PCDD/PCDF et PCB de type
dioxine - Partie 5: Prélèvement à long-terme de
PCDD/PCDF et PCB

Emissionen aus stationären Quellen - Bestimmung der
Massenkonzentration von PCDD/PCDF und dioxin-
ähnlichen PCB - Teil 5: Langzeitprobenahme von
PCDD/PCDF und PCB

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels

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Foreword

This document (CEN/TS 1948-5:2015) has been prepared by Technical Committee CEN/TC 264 "Air quality", the secretariat of which is held by DIN.

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Introduction

EN 1948-1, EN 1948-2, EN 1948-3 and EN 1948-4 describe reference methods for the determination of PCDD/PCDF/PCB, whereas this Technical Specification gives requirements for long-term sampling measurements in connection with the appropriate analytical methods (equivalent method). In contrast to the standard reference method (EN 1948-1) which refers to monitoring the limit value for compliance with emission limit values (ELVs) in Directives, such as Industrial Emission Directive (IED) [10], the long-term sampling is intended to determine the average concentration level during a longer period (see e.g. [12], [13]). CEN/TS 1948-5 provides a method for measuring long term average mass concentrations but it does not specify its potential use by the competent authority for demonstrating compliance with long term ELVs.

Long-term sampling methods are not automatic measurement methods and do not provide continuous emission monitoring data (real time display).

This Technical Specification in connection with EN 1948-2 and EN 1948-3 (extraction and analysis) are necessary for the performance of long-term sampling of PCDDs/PCDFs/ PCBs.

In some European Union countries PCDD/PCDF/PCB long-term sampling is an obligatory measurement for some incineration processes. In other countries of the European Union this may be obligatory in the future.

The European Organization for Standardization (CEN) draws attention to the fact that it is claimed that compliance with this document may involve the use of patents concerning the use of PCDD/PCDF/PCB long-term sampling systems, described in this document. This is valid for

- a) the filter/condenser method (see 5.2) and
- b) the cooled probe method (see 5.4).

CEN takes no position concerning the evidence, validity and scope of these patent rights.

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It should be mentioned that also a patent right existed for the dilution method (see 5.3). This patent was phased out in September 2014.

In Reference [1] the results of a round robin test for long-term sampling are presented.

WARNING All relevant national safety regulations shall be observed. The 2,3,7,8-chlorine substituted PCDDs/PCDFs belong to the most toxic of chemicals. In addition working at the sampling site may include exposure to a range of hazards such as poisonous/asphyxiating flue gases and working at heights. Appropriate measures shall be taken to minimize exposure to such hazards. Care shall be taken when transporting samples to avoid their breakage both to prevent contamination and to avoid sample losses.

1 Scope

This Technical Specification specifies the long-term sampling of PCDDs, PCDFs and PCBs. There are three different sampling methods, which use the three different principles described in EN 1948-1 modified for long-term sampling requirements:

- filter/condenser method;
- dilution method;
- cooled probe method.

Each sampling method is illustrated in detail in Annex D. The sampling methods described in this document are designed for a sampling duration of typically four weeks.

Additionally this document specifies a framework of quality control requirements for any long-term sampling method to be applied (see Annex C and Annex F).

With the methods described experiences were gained for a concentration range from typically 0,003 ng I-TEQ/m³ up to 4,0 ng I-TEQ/m³ and 0,003 ng WHO-TEQ/m³ up to 4,0 ng WHO-TEQ/m³ respectively at different stationary sources (e.g. waste incinerators, sinter plants, cement kilns).

For the complete measurement method the use of EN 1948-2 and EN 1948-3 describing extraction and clean-up and identification and quantification, respectively, is necessary in order to determine PCDDs/PCDFs. Also EN 1948-4 is necessary for the analyses of dioxin-like PCBs.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 1948-1:2006, *Stationary source emissions - Determination of the mass concentration of PCDDs/PCDFs and dioxin-like PCBs - Part 1: Sampling of PCDDs/PCDFs*

EN 1948-2:2006, *Stationary source emissions - Determination of the mass concentration of PCDDs/PCDFs and dioxin-like PCBs - Part 2: Extraction and clean-up of PCDDs/PCDFs*

EN 1948-3:2006, *Stationary source emissions - Determination of the mass concentration of PCDDs/PCDFs and dioxin-like PCBs - Part 3: Identification and quantification of PCDDs/PCDFs*

EN 1948-4:2010+A1:2013, *Stationary source emissions - Determination of the mass concentration of PCDDs/PCDFs and dioxin-like PCBs - Part 4: Sampling and analysis of dioxin-like PCBs*

EN 13284-1:2001, *Stationary source emissions - Determination of low range mass concentration of dust - Part 1: Manual gravimetric method*

EN 15259:2007, *Air quality - Measurement of stationary source emissions - Requirements for measurement sections and sites and for the measurement objective, plan and report*

EN 15267-1, *Air quality - Certification of automated measuring systems - Part 1: General principles*

EN 15267-2, *Air quality - Certification of automated measuring systems - Part 2: Initial assessment of the AMS manufacturer's quality management system and post certification surveillance for the manufacturing process*

EN 15267-3:2007, *Air quality - Certification of automated measuring systems - Part 3: Performance criteria and test procedures for automated measuring systems for monitoring emissions from stationary sources*

EN ISO 16911-1:2013, *Stationary source emissions - Manual and automatic determination of velocity and volume flow rate in ducts - Part 1: Manual reference method (ISO 16911-1:2013)*

3 Terms and definitions

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

3.1

sampling unit

different media including adsorber, absorber and filter in order to collect the PCDDs/PCDFs/PCBs

Note 1 to entry: Each sampling system may use different collection systems to collect PCDD/PCDF and PCBs in the gaseous and particulate form (e.g. filter, cartridge with sorbent). In this standard the whole collection system is considered as the sampling unit which is sent to the laboratory for analysis.

3.2

long-term sampling system

system to sample up to typically four weeks

3.3

standard reference method

SRM

sampling according to EN 1948-1 and extraction/clean-up/analysis according to EN 1948-2 and EN 1948-3

3.4

stand by

interruption of the measurement period due to plant shut down or during the changing of sampling support (filters and adsorbant)

3.5

yearly PCDD/PCDF/PCB surveillance

several repeated long-term PCDD/PCDF/PCB measurements during 1 year

3.6

standard flue gas sample volume

flue gas sample volume expressed at standard conditions of temperature (273,15 K) and pressure (101,3 kPa) on a dry basis and if required corrected to the reference concentration of oxygen

3.7

isokinetic sampling

sampling at a flow rate such that the velocity (amount and direction) of the gas entering the sampling nozzle are the same as the velocity of the gas in the duct at the sampling point

3.8

sampling standard

¹³C₁₂-labelled 2,3,7,8-chlorine substituted PCDFs/PCB added before sampling

3.9

extraction standard

¹³C₁₂-labelled 2,3,7,8-chlorine substituted PCDDs/PCDFs/PCB, added before extraction

Note 1 to entry: These standards are also used for calculation of results.

3.10

recovery standard

¹³C₁₂-labelled 2,3,7,8-chlorine substituted PCDDs/PCBs added before injection

3.11

type performance test

test to be performed by an independent test house in a laboratory and on site to test the conformity of the automatic samplers according to the defined performance criteria corresponding to the concerned application

Note 1 to entry: This step, in analogy to automated measuring systems (AMS), provides the information to perform the step QAL (see also introduction).

Note 2 to entry: The type performance testing is part of the certification process described, by analogy with AMS described in EN 15267–3.

Note 3 to entry: The test laboratory should have accreditation to EN ISO/IEC 17025 for the dioxin SRM. The analytical lab should have accreditation to EN ISO/IEC 17025 for the analysis (see 7.1. i), 8)).

[SOURCE: EN 14181 [2]]

3.12

validation of the installation and calibration in the field

Validation performed for each plant by an independent test house to check the defined performance criteria

Note 1 to entry: This step in analogy to AMS (automated measurement systems), corresponds to the QAL2 (see also introduction).

Note 2 to entry: The test laboratory should have accreditation to EN ISO/IEC 17025 for the dioxin SRM. The analytical lab should have accreditation to EN ISO/IEC 17025 for the analysis (see 7.1. i), 8)).

[SOURCE: EN 14181 [2]]

3.13

field blank value

value determined by a blank sample

Note 1 to entry: The described measurement procedure is employed to ensure that no significant contamination has occurred during all steps of the measurement and to check that the operator can achieve a quantification level adapted to the task.

3.14

accuracy

closeness of agreement between a measured quantity value and a true quantity value of a measurand

[SOURCE: VIM:2012 [3]]

3.15

maintenance interval

maximum admissible interval of time for which the performance characteristics will remain within a pre-defined range without servicing – cleaning, calibration or adjustment

3.16

measurement section

region of the flue gas duct which includes the measurement plane(s)

[SOURCE: EN 15259:2007; 3.12]

3.17

measurement cross section

plane normal to the centre line of the duct at the sampling position

Note 1 to entry: Measurement plane is also known as sampling plane.

[SOURCE: EN 15259:2007; 3.13]

3.18

availability

fraction of the total monitoring time for which data/sample material of acceptable quality has been collected

3.19

reference quantity

specified physical or chemical quantity which is needed for conversion of the measurand to standard conditions

Note 1 to entry: Reference quantities are e.g. temperature ($T_{\text{ref}} = 273,15 \text{ K}$), pressure ($p_{\text{ref}} = 101,325 \text{ kPa}$), water vapour volume fraction ($h_{\text{ref}} = 0 \%$) and oxygen volume fraction (o_{ref}).

3.20

purging

operation carried out to keep the probe clean during the stand-by modus

Note 1 to entry: Using oil-free instrument compressed air.

Note 2 to entry: The purging flow is in the direction of the nozzle (countercurrent if compared to the sampling flow, passing in the whole probe and nozzle). The aim of the purging is to prevent contamination of the nozzle while the instrument is in stand-by mode. Purging is not related to the standby cause. Probe's temperature is the one selected for stand-by mode.

3.21

rinsing

solvent based operation at least once a year

Note 1 to entry: See 7.1, i). and Annex B.

Note 2 to entry: Rinsing is carried out at the end of sampling run on all parts being in contact with the flue gas. Solvents are the same described in EN 1948-1:2006, 6.3 and they can be considered as part of the sample.

3.22

cleaning

operation requiring a complete removal and cleaning of the parts of the sampling train which can be affected e.g. by contamination

Note 1 to entry: Solvents used for cleaning of external parts of the sampling train are not to be considered part of the sample.

3.23

thermal desorption of the probe and sampling line

operation carried out at the end of the sampling run

Note 1 to entry: Temperature of the probe when heated, increases up to $200 \text{ }^{\circ}\text{C}$ to remove trace organic compounds which can be settled in the probe after a long-term sampling. The direction of the flow is from the nozzle to the sampling unit. Duration of the process is typically 15 min to 30 min.

3.24

substitute

value programmed in the memory of the control unit

Note 1 to entry: This substitute is used to continue the run of the sampling even in case of defect or missing of parameters of the system. For instance, if the external oxygen analyser is out of use, it is possible to switch to the substitute value for oxygen and to continue the run of the sampling. The value is calculated taking into account the mean value of the parameters since the last maintenance. In the case of e.g. oxygen analyser maintenance the last valid value is taken as long as the "Analyser Maintenance" signal applies. Attention is drawn to the fact that it should be reported, if values are substituted (see 7.2.4.4).

4 Symbols and abbreviations

For the purposes of this document the following symbols apply.

4.1 General

ELV	emission limit value
I-TEF	international toxic equivalent factor (for a detailed description see EN 1948–1)
I-TEQ	international toxic equivalent obtained by weighting the mass determined with the corresponding I-TEF (for a detailed description, see EN 1948–1)
LOQ	lower limit of quantification
PU foam	polyurethane foam used as adsorbent
ULOQ	upper limit of quantification
WHO-TEF	toxic equivalent factor proposed by WHO (for detailed description see EN 1948–1)
WHO-TEQ	toxic equivalent obtained by multiplying the mass determined with the corresponding WHO-TEF including PCDDs, PCDFs, PCBs (for detailed description see EN 1948–1)
XAD-2	used as adsorbent

4.2 Polychlorinated biphenyls, polychlorinated dibenzodioxins and polychlorinated dibenzofuranes

HpCDD	heptachlorodibenzo-p-dioxin
HpCDF	heptachlorodibenzofurane
HxCDD	hexachlorodibenzo-p-dioxin
HxCDF	hexachlorodibenzofurane
OCDD	octachlorodibenzo-p-dioxin
OCDF	octachlorodibenzofurane
PCDD/PCDF	polychlorinated dibenzo-p-dioxin/dibenzofurane
PCB	polychlorinated biphenyl
PeCDD	pentachlorodibenzo-p-dioxin
PeCDF	pentachlorodibenzofurane
TCDD	tetrachlorodibenzo-p-dioxin
TCDF	tetrachlorobenzofurane

5 Principle of long-term PCDD/PCDF/PCB sampling

5.1 General

For patent rights see Introduction.

The sampling is done isokinetically in the duct for a long time period usually from 24 h up to several weeks, typically four weeks. The sample gas flow rate is automatically controlled. The PCDDs/PCDFs/PCBs, both adsorbed on particles and in the gas phase, are collected in the long term sampling system. Dependent on the sampling system, the sampling unit can consist of different compartments, e.g. filter, condensate flask unit, solid or liquid adsorbent. The systems are based on the following sampling methods, described in EN 1948-1:

- filter/condenser method;
- dilution method;
- cooled probe method.

Schematic representations of the sampling methods according to EN 1948-1 including the modifications for long-term sampling are given in Figure 1, Figure 2 and Figure 3.

The sampling unit is spiked with $^{13}\text{C}_{12}$ -labelled PCDFs/PCBs before sampling to determine the sampling recovery rate of the congeners. The sample gas is brought to a temperature specific to the sampling system and the gaseous and particulate PCDDs/PCDFs/PCBs are trapped.

The minimum requirements of the long-term sampling procedure to be met are described in this part of EN 1948. Examples of operation are listed in Annex D. The described systems meet the minimum requirements of Clause 7 and Annex A, but differ in their approach.

After sampling, the sampling unit and the data storage device has to be sent to the analytical laboratory for the sample analysis. The necessary safety precautions, e.g. for transport, have to be fulfilled.

5.2 Long-term sampling based on the filter/condenser method

Long-term sampling systems based on the filter/condenser method filter the dust at a temperature above the flue gas dew point but below 125 °C and cool down the sampling gas to a temperature of about 20 °C as described in EN 1948-1. A higher temperature can be used if it is demonstrated that the minimum requirements for adsorption efficiency are fulfilled (see 7.1, e) and f) and 7.2.1, d)) The PCDDs/PCDFs/PCBs passing the filter are collected in an adsorption medium, e.g. XAD-2. The complete sampling unit comprises a filter and the cartridge with the adsorbent. The condensate is filtered through the adsorption cartridge and discarded via the condensate flask unit.

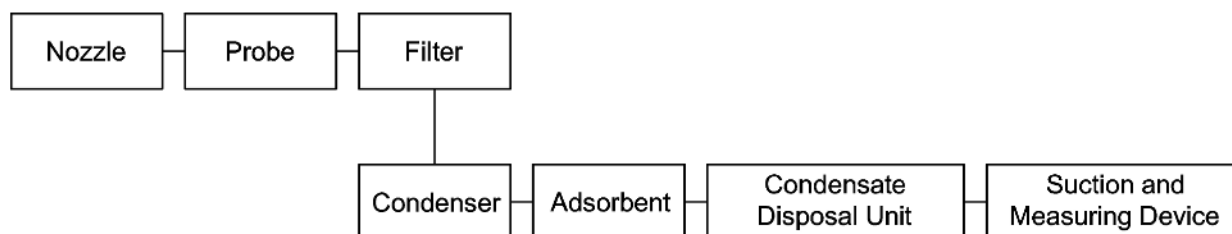


Figure 1 — Scheme of long-term sampling system based on filter/condenser method

5.3 Long-term sampling based on the dilution method

Long-term sampling systems based on the dilution method extract the flue gas from the stack and dilutes the flue gas with cleaned air to a temperature of about 40 °C as described in EN 1948-1 before filtering the dust and adsorption of the gaseous PCDD/PCDF/PCBs in a cartridge. A higher temperature can be used if it is demonstrated that the minimum requirements for adsorption efficiency are fulfilled (see 7.1, e) and f) and 7.2.1, d)). The long-term sampling using the dilution method adds dilution air to the sample in order to avoid the relative humidity of the sampling gas to reach 100 %.

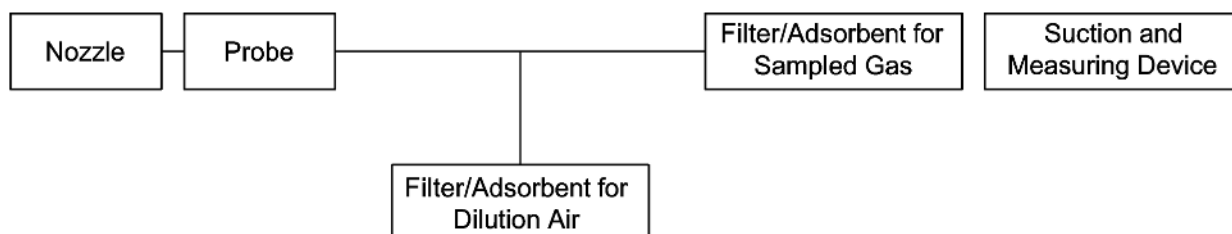


Figure 2 — Scheme of long-term sampling system based on dilution method

5.4 Long-term sampling based on the cooled probe method

Long-term sampling systems based on the cooled probe method extract the sample from the gas stream continuously through a probe, which is cooled to below 20 °C as mentioned in EN 1948-1. The wet sample is drawn through a sampling unit, which consists of a filter and a suitable adsorption medium, e.g. XAD-2. An upper temperature can be used if it is demonstrated that the minimum requirements for adsorption efficiency are fulfilled (see 7.1,e) and f) and 7.2.1,d)). The condensate is filtered through the adsorption cartridge and discarded via the condensate flask unit.

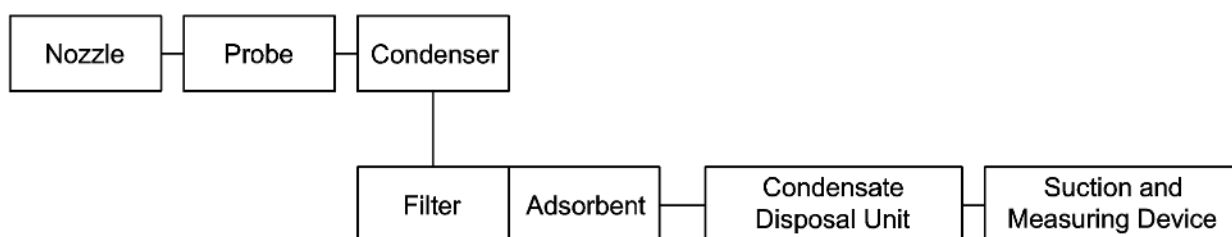


Figure 3 — Scheme of long-term sampling system based on cooled probe method

6 Sampling device and materials

6.1 General sampling device

- a) sampling train
- b) devices for measuring the flue gas parameters
- c) automatic controller
- d) measurement data storage system

6.2 Components for the sampling train

- a) nozzle with defined shape
- b) probes (cooled or heated)

- c) flue gas velocity measurement device: e.g. Pitot tube or zero pressure probe
- d) condenser, if part of the method
- e) tempered filter holder
- f) insulated and tempered adsorber cartridge
- g) volume measurement device
- h) pump to suck in the sample gas

6.3 Automatic controller

The automatic controller should be protected by a password to prevent inadvertent and unauthorized access or adjustment and shall have the following automatic functions:

- a) maintenance of the isokinetic sampling condition;
- b) leak test at the start and end of the sampling (or can be performed manually as an option);
- c) restart after power loss; (after start-up procedures are completed);
- d) automatic start and stop following the operation condition of the plant; the start and stop functions are also under control of the operators; the controller functions themselves are sufficiently described by demanding the functionality of reacting upon suitable external control signals, e.g. from oxygen measurement system.

6.4 Devices for measuring the flue gas parameters

Dependent on the type of plant, there are different components and flue gas parameters needed.

- a) flue gas velocity in accordance with EN ISO 16911-1 barometer and pressure gauge, if necessary;
- b) temperature of the flue gas, if necessary;
- c) flue gas composition, if necessary (e.g. taken by the plant data system);
- d) O₂ measurement device (e.g. taken by the plant data system);
- e) humidity measurement device (e.g. taken by the plant data system).

6.5 Materials

- a) All parts of the system in contact with the flue gas: nozzle, probe, glass tubes, filter holder, filter casing, condenser, bubbler, sampling unit etc. shall be made of non-PCDD/PCDF/PCB-adsorbing materials as titanium, quartz or glass.
- b) Sealings coming in direct contact with the flue gas shall be made of PTFE furthermore, filter supporting parts made of PTFE can be used if validated by the manufacturer.
- c) Particle filter shall be made of quartz or glass fibre, meeting the minimum requirements (see 7.1, e)).

- d) Solid adsorbent, XAD-2¹⁾, polyurethane foam (PU foam), other solid adsorbents (e.g. Porapak PS¹⁾) meeting the minimum requirements (see 7.2.1, e)). See EN 1948-2:2006, Annex A, for examples on material specification.

7 Minimum requirements for long-term PCDD/PCDF/PCB sampling methods

7.1 Certification of the sampling system

The minimum requirements and method validation criteria shall be fulfilled by the manufacturer of the long-term sampling system and documented and checked during the type performance test which is part of the certification process (see EN 15267-1). For this the following criteria shall be fulfilled.

- a) A validation trial shall be carried out at least once with each design of sampling equipment for the specified maximum sampling volume and the specified maximum sampling period (usually 2 weeks or 4 weeks). This trial has to be performed in order to validate the sampling efficiency and the long-term behaviour of the system for the specified measurement periods.
- b) Sufficient sample mass shall be collected during all tests of the validation trial so that the quantification limit is less than 5 % of the total amount collected (expressed in I-TEQ or WHO-TEQ).
- c) In the case of using two or more probes, it shall be ensured that the unused probes are protected against contamination or cleaned before use, if exposed for long time to stack atmosphere.
- d) In case, XAD-2 is used, the XAD-2 cartridge shall be mounted in a vertical direction in order to avoid channelling. Additionally, the flue gas shall flow from top to bottom of the XAD-2 cartridge.
- e) The filter efficiency shall be higher than 99,5 % on a test aerosol with a mean particle diameter of 0,3 µm, at the maximum flow rate anticipated (or 99,9 % on a test aerosol of 0,6 µm mean diameter). This efficiency shall be certified by the filter supplier.
- f) The overall deposits in the sampling train shall be determined at the end of the intended sampling duration (typically 4 weeks). More than 90 % of the total I-TEQ and WHO-TEQ respectively shall be found in the analysed compartments of the sampling train. This means that less than 10 % of total I-TEQ/WHO-TEQ are acceptable to be found in the sum of the breakthrough sample (see also 7.2.1) and in all compartments not analysed in routine measurements. The breakthrough test shall be performed covering the intended sampling duration using a second sampling unit in series.
- g) If the maximum temperature of the sampling gas mentioned in 5.2, 5.3 and 5.4 (filter/condenser method: 125 °C; dilution method: 40 °C; cooled probe method: 20 °C) is exceeded it shall be validated. This validated temperature shall not be exceeded during sampling.
- h) A leak check shall be performed before and after each sampling procedure according to 7.2.4.1 (see also D.3.4).
- i) A validation trial shall be carried out to demonstrate the comparability of the long-term method against the standard reference methods and shall be performed as follows:
 - 1) The sampling points of the two methods shall be as close as possible without any interference.
 - 2) For this validation trial the requirement of grid measurements according to EN 1948-1 are not required.

¹⁾ XAD-2 and Porapak PS are examples of suitable commercially available products. This indication is only made for information of the user of this European Technical Specification and does not constitute an endorsement by CEN of the product named. Equivalent products may be used.

- 3) The conditions for the validation of the long-term method and the standard reference methods shall be identical according to the specifications of the long-term measurements system apart from the sampling duration specified in 3.2).
- 4) Long-term sampling and standard reference sampling according to EN 1948-1:2006, 7.2, a) and 7.2, b) shall be performed in parallel during a specified time period (at least 40 h). The long-term sampling as well as the standard reference sampling is performed for 6 h to 8 h. The sampling unit including the filter of the standard reference methods are exchanged, whereas the sampling unit including the filter of the long-term method are kept for the specified time period. This results in one sample for the long-term method and a mean value of multiple, at least five samples for the standard reference methods.
- 5) During interruption of the sampling, the sampling probe of the long-term sampling system shall be secured against any contamination. This should be done in the same way as during regular interruptions in the sampling process, e.g. by thermal desorption and reverse flow purging or by closing the nozzle, if appropriate after having removed the probe.
- 6) At the end of this validation trial the probes of both systems shall be rinsed.
- 7) The difference between the mean value of the multiple samples of the standard reference methods and the single long-term sample shall be within $\pm 35\%$ of the value determined by the standard reference methods based on the corresponding I-TEQ/WHO-TEQ value. This data refers to measurement results in the range of $0,1 \text{ ng I-TEQ/m}^3$ (or WHO-TEQ) values at standard conditions (dry). If the measurement results are much lower than $0,1 \text{ ng I-TEQ/m}^3$ (or WHO-TEQ) the relevance of deviations between SRM result and the long-term sampling systems will be checked according to the normative Annex I.
- 8) These comparison measurements shall be performed by laboratories fulfilling the requirements of EN ISO/IEC 17025 [4].

Additional minimum requirements for individual systems are given to avoid increase of uncertainty.

j) Filter/condenser method

- 1) The probe temperature shall be adjusted to a temperature higher than the water dew point.
- 2) The filter shall adjusted to a temperature below 125°C in order to avoid chemical reactions on activated surfaces and shall be kept over the dew point to avoid condensation.
- 3) The condensate shall pass through the adsorbent.

k) Dilution method

- 1) Dilution air shall be filtered using a filter system with $99,5\%$ efficiency for $0,3 \mu\text{m}$ particulates and with efficiency $> 90\%$ for PCDDs/PCDFs/PCB. The filter system has to be exchanged at least once a year.
- 2) The probe temperature shall be adjusted to a temperature higher than the dew point.

l) Cooled probe method

- 1) The condensate shall pass through the sampling unit.

7.2 Validation of the installation/functioning on each plant to be fulfilled by the plant operator

7.2.1 Long-term PCDD/PCDF/PCB sampling systems using sampling units which have to be prepared in the laboratory

- a) Before use, the sampling unit has to be cleaned carefully to avoid any contamination (see EN 1948-1).
- b) The sampling standard shall be added to the filter and/or the adsorbents of the sampling unit.
- c) The prepared sampling unit shall be securely sealed against contamination and placed in clean containers for transport.
- d) The filter efficiency shall be higher than 99,5 % on a test aerosol with a mean particle diameter of 0,3 µm, at the maximum flow rate anticipated (or 99,9 % on a test aerosol of 0,6 µm mean diameter). This efficiency shall be certified by the filter supplier.
- e) The overall deposits in the sampling train shall be determined at each installation. More than 90 % of the total I-TEQ and WHO-TEQ respectively shall be found in the analysed compartments of the sampling train. This means that less than 10 % of total I-TEQ/WHO-TEQ are acceptable to be found in sum in the breakthrough sample (as determined during the type performing test) and in the deposit of the probe.
- f) The O₂ measurement data can be taken from the plant (measured by a certified measurement device). In this case the O₂ probe should be situated near to the PCDD/PCDF/PCB sampling probe or representative O₂ comparative measurements shall be performed.

7.2.2 Minimum requirements for set-up

- a) All available information on the plant relevant to estimate the emission behaviour shall be considered to configure the long-term sampling system. The requirements of EN 15259 shall be considered.
- b) The measurement section shall allow the sampling and execution of measurements at a suitable measurement cross section and shall be selected according to EN 15259:2007, Clause 6.
- c) The system shall be assembled and set-up according to the manufacturer's installation instructions.
- d) Deposits in the probe should be minimized by the following measures:
 - 1) minimization of the probe length in dependency of the sampling point (see Annex F);
 - 2) for dust concentrations > 10 mg/m³ the minimum flow velocity in the probe should be at least 10 m/s and the inner diameter of the probe shall be adapted accordingly.
- e) All relevant parameters listed in 7.3.2 shall be recorded as a reference basis for the upcoming maintenance.

7.2.3 Minimum requirements for selecting the sampling point

- a) Sampling should be done at representative position(s) in the duct. The measurement for identifying the representative sampling point(s) shall be performed during normal plant operating conditions. EN 15259:2007, 7.2.2 (see Annex F) shall be considered.
- b) The determination of the representative sampling point(s) shall be performed depending on the dust concentration of the plant.

- c) If the dust concentration in the flue gas is below 10 mg/m^3 (reference conditions) and the requirements of EN 15259:2007, 6.2.1 c), are fulfilled, the determination of the representative sampling point(s) shall be performed according to EN 15259:2007, 8.4 for gaseous components (e.g. O_2 or NO_x) (see Annex H).
- d) Any existing data of flow velocity and the oxygen profile from the plant may be used for selecting the sampling point.
- e) If the dust concentration in the waste gas exceeds 10 mg/m^3 (reference conditions) or the requirements of EN 15259:2007, 6.2.1, c) are not fulfilled, the representative sampling point(s) determined according to 7.2.3, c) shall be confirmed by a minimum of three comparison measurements of dust. These dust comparison measurements shall be performed simultaneously as a single point measurement at the selected representative sampling point(s) and as a grid measurement according to EN 13284-1. The acceptable deviation Δ between the two average measurements (average value of the measurement at the representative sampling point and the average value of the three comparison measurements) shall be below or equal to the uncertainty of the method for the determined concentration.
- f) In order to confirm the selected sampling point(s) and the proper configuration of the long-term sampling system, comparison measurements of PCDD/PCDF with the standard reference methods according to EN 1948-1:2006, 7.2, a) and 7.2, b) shall be performed for 6 h to 8 h.

The difference between the mean value of the multiple samples of the standard reference methods and the single long-term sample shall be within $\pm 35 \%$ of the value determined by the standard reference methods based on the corresponding I-TEQ/WHO-TEQ value. This data refer to measurement results in the range of $0,1 \text{ ng I-TEQ/m}^3$ (or WHO-TEQ) values. If the measurement results are much lower than $0,1 \text{ ng I-TEQ/m}^3$ (or WHO-TEQ) the relevance of deviations between SRM result and the long-term sampling systems will be checked according to the normative Annex I.

- g) The nozzle(s) should be placed as close as possible to the selected sampling point(s). If several representative points are identified, the point which corresponds to the shortest probe should be selected in order to minimize dust deposits in the probe. Nozzle positioning close to the sampling point for O_2 and flanges for reference measurements of the plant is preferable.

7.2.4 Minimum requirements for sampling

7.2.4.1 Leak test

The integrity of the all connections and the tightness of the system shall be tested by a leak check after set-up, before and after each sampling procedure. The leak check is performed in the following way:

- a) For set-up and maintenance the complete sampling train shall be assembled including the nozzle and shall be checked by a leak check according to EN 1948-1:2006, 7.2, c). The leak rate shall be less than 5 % of the normal flow rate.
- b) A leak check before and after each sampling procedure shall be performed. The leak test should include the complete assembled train including the nozzle and the sampling probe. The leak rate shall be less than 5 % of the normal flow rate.

If it is not possible to include the nozzle and the sampling probe into the leak check it can be accepted that the leak check only needs to include all parts, which are installed newly before the start (e.g. sampling unit), and all parts downstream of the sampling unit, adsorbent and/or filter. The leak rate shall be less than 5 % of the normal flow rate. In this case the tightness of the complete system should be checked according to a) at least once a year.

During the sampling it is recommended to monitor the tightness of the system by continuously measuring oxygen, humidity and/or pressure.

7.2.4.2 Isokinetic sampling

- a) Isokinetic sampling shall be performed. To ensure isokinetic sampling an alarm system shall be installed to indicate if the isokinetic rate differs by more than -5% to $+15\%$ from the ideal isokinetic condition. If the deviation of the isokinetic rates cannot be kept between these limits for more than 15 min, then the system should stop the sampling and the stop should be indicated by an alarm signal. The alarm should be included in the sampling protocol.
- b) Preferably, the isokinetic sampling should be done according to EN 13284-1:2001 using a nozzle with minimum diameter of 6 mm.
- c) If the use of a 6 mm nozzle is not possible, the nozzle diameter can be reduced to a minimum of 3 mm under the following prerequisites:
 - 1) Particle concentration $< 10 \text{ mg/m}^3$ under normal plant operating conditions (demonstrated e.g. by online particle monitoring systems of the plant).
 - 2) This deviation from the requirements described in EN 13284-1 has to be validated, explained and reported.
- d) According to EN 13284-1:2001, 6.2.4 (see also Annex E), the uncertainty of the effective sampling area of the nozzle should be less than 10 % in order to fulfil isokinetic sampling.
- e) The nozzle diameters used and the corresponding sampling volume shall be validated in the method validation tests (see 7.1).
- f) Any deposition on the nozzle entry shall be minimized in order to ensure isokinetic sampling. Depending on the plant emissions the cleaning frequencies of the nozzle shall be increased with regard to the normal maintenance frequencies (see 7.1, f)).

7.2.4.3 Further minimum requirements for sampling

- a) The sampling train is spiked with $^{13}\text{C}_{12}$ -labelled standards specified in Table 3 and Table 4. The spiked parts of the equipment depend on the method used and are specified in EN 1948-1:2006, 7.2 f).
- b) The recovery rate of each sampling standard shall be greater than 50 % calculated on the basis of the relevant extraction standard (see EN 1948-3).
- c) In case that XAD-2 is used, the XAD-2 cartridge shall be mounted in a vertical direction in order to avoid channels. Additionally, the flue gas shall flow from top to bottom of the XAD-2 cartridge.
- d) The maximum adsorber temperature of the long-term sampling system during normal operation shall not exceed the maximum adsorber temperature tested during the method validation.
- e) The permissible limit of quantification (LOQ_i) for the individual congener i shall be as follows:

The field blank shall be less than 10 % of the limit value; if emission values are far below the limit value (e.g. 5 % to 10 % of the limit value) are less or in the same order than the field blank value, the result shall be reported as less or equal to the blank. For the particular case of the field blanks it is not allowed to aliquote the field blank extracts.

If analysing an aliquot as recommended in 9.1 it should be considered that the LOQ_i is increased by the factor of dilution. A field blank shall be performed twice a year for the plant in the following way:

- The blank sample comprises the complete sampling unit consisting of the dust filter and adsorber.
- A new adsorbent unit is inserted as blank sample.

- No gas is drawn through the sampling train.
- A leak check is performed.
- The sampling unit is dismantled and analysed.

If only PCDD/PCDF have to be measured the results are reported as I-TEQ and in case of PCDD/PCD/PCB measurements the result has to be reported as WHO-TEQ.

The permissible limits of quantification (LOQ) for the individual congener i shall be as follows:

$$LOQ_i \leq \frac{0,5 \text{ pg} / \text{m}^3}{I - TEF_i} \quad (1)$$

where

LOQ_i is the limit of quantification for congener i ;
 $I-TEF_i$ is the international toxic equivalency factor of congener i

- f) After exceptional plant conditions (e.g. high dust load in flue gas due to maintenance work) the parts of the sampling train coming into contact with the exhaust gas shall be cleaned according to Annex B. An additional rinsing shall be performed and stored. If the following sample gives abnormal results the rinsing solution can be analysed to prove the cleanness of the sampling train.
- g) For long-term sampling systems rinsing of the sampling train upstream of the first sampling unit after each sampling is not mandatory but shall be performed once a year according to EN 1948-1:2006, 9.3.5.1, and the rinsing solution shall be analysed. If the rinsing is performed more often than once a year the rinsing solutions shall be combined and analysed. More than 90 % of the total annual mass in I-TEQ and WHO-TEQ respectively shall be found in the analysed compartments of the sampling train. This means that less than 10 % of total I-TEQ and WHO-TEQ respectively is acceptable to be found in sum in the breakthrough sample and in all compartments not analysed in routine measurements.
- h) The deposits in the probe can be minimized by the following measures:
 - reduction of the length of the probe;
 - enhancement of flow velocity;
 - rinsing (solution is to be added to the analysed sample);
 - thermal desorption (mass to be included in the sample).

The deposits in the sampling line upstream the first sampling unit should be determined at least once a year. The mass of the PCDDs/PCDFs/PCBs in the rinsing solution shall be documented in every measurement report as absolute mass value and in terms of the percentage of the corresponding total mass I-TEQ and WHO-TEQ respectively. The loss shall be lower than 10 % (see 7.2.1, e)). If this requirement is not fulfilled, the sampling train shall be checked. A correction of measured values by the quantified loss of less than 10 % is not performed.

- i) The PCDD/PCDF/PCB sampling system shall be able to be operated at a minimum velocity of 3 m/s. (or differential pressure of 5 Pa, as specified by EN ISO 16911-1).
- j) Depending on the application and the operating conditions which were defined before for this specific application the sampling system should be able to stop or break the sampling if limit values (e.g. velocity

and/or temperature of flue gas, static pressure in stack, O₂ and/or CO₂ content of flue gas, temperature of the adsorption cartridge, etc.) are exceeded.

Each of these breaks has to be registered in the sampling protocol with reason of the break and the total period of how long the break applied.

7.2.4.4 Minimum requirements for data storage system

The measurement data storage system shall store all necessary information regarding sampling, sampling conditions, plant conditions and identification of plant and sample at a suitable data registration rate.

For quality assurance the long-term PCDD/PCDF/PCB sampling system shall record relevant measurement parameters at least every 30 min as 30 min mean values. Shorter record periods can be used, whereby then the mean value calculation should be adapted to the record period (e.g. 15 min mean value if record period is 15 min). Interruptions, events and standby periods have to be recorded at the time they occur. All other parameters are recorded in addition then for the time period since the last record.

As minimum the following parameters shall be recorded and reported:

- a) flue gas velocity (mean value);
- b) mean flue gas temperature during the last period;
- c) maximum adsorber temperature during the last period;
- d) flue gas pressure;
- e) total sampled volume in standard conditions dry;
- f) identifiers for the different kind of records (start/end of sampling, interruptions, events);
- g) maximum filter temperature during the last period, if appropriate, depending on sampling technique;
- h) temperature of the sampling probe, if appropriate;
- i) flue gas humidity, if appropriate;
- j) O₂ content of the flue gas;
- k) dilution factor, if appropriate.

Additional parameters could be stored if necessary or helpful, e.g.

- l) minimum/maximum flue gas temperature during the last period;
- m) temperature at the gas meter (or volume measurement device);
- n) total pressure at the gas meter (or volume measurement device);
- o) density factor of gas meter (or volume measurement device);
- p) collected condensate value;
- q) CO₂ content.

If substitute values are used for any of the above mentioned parameters it shall be also reported in the sampling protocol (see 3.24). The recording should be done in record sets suitable for automatic processing

during data evaluation and reporting. This means the structure of each record set has to be equal. Changes in the data structure within the same recording table should be avoided. Data processing shall be possible by using normal database programs.

The long-term PCDD/PCDF/PCB sampling system shall have an interface to download the recorded data, e.g. to a computer and/or external storage device.

7.3 Minimum requirements for on-going operations on each plant to be fulfilled at regular time intervals by the plant operator

7.3.1 Regular check-up

During the exchange procedure of the sampling unit a plausibility check of the following parameters shall be performed.

- a) Check of the volume measurement device (i.e. gas meter, flow meter, flow-control unit) is still functional.
- b) Visual inspection of the adsorption cartridge i.e. colour, dust content, contamination.
- c) Check the proper operation of the adsorption cartridge sealing, when the cartridge is changed.
- d) Visual inspection of the complete system in relation of contamination, tightness of connections and hoses.
- e) A field blank shall be performed twice a year in the following way:
 - 1) The blank sample comprises the complete sampling unit consisting of the dust filter and adsorber. (apart from the sampling device upstream of the first sampling unit).
 - 2) A new adsorbent unit is inserted as blank sample.
 - 3) No gas is drawn through the sampling train.
 - 4) A leak check is performed.
 - 5) The sampling unit is dismantled.

7.3.2 Maintenance

- a) A maintenance procedure shall be performed once per year.
- b) The system shall be dismantled and all parts upstream of the sampling unit coming into contact with the flue gas shall be cleaned. But the requirements of 7.2.4.3, g) shall be fulfilled.
- c) The integrity of the nozzle to probe connection shall be checked.
- d) The maintenance procedure shall also include the following devices and parameters, to be calibrated where appropriate:
 - 1) device(s) for monitoring and controlling the isokinetic conditions, i.e. zero point of differential pressure sensor, static pressure sensor, plugs and wires of temperature sensors,
 - 2) volume measurement device,
 - 3) temperature sensor at the sampling unit,
 - 4) vacuum pump,