SLOVENSKI STANDARD
SIST EN 1948-3:1999

01-september-1999

Emisije nepremiļnih virov ï Doloļevanje masne koncentracije PCDD/PCDF ï 3. del: Identifikacija in kvantifikacija

Stationary source emissions - Determination of the mass concentration of PCDDs/PCDFs - Part 3: Identification and quantification

Emissionen aus stationären Quellen - Bestimmung der Massenkonzentration von PCDDs/PCDFs - Teil 3: Identifizierung und Quantifizierung

Emissions de sources fixes - Détermination de la concentration massique en PCDDs/PCDFs - Partie 3: Identification et quantification

Ta slovenski standard je istoveten z: **EN 1948-3:1996**

ICS:

13.040.40	Emisije nepremiļnih virov	Stationary source emissions

SIST EN 1948-3:1999 **en**

EUROPEAN STANDARD

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Descriptors: Air, quality, air pollution, gaseous effluents, emission, determination, concentration, PCDD, PCDF, identification, quantity inspection, gas chromatography, mass spectrometry

English version

Stationary source emissions — Determination of the mass concentration of PCDDs/PCDFs — Part 3: Identification and quantification

Emissions de sources fixes — Détermination de la concentration massique en PCDDs/PCDFs — Partie 3: Identification et quantification

Emissionen aus stationären Quellen — Bestimmung der Massenkonzentration von PCDDs/PCDFs — Teil 3: Identifizierung und Quantifizierung

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.

This European Standard exists 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, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

CEN

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

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

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 G, 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 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.

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

NFX 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

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.

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 at very small concentrations. The 2,3,7,8-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 Part of the Standard.

Contents

1 Scope

The present Part of the Standard specifies the identification and quantification procedures of the sampled PCDDs/PCDFs. It is an integral part of the complete measurement procedure. The use of the other two Parts of this Standard, EN 1948-1 : 1996 and EN 1948-2 : 1996, describing sampling and extraction and clean-up, 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/m^3 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 identification and quantification 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 to complete the measurement procedure.

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

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**. 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 the EN 1948 : 1996 lays down requirements in order to measure every 2,3,7,8-chlorine substituted PCDD/PCDF congener 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 1948-2 : 1996 *Stationary source emissions — Determination of the mass concentration of PCDDs/PCDFs — Part 2: Extraction and clean-up*

ISO 5725-2 : 1994 *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

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 $^{13}C_{12}$-labelled PCDD/PCDF 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

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 [ISO 6879 : 1995].

3.1.4 statistical performance characteristics

Quantify, for measured values, the possible deviations resulting from the random part of the measuring process; these are, for example, repeatability or instability [ISO 6879 : 1995].

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

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

3.1.8 extraction standard

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

3.1.9 syringe standard

$^{13}C_{12}$-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 (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 HRGC

High resolution gas chromatography.

3.2.5 HRMS

High resolution mass spectrometry.

3.2.6 TCDD

Tetrachlorodibenzo-p-dioxin.

3.2.7 PeCDD

Pentachlorodibenzo-p-dioxin.

3.2.8 HxCDD

Hexachlorodibenzo-p-dioxin.

3.2.9 HpCDD

Heptachlorodibenzo-p-dioxin.

3.2.10 OCDD

Octachlorodibenzo-p-dioxin.

3.2.11 TCDF

Tetrachlorodibenzofuran.

3.2.12 PeCDF

Pentachlorodibenzofuran.

3.2.13 HxCDF

Hexachlorodibenzofuran.

3.2.14 HpCDF

Heptachlorodibenzofuran.

3.2.15 OCDF

Octachlorodibenzofuran.

3.2.16 PCDD/PCDF

Polychlorinated dibenzo-p-dioxin/dibenzofuran.

3.2.17 PTFE

Polytetrafluoroethylene.

4 Principles of identification and quantification

This Standard is based on the use of the gas chromatography/mass spectrometry combined with the isotope dilution technique to enable the separation, detection and quantification of PCDD/PCDF in the extracts of emission samples. These extracts are prepared in accordance with EN 1948-2 : 1996 and contain the two syringe standards. The gas chromatographic parameters offer information which enables the identification of isomers (position of Cl substituents) whereas the mass spectrometric parameters enable the differentiation between congeners with different numbers of chlorine substituents and between dibenzo-p-dioxins and furans.

5 Reagents, materials and equipment

See examples of operation in annex A.

6 Safety measures

All relevant national safety regulations should be observed. The 2,3,7,8-chlorine substituted PCDDs/PCDFs are among the most toxic of chemicals. All work with PCDDs/PCDFs requires therefore the utmost care; the national safety measures which correspond to those for toxic substances should be strictly adhered to.

7 Quality control requirements for identification and quantification

7.1 Minimum requirements for identification of PCDD/PCDF congeners

High resolution gas chromatography/high resolution mass spectrometry at a resolution of greater or equal to 10 000 is at present required to achieve adequate sensitivity, selectivity and to allow the use of all the $^{13}C_{12}$-labelled standards. Resolution in the range of 6 000 to 10 000 might be acceptable if the absence of interferences is documented. Other techniques which show that they meet the requirements described in this Standard may be used.

For each 2,3,7,8-chlorine substituted congener at least two ions of the molecular isotope cluster shall be recorded for both the native and the added $^{13}C_{12}$-labelled congeners (see annex C). A positive identification of a congener is made if all the following requirements are met.

a) The isotope ratio between the ions monitored shall match the theoretical value within ± 20 % (see annex D).

b) The retention time of a native 2,3,7,8-chlorine substituted isomer (Cl_4-Cl_6- congeners) shall be within a time window of $^{+3}_{0}$ s based on the retention time of the corresponding $^{13}C_{12}$-labelled isomer in the sample. For hepta- and octachloro congeners deviations of $^{+3}_{-2}$ s are acceptable. Alternatively, relative retention times based on 1,2,3,7,8-PeCDF can be calculated. The difference shall not be more than 0,25 % compared with the calibration standard.

c) The signal-to-noise ratio of the raw data as documented in figure 1 shall be at least 3 : 1 for the signal used for quantification.

The base line noise shall be measured in front of the signal within a signal-free window corresponding to 10 times the signal width at half height. Peak-to-peak values are taken.

7.2 Isomer sums of PCDD/PCDF congeners

If the sum of the concentrations of isomer groups are needed the following requirements shall be met.

a) The retention time window for all isomers of an isomer group shall be measured by a solution containing all native PCDDs/PCDFs. A fly ash extract can be used for this purpose. Each group of isomer (tetra to octa) should be defined via relative retention times (of the central isomer first). The pattern of overlapping windows (tetra/penta) should be 'constant' during one series of measurements; the changes in the relative retention times shall not be greater than ± 10 %.

b) The retention times of all congeners attributed to an isomer group shall be within the time window of the first and last eluting isomer.

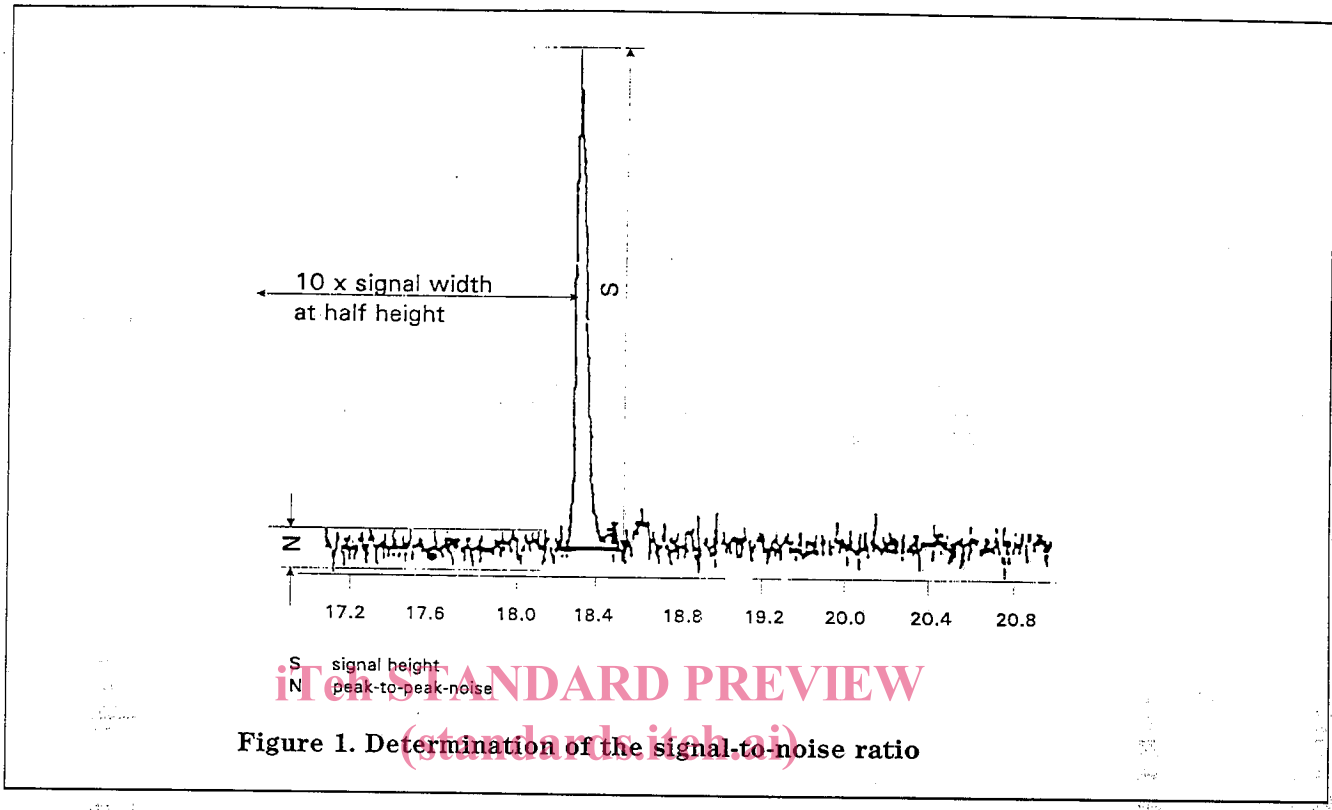

Figure 1. Determination of the signal-to-noise ratio

7.3 Minimum requirements for quantification

In addition to the requirements for identification, the following points shall be fulfilled as quantification requirements.

a) At present, there is no chromatographic column available that is able to separate all 2,3,7,8-chlorine substituted congeners from all other, non-2,3,7,8-chlorine substituted congeners. Complete separation can be achieved by multi-analysis of the sample on different columns of different nature (polarity). To avoid laborious and costly laboratory procedures, this Standard will accept a result as valid if it is shown that the contribution to the resulting I-TEQ from non-toxic congeners is less than 5 %.

b) The peak shape of the gas chromatographic signal of an congener shall contain ten or more sampling points (scanning units).

c) The gas chromatographic separation column shall separate the 2,3,7,8-chlorine substituted congeners from interfering congeners with a 90 % valley relative to the highest peak. 2,3,7,8-TCDF shall be separated from all other interfering isomers within a 25 % valley below the top of the minor peak with respect to the height of that peak.

d) The recovery rate of each individual 2,3,7,8-chlorine substituted PCDD/PCDF of the extraction standards in each sample shall be within:

1) 50 % to 130 % for the tetra- to hexa-chlorinated congeners;

2) 40 % to 130 % for the hepta- and octa-chlorinated congeners.

If the above ranges are exceeded, then provided the sum of the contributions to the total I-TEQ in the sample from all the congeners with recoveries not within these ranges does not exceed 10 %, the acceptable ranges shall be:

3) 30 % to 150 % for the tetra- to hexa-chlorinated congeners;

4) 20 % to 150 % for the hepta- and octa-chlorinated congeners.

e) The signal-to-noise ratio of the signal of the $^{13}C_{12}$-labelled congeners used for quantification shall be > 20 : 1.

f) The measuring range shall be linear (at least over a concentration range of a factor of 100). The standard deviation of the slope of the regression line shall not exceed ± 10 % and shall be based on a minimum of five measuring points over the whole range.

g) A control blank shall be taken. The blank of all 2,3,7,8-chlorine substituted congeners shall be equal or less than the detection limit of the method. Alternatively, the levels found shall be at least a factor of 10 below the lowest measured concentrations in the series of samples.

h) The permissible lower detection limits (LODs) with a signal-to-noise ratio, as defined in **7.1** for the individual congener (i) shall be as follows:

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

8 Quality assurance criteria for extraction/clean-up/quantification procedure blanks

The extraction blank value of all 2,3,7,8-chlorine substituted congeners shall be measured in a blank sample covering the complete analytical procedure including extraction, clean-up, and quantification when one of the following situations occurs.

a) After a series of no more than 10 samples.

b) After major changes in the extraction or clean-up procedure such as:

1) use of new or repaired equipment;

2) use of new batches of solvents or adsorbents.

c) After the analysis of a sample with unusually high levels exceeding average concentration levels by a factor of 10.

An extraction blank can be accepted when all requirements given in clause **7** are fulfilled.

Further recommendations which should be observed for separation, identification and quantification are given in annex E (informative).

9 Calibration of the GC/MS

The calibration is carried out with at least five calibration solutions. These solutions contain all native PCDDs/PCDFs in precisely defined amounts and all $^{13}C_{12}$-labelled standards (sampling, extraction and syringe standards). The calibration range should encompass the PCDD/PCDF concentrations of the sample. The calibration curve is used to calculate the analyte relative response factors (see also annex E).

The relative response factors are used together with the $^{13}C_{12}$-labelled congeners added to the sample to quantify the mass of the native PCDDs/PCDFs by the isotope dilution method.

Calibration frequency depends on the stability of the instrument. Daily calibration checks shall be run. In addition a full calibration shall be repeated after major changes such as:

a) use of new or repaired equipment;

b) replacement of GC columns;

c) after cleaning of the separation and detection systems;

d) if the deviation of an injected control calibration standard exceeds 20 %.

The relative response factor for congener i is defined and calculated as follows:

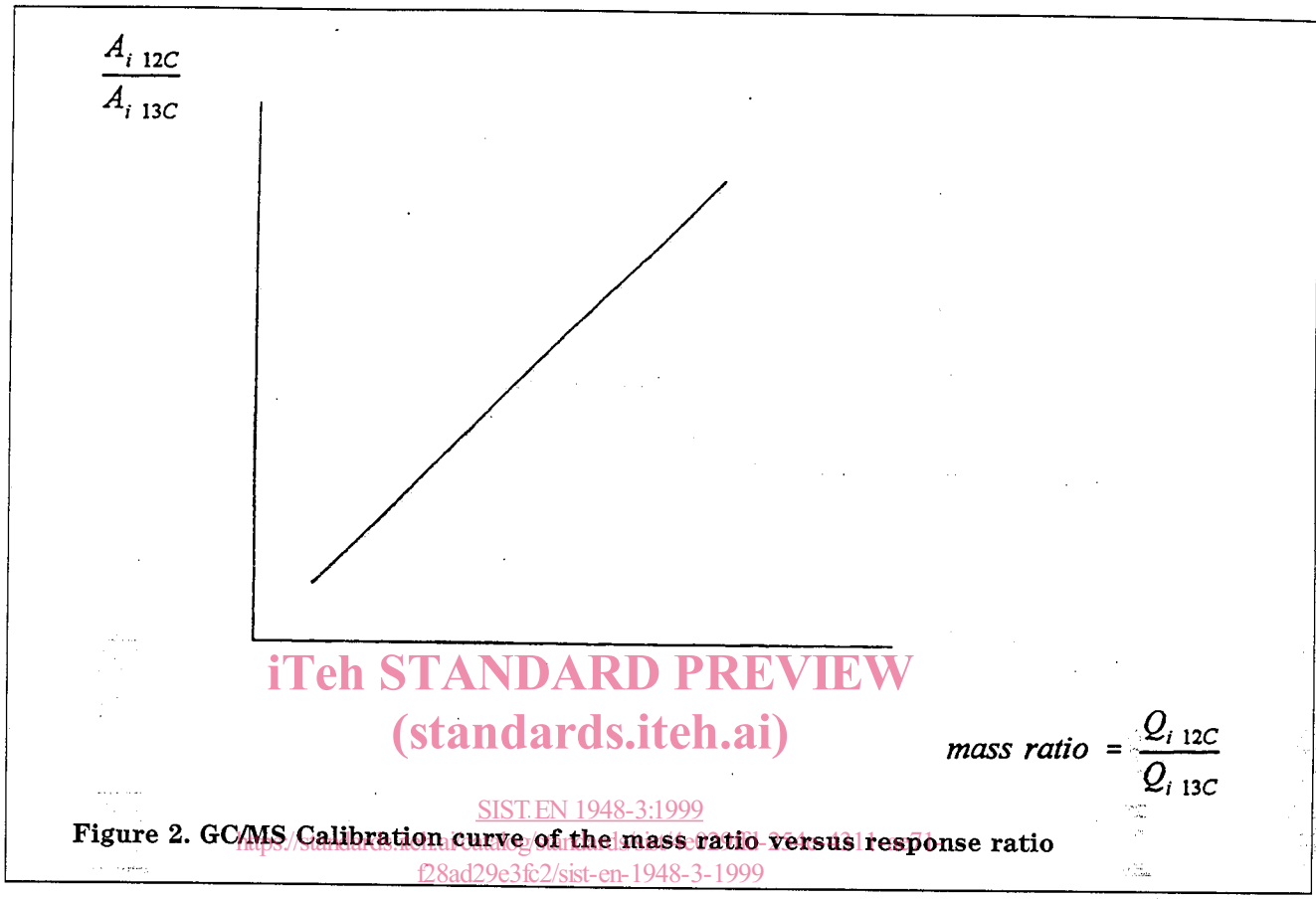

Figure 2. GC/MS Calibration curve of the mass ratio versus response ratio

$$rrf_i = \frac{A_{i\ 12C}}{A_{i\ 13C}} \cdot \frac{Q_{i\ 13C}}{Q_{i\ 12C}} \qquad (2)$$

Where:

rrf_i is the relative response factor of native congener i relative to $^{13}C_{12}$-labelled congener i

$\frac{A_{i\ 12C}}{A_{i\ 13C}}$ is the response ratio of native congener i and $^{13}C_{12}$-labelled congener i

$\frac{Q_{i\ 13C}}{Q_{i\ 12C}}$ is the mass ratio of $^{13}C_{12}$-labelled congener i and native congener i

The calibration curve is a plot of the mass ratio (x-axis) versus response ratio (y-axis). (See figure 2.)

10 Quantification of GC/MS results

10.1 Quantification of the sample

The mass of congener i in the sample is calculated as follows:

$$Q_{i\ 12C} = \frac{Q_{i\ 13C}}{rrf_i} \cdot \frac{A_{i\ 12C}}{A_{i\ 13C}} \qquad (3)$$

Where:

$Q_{i\ 13C}$ is the mass of the $^{13}C_{12}$-labelled congener i added to the sample

$\frac{A_{i\ 12C}}{A_{i\ 13C}}$ is the response ratio of native congener i and $^{13}C_{12}$-labelled congener in the sample

rrf_i is the relative response factor of congener i relative to $^{13}C_{12}$-labelled congener i

The responses of all detected masses of the PCDDs/PCDFs in the samples shall be within the linear range of the method (see clause **7**). Overlap in the mass window between high isotopic (i.e. M+12, M+14) of the native PCDD/PCDF with the lower isotopic ions (M, M+2) of the $^{13}C_{12}$-labelled standards will result in a significant deviation from linearity beyond a mass ratio of 10, especially for higher chlorinated congeners.

Annex D shows the theoretical isotope ratio for all PCDDs/PCDFs with 4 to 8 chlorine substituents.

For some native congeners the corresponding $^{13}C_{12}$-labelled congeners are used as sampling or syringe standards and so cannot be used for calculation of the relative response factors. In this case a congener with similar properties is used. The $^{13}C_{12}$-labelled congeners to be used are given in table 1.

Table 1. Quantification scheme for PCDDs/PCDFs in emission samples

Analyte	Extraction standard
2,3,7,8-TCDD	$^{13}C_{12}$-2,3,7,8-TCDD
1,2,3,7,8-PeCDD	$^{13}C_{12}$-1,2,3,7,8-PeCDD
1,2,3,4,7,8-HxCDD	$^{13}C_{12}$-1,2,3,4,7,8-HxCDD
1,2,3,6,7,8-HxCDD	$^{13}C_{12}$-1,2,3,6,7,8-HxCDD
1,2,3,7,8,9-HxCDD	$^{13}C_{12}$-1,2,3,6,7,8-HxCDD
1,2,3,4,6,7,8-HpCDD	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD
OCDD	$^{13}C_{12}$-OCDD
2,3,7,8-TCDF	$^{13}C_{12}$-2,3,7,8-TCDF
1,2,3,7,8-PeCDF	$^{13}C_{12}$-1,2,3,7,8-PeCDF
2,3,4,7,8-PeCDF	$^{13}C_{12}$-2,3,4,7,8-PeCDF
1,2,3,4,7,8-HxCDF	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF
1,2,3,6,7,8-HxCDF	$^{13}C_{12}$-1,2,3,6,7,8-HxCDF
1,2,3,7,8,9-HxCDF	$^{13}C_{12}$-2,3,4,6,7,8-HxCDF
2,3,4,6,7,8-HxCDF	$^{13}C_{12}$-2,3,4,6,7,8-HxCDF
1,2,3,4,6,7,8-HpCDF	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF
1,2,3,4,7,8,9-HpCDF	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF
OCDF	$^{13}C_{12}$-OCDF

10.2 Calculation of the recovery rates of the extraction standards

The extraction standards are quantified against the syringe standards as given in table 2 using equation 4.

$$R_{i\,e} = \frac{100}{Q_{i\,e}} \cdot \frac{Q_{i\,sy}}{rrf_i} \cdot \frac{A_{i\,e}}{A_{i\,sy}} \qquad (4)$$

Where:

$R_{i\,e}$ is the recovery rate of the extraction standard in percent

$Q_{i\,e}$ is the mass of the individual extraction standard added

$Q_{i\,sy}$ is the mass of the $^{13}C_{12}$-labelled syringe standard added to the sample

$\frac{A_{i\,e}}{A_{i\,sy}}$ is the response ratio of the extraction standard i and the relevant syringe standard in the sample

rrf_i is the relative response factor of extraction standard i relative to syringe standard i

Table 2. Calculation scheme for the recovery rates of the extraction standards

Extraction Standard	Syringe Standard
$^{13}C_{12}$-2,3,7,8-TCDD	$^{13}C_{12}$-1,2,3,4-TCDD
$^{13}C_{12}$-1,2,3,7,8-PeCDD	$^{13}C_{12}$-1,2,3,4-TCDD
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
$^{13}C_{12}$-OCDD	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
$^{13}C_{12}$-2,3,7,8-TCDF	$^{13}C_{12}$-1,2,3,4-TCDD
$^{13}C_{12}$-2,3,4,7,8-PeCDF	$^{13}C_{12}$-1,2,3,4-TCDD
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
$^{13}C_{12}$-OCDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD

10.3 Calculation of the recovery rates of the sampling standards

The sampling standards are quantified against the appropriate extraction standards as given in table 3 using equation 5.

$$R_{isa} = \frac{100}{Q_{i\,sa}} \cdot \frac{Q_{i\,e}}{rrf_i} \cdot \frac{A_{i\,sa}}{A_{i\,e}} \qquad (5)$$