

# Method Implementation Document (MID 1948)

BS EN 1948 Parts 1-3: 2006

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

Part 1: Sampling of PCDDs/PCDFs

Part 2: Extraction and clean-up of PCDDs/PCDFs

Part 3: Identification and quantification of PCDDs/PCDFs

Environment Agency

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Version 2.2





## Record of amendments

Version number	Date	Amendment
1.1	Nov 2006	Clarified relationship between sample flow rate and nozzle diameter (section 7.2)
2	Sept 2008	Added information on measuring resin trap temperatures (section 6.2.2)
		Specified sample train cleaning requirements (section 6.3)
		Added note about the use of oxygen measurements to detect leaks in sampling equipment (section 7.2c)
		Added information about the use of ice to cool stack gas before entering the resin trap (section 7.4)
		Amended text to reflect changes to sample train cleaning requirements (B4.1)
		Clarified that the first impinger (condensate flask) shall be cleaned using the procedure given in section 6.3 (section B4.3)
		Clarified that the condensate collected in the first impinger shall be analysed (section B4.9)
2.1	Oct 2008	Added clarification about assembly of sampling equipment (Section 9.3.3)
		Added clarification on measuring the resin trap temperature (Section 6.2.2)
		Clarified requirements for cleaning probe liners (Section 6.3).
2.2	Feb 2010	Update on cleaning glassware (Section 6.3)
		The 5% leak check requirement in the standard is acceptable. Deleted 2% leak check requirement specified by previous MID's (Section 7.2 & Annex B).

## Status of this document

This MID may be subject to review and amendment following publication. The most recent version of this MID is available on our website at:

[www.mcerts.net](http://www.mcerts.net)

## Implementation date

It is expected that organisations, which hold MCERTS accreditation for BS EN 1948-1, will have met the requirements of this MID by March 2010.

## Feedback

If you have any comments on this document please contact Rupert Standing at [rupert.standing@environment-agency.gov.uk](mailto:rupert.standing@environment-agency.gov.uk)

## **Role of Method Implementation Documents**

The Environment Agency recognises that European and International standards may need supplementing by Method Implementation Documents (MIDs) to ensure they are being implemented consistently.

We have established our Monitoring Certification Scheme: MCERTS to deliver quality environmental measurements. Organisations wishing to include a standard in their schedule of MCERTS accreditation shall follow the requirements of the standard and, where available, the associated MID.

It may not be necessary to produce a MID for every standard but where required they will be used to supplement standards called up by Technical Guidance Note M2. MIDs provide details on how the preferred standards shall be used for regulatory monitoring.

MIDs are produced in collaboration with the Source Testing Association (STA) and its members.

Further information on MCERTS including copies of performance standards and guidance can be obtained from our web-site at:

[www.MCERTS.net](http://www.MCERTS.net)

Copies of MIDs are available from the STA web-site at:

[www.S-T-A.org](http://www.S-T-A.org)

## Contents

Foreword .....	1
Introduction .....	1
<b>Part 1: Sampling of PCDDs/PCDFs.....</b>	<b>1</b>
1 Scope .....	1
2 Normative references .....	2
3 Terms and definitions .....	2
4 Symbols and abbreviations.....	2
5 Principle of the complete PCDD/PCDF measurement procedure.....	2
6 Sampling device and materials.....	3
7 Minimum requirements for sampling .....	5
8 Safety measures and transport.....	6
9 Preparations and sampling.....	6
Annex A (informative) Toxicity and toxic equivalency .....	8
Annex B Examples of operation.....	8
Annex C Examples of adsorbents and their preparations and cleaning.....	10
Annex D (informative) Sampling measurement record.....	10
Annex Za (informative) Clauses of this European Standard addressing essential requirements or other provisions of EU Directives. ....	10
<b>Part 2: Extraction and clean-up of PCDDs/PCDFs .....</b>	<b>11</b>
1 Scope .....	11
2 Normative references .....	11
3 Terms and definitions .....	11
4 Symbols and abbreviations.....	11
5 Principle of the extraction and clean-up procedure.....	11
6 Device, materials and <sup>13</sup> C <sub>12</sub> -labelled standards .....	11
7 Method validation and quality control requirements.....	11
8 Sample pre-treatment and extraction.....	12
9 Report.....	13
Annex A (informative) Examples of operation of extraction and clean-up methods.....	13
Annex Zz(informative) Clauses of this European Standard addressing essential requirements or other provisions of EU Directives .....	13

<b>Part 3: Identification and quantification of PCDDs/PCDFs .....</b>	<b>14</b>
<b>1 Scope .....</b>	<b>14</b>
<b>2 Normative references .....</b>	<b>14</b>
<b>3 Terms and definitions .....</b>	<b>14</b>
<b>4 Symbols and abbreviations.....</b>	<b>14</b>
<b>5 Principles of identification and quantification .....</b>	<b>14</b>
<b>6 Reagents, materials and equipment.....</b>	<b>14</b>
<b>7 Safety measures .....</b>	<b>14</b>
<b>8 Quality control requirements for identification and quantification.....</b>	<b>14</b>
<b>9 Quality assurance criteria for extraction/clean-up/quantification procedure blanks .....</b>	<b>15</b>
<b>10 Calibration of the HRGC/HRMS .....</b>	<b>15</b>
<b>11 Quantification of HRGC/HRMS results .....</b>	<b>15</b>
<b>12 Calculation of the measurement results .....</b>	<b>15</b>
<b>13 Analytical report.....</b>	<b>16</b>
<b>14 Performance characteristics.....</b>	<b>16</b>
<b>15 Interferences .....</b>	<b>16</b>
<b>Annex A - Zz (informative) Examples of operation of gas chromatography / mass spectrometry analysis of PCDDs/PCDFs in emission samples .....</b>	<b>16</b>
<b>Annex B (informative) Estimation of the measuring uncertainty and precision of the determination of polychlorinated dibenzo-p-dioxins and dibenzofurans.....</b>	<b>16</b>
<b>Annex C (informative) Mass of ions monitored for PCDDs and PCDFs.....</b>	<b>16</b>
<b>Annex D (informative) Relative abundance of chlorine isotope ions and their ratio for PCDD/PCDF congeners containing 4 to 8 chlorine substituents.....</b>	<b>17</b>
<b>Annex E (informative) Further general recommendations for separation, detection and quantification of PCDDs/PCDFs.....</b>	<b>17</b>
<b>Annex F (informative) Variability determination based on the results of the validation tests .....</b>	<b>17</b>
<b>Annex Zz(informative).....</b>	<b>17</b>
<b>Clauses of this European Standard addressing essential requirements or other provisions of EU Directives. ....</b>	<b>17</b>

## Foreword

This MID follows the numbering in the standards and like the standard is split into three parts. However, unlike the standard these are combined in the same document. The three standards must be read and used in conjunction with one another. It is also necessary for the sampling organisation to liaise with the analytical laboratory.

## Introduction

No additional comments

## Part 1: Sampling of PCDDs/PCDFs

### 1 Scope

BS EN 1948 Stationary source emissions — Determination of the mass concentration of PCDDs/PCDFs and dioxin-like PCBs (referred to in this document as EN 1948) is published in three parts:

- Part 1: Sampling of PCDDs/PCDFs
- Part 2: Extraction and clean-up of PCDDs/PCDFs
- Part 3: Identification and quantification of PCDDs/PCDFs

Part 1 of the standard includes several different approaches to sampling:

- Filter/condenser method
- Dilution method
- Cooled probe method

The standard also includes the following approaches to the filter condenser method:

- Liquid absorbent downstream of the condensate flask
- Solid absorbent downstream of the condensate flask
- Solid absorbent upstream of the condensate flask
- Flow division to enable side stream sampling to be carried out

This MID provides information on the filter/condenser method<sup>1</sup>, using a solid absorbent upstream of the condensate flask, without flow division.

In order to obtain MCERTS accreditation to measure PCDDs/PCDFs it is necessary to meet the requirements of EN1948 Part 1 and this MID. This MID specifies that the requirements of the filter condenser method (See Annex B, section B4) of the standard shall be met.

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<sup>1</sup> In 2006 the STA carried out a questionnaire of its members with UKAS accreditation to use BS EN 1948. This confirmed that sampling organisations in the UK use the filter condenser method. The equipment used follows the specifications outlined in US EPA method 23.

Furthermore, it is necessary for the analytical laboratory to be accredited for the analysis specified in EN1948 Part 2 -3 and in this MID. This should be stated on the analytical laboratory's schedule of accreditation.

A test carried out to measure PCDDs/PCDFs cannot also be used to measure PAHs<sup>1</sup>.

## **2 Normative references**

MID 13284-1, Environment Agency.

MCERTS for manual stack emission monitoring - performance standard for organisations, Environment Agency.

## **3 Terms and definitions**

No additional information

## **4 Symbols and abbreviations**

No additional information

## **5 Principle of the complete PCDD/PCDF measurement procedure**

### **5.1 Sampling**

#### **5.1.1 General**

This MID specifies the use of the filter/condenser method, using solid absorbent upstream of the condenser, without flow division.

#### **5.1.2 Filter/condenser method**

No additional comments

#### **5.1.3 Dilution method**

This approach is not relevant to this MID.

#### **5.1.4 Cooled probe method**

This approach is not relevant to this MID.

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<sup>1</sup>There is currently no CEN or ISO procedure that combines the measurement of PCDDs/PCDFs with PAHs. Without validation evidence and an agreed industry wide procedure it is not possible to obtain MCERTS accreditation for a combined method. PAH should be measured using ISO 11338.

## **5.2 Extraction and clean-up**

No additional comments

## **5.3 Identification and quantification**

No additional comments

# **6 Sampling device and materials**

## **6.1 General sampling device and sub sections**

No additional requirements

## **6.2 Materials**

### **6.2.1 General**

No additional requirements

### **6.2.2 Sampling system**

Titanium may be used. Commercial titanium is made to different specifications based on the purity of the titanium. An appropriate grade of titanium shall be used.

Titanium will degrade in gas streams with a fluoride content of greater than 20ppm.

Titanium may degrade at temperatures greater than 350°C (refer to supplier's information).

The resin trap shall be designed to allow the gas temperature within the resin trap to be measured. However, it is not practical to install the thermocouple within the gas stream itself. Instead the resin trap outer wall shall have an indentation in it. The thermocouple shall be inserted into the indentation, so that a temperature that is representative of the gas inside the resin trap can be obtained.

### **6.2.3 Sealings (in contact with flue gas)**

PTFE must not be used in temperatures above 200°C (refer to supplier's information).

### **6.2.4 Filter**

Quartz filters are recommended.

### **6.2.5 Quartz wool (if used)**

The use of quartz wool is not recommended.

### **6.2.6 Solid adsorbent**

An XAD-2 resin filled container (resin trap) is used for the method described by this MID.

### **6.2.7 Cooling fluid**

Filter/condenser method uses an ice bath, with a re-circulating pump submerged in iced water for cooling the flue gas and resin trap.

### **6.2.8 Ice bath**

No additional requirements

### **6.2.9 Silica gel**

Indicating silica gel shall be used to ensure moisture does not break through to the control box and dry gas meter.

### **6.2.10 Reagents**

No additional requirements

## **6.3 Cleaning the sampling device**

The standard gives three cleaning procedures. Cleaning procedure number (1) shall be carried out before a sampling campaign. The solvent wash and treatment in a muffle oven shall be carried out.

The muffle oven shall be at 400 to 480°C, except when using titanium. When using titanium the temperature shall be between 300 to 380°C.

The sampling equipment shall be kept in the muffle oven for at least three hours. Due the length of probe linings it is not practical to treat them in a muffle oven. The probe liners shall be heated within their outer sheaves to the maximum temperature practical for the probe.

Note: due to limitations on the probe heaters it is not usually practical to heat probes to between 300 to 380°C.

It is permissible to use an alternative cleaning procedure if an analytical laboratory provides evidence to UKAS that the procedure is equivalent to the procedure specified in this MID.

Note 1: consistently low field blank results help demonstrate that an alternative cleaning procedure is effective.

Note 2: an alternative cleaning procedure may, for example, include prolonged exposure to UV light.

## **7 Minimum requirements for sampling**

### **7.1 Method validation criteria**

- a) No additional requirements
- b) The absorbent material (XAD-2) shall be sufficiently well packed and evenly distributed in the resin trap to prevent the formation of channels, which stack gas may pass through without coming into close contact with the absorbent.
- c) In order to validate the adsorption stage efficiency for gaseous PCDDs/PCDFs, it is necessary to put two resin traps in sequence.

### **7.2 Minimum requirements for sampling**

- a) BS EN13284-1 states that nozzles with an internal diameter of less than 6mm shall be avoided. The sample train used for the filter condenser method\* (see Annex B4) is designed to have a sample gas flow rate of around 5 – 25 l/min. At high stack gas velocities (typically above 25 m/s) it may be necessary to use a nozzle with a diameter of less than 6mm in order to remain within the designed flow rate of the sampling train.

If a nozzle with a smaller diameter than 6mm is used:

- sufficient sample volume shall be extracted to meet the limit of detection required by the analytical laboratory;
- justification for the deviation from EN 13284-1 shall be provided in the monitoring report.

- b) No additional requirements

- c) No additional requirements

Note: as measurements can be required for several hours it is good practice to have a system in place that will detect leaks during the sample run. This can be done on combustion processes by using an analyser to measure the oxygen concentration of the gas exiting the isokinetic sampling equipment and comparing it to the dry stack gas oxygen concentration. An increase in the oxygen concentration from the isokinetic sampling equipment relative to the dry stack gas oxygen may indicate a leak. If this occurs the sample run can be stopped and the leak rectified before continuing with the measurement.

- d - k) No additional requirements

### **7.3 Field blank requirements**

The field blank shall be performed before a measurement series (i.e. consecutive measurements on one stack). However, it is acceptable to apply a field blank to more than one stack provided the stacks are in the same location (i.e. sampling platform) and

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\* Variant with solid absorbent upstream of the condensate flask

the process conditions are the same. In order to apply a blank to more than one stack in the same location, the glassware used shall:

- be the same as used on the previous stack; or
- shall have been prepared/cleaned in the permanent laboratory at the same time and conditions as the glassware used on the previous stack.

#### **7.4 Additional sampling minimum requirements for the filter/condenser method**

During the production of a Site Specific Protocol<sup>1</sup> it is important to specify whether an in or out of stack filter will be used.

In the UK it is general custom and practice to use an out of stack filter system to measure PCDDs/PCDFs. Annex B of this document shows the sample train with an out of stack filter configuration.

The resin trap temperature shall be kept below 20°C throughout the measurement. The sampling team shall have planned in advance where they will obtain ice and how much ice is required for the measurement. The amount of ice required shall be recorded in the SSP.

Note: the amount of ice required is based on the stack gas temperature, the duration of the test and the moisture content of the stack gas.

Note: it is strongly recommended that the sampling team have access to an ice making machine to ensure they have sufficient ice available throughout the measurement.

#### **7.5 Additional sampling minimum requirements for the dilution method**

This approach is not applicable to this MID.

#### **7.6 Additional minimum requirements for the cooled probe method**

This approach is not applicable to this MID.

### **8 Safety measures and transport**

No additional requirements

### **9 Preparations and sampling**

#### **9.1 Advance preparations at the sampling site**

##### **9.1.1 Selection of suitable sampling location**

No additional requirements

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<sup>1</sup> Refer to MCERTS for manual stack emission monitoring - performance standard for organisations

## **9.2 Advance preparations at the laboratory**

### **9.2.1 Preparation of equipment**

No additional requirements

### **9.2.2 Preparation of parts of the sampling train in contact with the sample**

No additional requirements

## **9.3 Sampling at the plant**

### **9.3.1 Initial checks**

No additional requirements

### **9.3.2 Preliminary survey**

No additional requirements

### **9.3.3 Assembly of apparatus**

See section 7.2 of this MID for information on selecting nozzle sizes.

The following components of the sample train shall be prepared/assembled and disassembled/recovered in a clean area:

- filter and filter holder
- condenser, resin trap and impinger train glassware

When transferring the sample train or train components from and back to the clean area all openings shall be sealed.

Note due to safety issues it may not be practical to assemble the entire sample train in a clean area before carrying the equipment to a clean area.

### **9.3.4 Sampling**

#### **9.3.4.1 Record keeping**

No additional requirements

#### **9.3.4.2 Changing sampling line**

No additional requirements

### **9.3.5 After sampling**

#### **9.3.5.1 Sample recovery from the sampling train**

When working on a platform only the probe and filter housing should be disconnected at a location other than the designated clean area. All other parts shall be disassembled at a designated clean area. An exposed working platform should not be used as a designated clean area.

#### **9.3.5.2 Sample storage**

No additional requirements

### **9.4 Calculation of flue gas sample volume**

No additional requirements

### **9.5 Sampling report**

If carrying out MCERTS accredited work the monitoring report shall be submitted in the report format specified in the Manual stack emission monitoring performance standard for organisations (Annex H) available from [www.mcerts.net](http://www.mcerts.net). The report shall also include specific requirements required by EN1948 and this MID.

### **Annex A (informative) Toxicity and toxic equivalency**

No additional information

### **Annex B Examples of operation**

#### **B1 General**

Annex B in the standard is informative, as it contains several different examples of operation. These examples have been validated by CEN. This MID specifies that the requirements of Section B4 shall be followed.

#### **B4 Filter/condenser method - Variant with solid adsorbent upstream of the condensate flask**

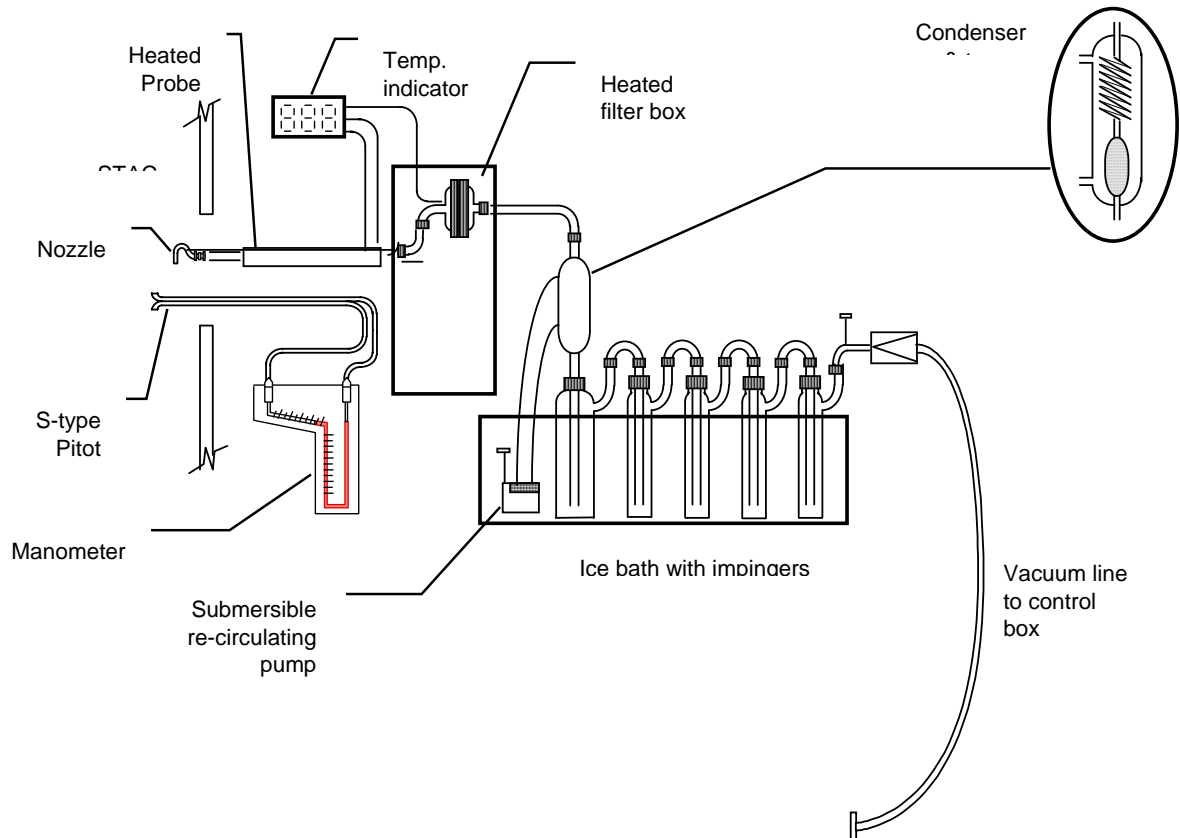
##### **B4.1. Summary of apparatus design**

Equipment used in the UK to sample PCDDs/PCDFs differs from the example shown in Figure B5 of the standard. Figure 1 below shows the configuration of equipment commonly used in the UK<sup>1</sup>.

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<sup>1</sup> Equipment used in the UK follows the specifications outlined in US EPA method 23.

Section B.4.1 of the standard specifies the use of a glass-lined probe. It is also acceptable to use titanium (see Section 6.2.2).



**Figure 1 Dioxin sample train**

#### **B4. 2. Adsorbent and cleaning procedure**

No additional requirements

#### **B4. 3. Sampling train cleaning procedure before sampling**

The cleaning procedure given in Section 6.3 of this MID shall be used. The first impinger (condensate flask) shall also be cleaned using the procedure given in Section 6.3.

#### **B4. 4. Spiking position**

B4 specifies that the filter is spiked. It is acceptable to spike the resin trap only or to spike both the resin trap and the filter.

#### **B4. 5. Equipment assembly procedure**

No additional requirements

#### **B4. 6. Leak check procedure**

No additional requirements

#### **B4. 7. Sample gas flow rate control**

No additional requirements

#### **B4. 8. Sampling**

No additional requirements

#### **B4. 9. Equipment disassembly procedure**

The condensate collected in the first impinger (condensate flask) shall be stored for analysis.

### **Annex C Examples of adsorbents and their preparations and cleaning**

Annex C in the standard is informative, as it contains several different examples. This MID specifies that the requirements of Section C.2 XAD-2 shall be followed.

### **Annex D (informative) Sampling measurement record**

No additional information

### **Annex Za (informative) Clauses of this European Standard addressing essential requirements or other provisions of EU Directives.**

No additional information

## **Part 2: Extraction and clean-up of PCDDs/PCDFs**

### **1 Scope**

It is important that close co-operation takes place between sampling and analytical laboratories. Documented evidence for this shall be available.

### **2 Normative references**

No additional requirements

### **3 Terms and definitions**

No additional information

### **4 Symbols and abbreviations**

No additional information

### **5 Principle of the extraction and clean-up procedure**

#### **5.1 Principles**

No additional information

#### **5.2 Minimum information prior to analyses**

No additional information

### **6 Device, materials and $^{13}\text{C}_{12}$ -labelled standards**

#### **6.1 Device and materials**

No additional requirements

#### **6.2 $^{13}\text{C}_{12}$ -labelled standards**

Standards cannot be reliably split into different sampling train components based upon presence of native PCDDs/PCDFs, as there is unlikely to be any knowledge of how they are distributed. It is acceptable to use the masses provided in Table 1.

### **7 Method validation and quality control requirements**

#### **7.1 Use of $^{13}\text{C}_{12}$ -labelled standards**

No additional requirements

## **7.2 Sample pre-treatment**

No additional requirements

## **7.3 Clean-up**

### **7.3.1 Method validation**

No additional requirements

### **7.3.2 Minimum requirements for measurements**

No additional requirements

## **8 Sample pre-treatment and extraction**

### **8.1 Addition of extraction standards**

Standards cannot be reliably split into different sampling train components based upon presence of native PCDDs/PCDFs, as there is unlikely to be any knowledge of how they are distributed. It is acceptable to use the masses provided in Table 1.

### **8.2 Sample storage**

Brown glass bottles with screw-caps and PTFE-lined seals shall be used. Traps and filters may be wrapped in foil to exclude light.

### **8.3 Extraction**

a) Some particulates on filters are extremely hydrophobic, which may mean that the pre-treatment with hydrochloric acid does not wet the entire surface but forms droplets on the surface of the filter.

### **8.4 Clean-up**

Column chromatography on alumina may be used to separate PCBs from PCDDs/PCDFs.

### **8.5 Final concentration of the sample extract**

No additional requirements

### **8.6 Addition of recovery standards**

The text in the standard stating that recovery standards are used to measure the recovery of the sampling standards should state that recovery standards are used to measure recovery of the final quantification step.

## **9 Report**

No additional requirements

### **Annex A (informative) Examples of operation of extraction and clean-up methods**

No additional requirements

### **Annex Zz(informative) Clauses of this European Standard addressing essential requirements or other provisions of EU Directives**

No additional information

## **Part 3: Identification and quantification of PCDDs/PCDFs**

### **1 Scope**

No additional information

### **2 Normative references**

No additional requirements

### **3 Terms and definitions**

No additional information

### **4 Symbols and abbreviations**

No additional information

### **5 Principles of identification and quantification**

No additional information

### **6 Reagents, materials and equipment**

No additional requirements

### **7 Safety measures**

No additional requirements

### **8 Quality control requirements for identification and quantification**

#### **8.1 Minimum requirements for identification of PCDF/PCDD congeners**

No additional requirements

#### **8.2 Isomer sums of PCDD/PCDF congeners**

No additional requirements

#### **8.3 Minimum requirements for quantification**

- a) no additional requirements
- b) no additional requirements
- c) the quantification requirement in this section applies to an initial system suitability check only. It is not necessary to carry out this step on each sample containing 2,3,7,8-TCDD.

- d) no additional requirements
- e) no additional requirements
- f) this applies to native PCDDs/PCDFs only, since labelled compound are present at the same concentration in the standards.
- g) no additional requirements
- h) the equation shown in the standard relates the minimum sensitivity of the measurement to the LOQ. In the original version of the standard this same relationship was presented with the LOD defined in the same manner. Meeting the terms of the original expression is acceptable when stating conformity with the standard.
- i) the standard states one of the criteria for identifying a congener is if the isotope ratio between the ions matches the theoretical value. If interference results in only one ion being quantified the ion ratio limits would not have been met. However, it does allow quantification of a single ion if interference is unavoidable on the second trace. The use of one ion only must be reported.

## **9 Quality assurance criteria for extraction/clean-up/quantification procedure blanks**

No additional requirements

## **10 Calibration of the HRGC/HRMS**

If the calibration range is exceeded and the results are over range this should be stated in the report.

## **11 Quantification of HRGC/HRMS results**

### **11.1 Quantification of the sample**

No additional requirements

### **11.2 Calculation of the recovery rates of the extraction standards**

No additional requirements

### **11.3 Calculation of the recovery rates of the sampling standards**

No additional requirements

## **12 Calculation of the measurement results**

Results shall be reported to the reference conditions specified in the Permit.  
Results should be reported to the toxic equivalency system specified in the Permit. If the Permit does not specify the system used then the factors in Table A1 of the standard should be applied.

### **13 Analytical report**

No additional requirements

### **14 Performance characteristics**

#### **14.1 General aspects**

No additional requirements

#### **14.2 Intra-laboratory results**

No additional requirements

#### **14.3 Inter-laboratory results**

No additional requirements

#### **14.4 Quantification limits**

No additional requirements

### **15 Interferences**

No additional requirements

### **Annex A - Zz (informative) Examples of operation of gas chromatography / mass spectrometry analysis of PCDDs/PCDFs in emission samples**

No additional requirements

### **Annex B (informative) Estimation of the measuring uncertainty and precision of the determination of polychlorinated dibenzo-p-dioxins and dibenzofurans**

No additional requirements

### **Annex C (informative) Mass of ions monitored for PCDDs and PCDFs**

No additional requirements

**Annex D (informative) Relative abundance of chlorine isotope ions and their ratio for PCDD/PCDF congeners containing 4 to 8 chlorine substituents**

No additional requirements

**Annex E (informative) Further general recommendations for separation, detection and quantification of PCDDs/PCDFs**

No additional requirements

**Annex F (informative) Variability determination based on the results of the validation tests**

No additional requirements

**Annex Zz(informative) Clauses of this European Standard addressing essential requirements or other provisions of EU Directives.**

No additional requirements