SLOVENSKI STANDARD
SIST-TS CEN/TS 16190:2012

01-junij-2012

Blato, obdelani biološki odpadki in tla - Določevanje dioksinov in furanov in dioksinom podobnih polikloriranih bifenilov s plinsko kromatografijo z masno selektivnim detektorjem visoke ločljivosti (HR GC/MS)

Sludge, treated biowaste and soil - Determination of dioxins and furans and dioxin-like polychlorinated biphenyls by gas chromatography with high resolution mass selective detection (HR GC-MS)

Schlamm, behandelter Bioabfall und Boden - Bestimmung von Dioxinen und Furanen sowie Dioxin vergleichbaren polychlorierten Biphenylen mittels Gaschromatographie und hochauflösender massenspektrometrischer Detektion (HR GC-MS)

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Boue, biodéchet traité et sol - Détermination des dioxine et furane et biphényls polychlorés de type dioxine par chromatographie en phase gazeuse-spectrométrie de masse (HR GC-MS)

Ta slovenski standard je istoveten z: CEN/TS 16190:2012

ICS:

13.030.20	Tekoči odpadki. Blato	Liquid wastes. Sludge
13.080.10	Kemijske značilnosti tal	Chemical characteristics of soils

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2003-01.Slovenski inštitut za standardizacijo. Razmnoževanje celote ali delov tega standarda ni dovoljeno.

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TECHNICAL SPECIFICATION
SPÉCIFICATION TECHNIQUE
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CEN/TS 16190

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ICS 13.030.01

English Version

Sludge, treated biowaste and soil - Determination of dioxins and furans and dioxin-like polychlorinated biphenyls by gas chromatography with high resolution mass selective detection (HR GC-MS)

Boues, biodéchets traités et sols - Détermination des dioxines et furanes et polychlorobiphényles de type dioxine par chromatographie en phase gazeuse avec spectrométrie de masse à haute résolution (CG-SMHR)

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This Technical Specification (CEN/TS) was approved by CEN on 24 April 2011 for provisional application.

The period of validity of this CEN/TS is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the CEN/TS can be converted into a European Standard.

CEN members are required to announce the existence of this CEN/TS in the same way as for an EN and to make the CEN/TS available promptly at national level in an appropriate form. It is permissible to keep conflicting national standards in force (in parallel to the CEN/TS) until the final decision about the possible conversion of the CEN/TS into an EN is reached.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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Foreword

This document (CEN/TS 16190:2012) has been prepared by Technical Committee CEN/TC 400 “Project Committee - Horizontal standards in the fields of sludge, biowaste and soil”, the secretariat of which is held by DIN.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

The preparation of this document by CEN is based on a mandate by the European Commission (Mandate M/330), which assigned the development of standards on sampling and analytical methods for hygienic and biological parameters as well as inorganic and organic determinants, aiming to make these standards applicable to sludge, treated biowaste and soil as far as this is technically feasible.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to announce this Technical Specification: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

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Introduction

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

A group of chlorinated aromatic compounds similar to polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) is known as polychlorinated biphenyls (PCBs) which consist of 209 individual substances.

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 dibenzo-p-dioxins/dibenzofurans are the 74 monochlorinated to trichlorinated dibenzo-p-dioxins/dibenzofurans.

PCBs have been produced over a period of approximately 50 years until the end of the 1990s for the purpose of different use in open and closed systems, e. g. as electrical insulators or dielectric fluids in capacitors and transformers, as specialised hydraulic fluids, as a plasticizer in sealing material. Worldwide more than one million tons of PCBs were produced.

PCDD/F as well as PCBs are emitted during thermal processes as e. g. waste incineration. In 1997 a group of experts of the World Health Organisation (WHO) fixed toxicity equivalent factors (TEF) for PCDD and twelve PCBs, known as dioxin-like PCBs (see Annex A). These twelve dioxin-like PCBs consist of four non-ortho PCBs and eight mono-ortho PCBs (no or only one chlorine atoms in 2-, 2'-, 6- and 6'-position), having a planar or mostly planar structure. Dioxin-like PCB can contribute considerably to the total WHO-TEQ.

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Only skilled operators who are trained in handling highly toxic compounds should apply the method described in this Technical Specification.

This Technical Specification is applicable for several types of matrices and validated for municipal sludge (see also Annex B for the results of the validation).

WARNING — Persons using this Technical Specification should be familiar with usual laboratory practice. This Technical Specification does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

IMPORTANT — It is absolutely essential that tests conducted according to this Technical Specification be carried out by suitably trained staff.

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1 Scope

This Technical Specification specifies a method for quantitative determination of 17 2,3,7,8-chlorine substituted dibenzo-p-dioxins and dibenzofurans and dioxin-like polychlorinated biphenyls in sludge, treated biowaste and soil using liquid column chromatographic clean-up methods and GC/HRMS.

The analytes to be determined with this Technical Specification are listed in Table 1.

Table 1 — Analytes and their abbreviations

Substance	Abbreviation
Tetrachlorodibenzo-p-dioxin	TCDD
Pentachlorodibenzo-p-dioxin	PeCDD
Hexachlorodibenzo-p-dioxin	HxCDD
Heptachlorodibenzo-p-dioxin	HpCDD
Octachlorodibenzo-p-dioxin	OCDD
Tetrachlorodibenzofuran	TCDF
Pentachlorodibenzofuran	PeCDF
Hexachlorodibenzofuran	HxCDF
Heptachlorodibenzofuran	HpCDF
Octachlorodibenzofuran	OCDF
Polychlorinated biphenyl	PCB
Trichlorobiphenyl	TCB
Tetrachlorobiphenyl	TeCB
Pentachlorobiphenyl	PeCB
Hexachlorobiphenyl	HxCB
Heptachlorobiphenyl	HpCB
Decachlorobiphenyl	DecaCB

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The limit of detection depends on the kind of sample, the congener, the equipment used and the quality of chemicals used for extraction and clean-up. Under the conditions specified in this Technical Specification, limits of detection better than 1 ng/kg (expressed as dry matter) can be achieved.

This method is "performance based". It is permitted to modify the method if all performance criteria given in this method are met.

NOTE In principle this method can also be applied for sediments, mineral wastes and for vegetation. It is the responsibility of the user of this Technical Specification to validate the application for these matrices. For measurement in complex matrices like fly ashes adsorbed on vegetation it can be necessary to further improve the clean up. This can also apply to sediments and mineral wastes.

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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 16179, *Sludge, treated biowaste and soil — Guidance for sample pretreatment*

3 Abbreviations

PCB	Polychlorinated biphenyls
PCDD/PCDF or PCDD/F	Polychlorinated dibenzo-p-dioxins/dibenzofurans
I-TEF NATO/CCMS	International toxic equivalent factor proposed by NATO-CCMS in 1988 (for detailed description, see Annex A)
I-TEQ	International toxic equivalent obtained by multiplying the mass determined with the corresponding I-TEF including PCDDs and PCDFs (for detailed description, see Annex A). Should only be used for comparison with older data
WHO-TEF	Toxic equivalent factor proposed by WHO in 2005 (for detailed description, see Annex A)
WHO-TEQ	Toxic equivalent obtained by multiplying the mass determined with the corresponding WHO-TEF including PCDD, PCDF and PCB (for detailed description, see Annex A). WHO-TEQ_{PCB}, WHO-$TEQ_{PCDD/F}$ should be used to distinguish different compound classes

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4 Principle

This Technical Specification is based on the use of gas chromatography/mass spectrometry combined with the isotope dilution technique to enable the separation, detection and quantification of PCDD/PCDF and dioxin-like PCB in sludge, biowaste and soil. For the isotope dilution method 17 labelled PCDD/F and 12 labelled PCB internal standards are used. The extracts for the GC-MS measurements contain one or two recovery standards. The gas chromatographic parameters offer information which enables the identification of congeners (position of chlorine substitutes) whereas the mass spectrometric parameters enable the differentiation between isomers with different numbers of chlorine substitutes and between dibenzo-p-dioxins, furans and PCB.

$^{13}C_{12}$-labelled PCDD/F and PCB congeners are added to the sample prior to extraction and HRGC/HRMS measurement. Losses during extraction and clean-up are detected and compensated by using these added congeners as internal standards for quantification together with recovery standards which are added just before the HRGC/HRMS analysis. For the determination of these substances it is necessary to separate PCBs from PCDDs/PCDFs and vice versa.

The main purpose of the clean-up procedure of the raw sample extract is the removal of sample matrix components, which may overload the separation method, disturb the quantification or otherwise severely impact the performance of the identification and quantification method and the separation of PCDD/F from dioxin-like PCB. Furthermore, the enrichment of the analytes in the final sample extract is achieved. Extraction procedures are usually based on Soxhlet or equivalent extraction methods of dried, preferably freeze dried, samples. Sample clean-up is usually carried out by multi-column liquid chromatographic techniques using different adsorbents. The determination of PCDD/F and PCBs is based on quantification by the isotope-dilution technique using HRGC/HRMS.

5 Reagents

5.1 Chemicals

Solvents used for extraction and clean-up shall be of pesticide grade or equivalent quality and checked for blanks. Adsorbents like aluminium oxide, silica gel, diatomaceous earth and others used for clean-up shall be of analytical grade quality or better and pre-cleaned and activated if necessary.

NOTE See Annex C for a specific list of solvents and chemicals.

5.2 Standards

- ^{13}C-spiking solution for PCDD/F (internal Standard);
- ^{13}C-spiking solution for PCB (internal Standard);
- Calibration solutions PCDD/F;
- Calibration solutions PCB;
- Recovery standard PCDD/F;
- Recovery standard PCB.

NOTE See Annex C for examples of concentration of the standard solutions.

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6 Apparatus and materials

6.1 General

The apparatus and materials listed below are meant as minimum requirements for "conventional" sample treatment with Soxhlet extraction and column chromatographic clean-up. Additional apparatus and materials may be necessary due to different methods of sample extraction and clean-up methods.

6.2 Equipment for sample preparation

6.2.1 Laboratory fume hood, of sufficient size to contain the sample preparation equipment listed below.

6.2.2 Desiccator.

6.2.3 Balances, consisting of an analytical type capable of weighing 0,1 mg and a top-loading type capable of weighing 10 mg.

6.3 Soxhlet extractor

6.3.1 Soxhlet, 50 mm internal diameter, 150 ml or 250 ml capacity with 500 ml round bottom flask.

6.3.2 Thimble, 43 mm × 123 mm, to fit Soxhlet.

6.3.3 Hemispherical heating mantle, to fit 500 ml round-bottom flask.

6.4 Clean-up apparatus

6.4.1 Disposable pipettes, either disposable Pasteur pipettes, or disposable serological pipettes.

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6.4.2 Glass chromatographic columns, of the following sizes:

- 150 mm length × 8 mm internal diameter, with coarse-glass frit or glass-wool plug, 250 ml reservoir and glass or polytetrafluoroethylene (PTFE) stopcock;
- 200 mm length × 15 mm internal diameter, with coarse-glass frit or glass-wool plug, 250 ml reservoir and glass or PTFE stopcock;
- 300 mm length × 25 mm internal diameter, with coarse-glass frit or glass-wool plug, 300 ml reservoir and glass or PTFE stopcock.

6.4.3 Oven, capable of maintaining a constant temperature (± 5 °C) in the range of 105 °C to 450 °C for baking and storage of adsorbents.

6.5 Concentration apparatus

6.5.1 Rotary evaporator, equipped with a variable temperature water bath and:

- vacuum source for rotary evaporator equipped with shutoff valve at the evaporator and vacuum gauge;
- recirculating water pump and chiller, providing cooling water of (9 ± 4) °C (use of tap water for cooling the evaporator wastes large volumes of water and can lead to inconsistent performance as water temperatures and pressures vary);
- round-bottom flask, 100 ml and 500 ml or larger, with ground-glass fitting compatible with the rotary evaporator.

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6.5.2 Nitrogen blowdown apparatus, equipped with either a water bath controlled in the range of 30 °C to 60 °C or a heated stream of nitrogen, installed in a fume hood.

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6.5.3 Kuderna-Danish[1] **concentrator.**

6.5.4 Sample vials, of the following types:

- amber glass, nominated volume 2 ml to 5 ml, with PTFE-lined screw cap;
- glass, 0,3 ml, conical, with PTFE-lined screw or crimp cap.

6.6 Other equipment

6.6.1 Gas chromatograph, equipped with a splitless or on-column or temperature programmed injection port for the use with capillary columns, and an oven temperature programme which enables isothermal hold.

6.6.2 GC column for PCDDs/PCDFs and for isomer specificity for 2,3,7,8-TCDD (e. g. 60 m length × 0,32 mm internal diameter; 0,25 µm; 5 % phenyl, 94 % methyl, 1 % vinyl silicone bonded-phase fused-silica capillary column).

6.6.3 Mass spectrometer, 28 eV to 80 eV electron impact ionization, capable of repetitively selectively monitoring of twelve exact masses minimum at high resolution (> 10 000) during a period of approximately 1 s.

6.6.4 Data system, capable of collecting, recording, and storing mass spectrometric data.

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1) Kuderna Danish is an example of a suitable product available commercially. This information is given for the convenience of users of this Technical Specification and does not constitute an endorsement by CEN of this product.

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7 Sample storage and sample pretreatment

7.1 Sample storage

Samples should be stored in suitable containers with an appropriate closure material such as polytetrafluoroethylene (PTFE). Samples to be frozen may be stored in aluminium containers pre-cleaned by heating to 450 °C for minimum 4 h or by rinsing with a non-chlorinated solvent.

Samples should be kept cold (< 8 °C) and in the dark. The sample pretreatment should take place within three days of sampling. Alternatively, samples may be frozen (–18 °C) directly after sampling and kept frozen before sample pretreatment.

7.2 Sample pretreatment

Drying and homogenization should be carried out according to EN 16179, if not otherwise specified. Store the ground material in a desiccator or a tightly closed glass container.

8 Extraction and clean-up

8.1 General

In this Technical Specification the minimum requirements for extraction and clean-up to be met are described as well as examples of operation. The analyst may use any of the procedures given below and in Annex C or any suitable alternative procedures.

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The determination of PCDDs/PCDFs is based on quantification by the isotope-dilution technique using HRGC/HRMS. $^{13}C_{12}$-labelled 2,3,7,8-chlorine substituted PCDD/PCDF congeners are added at different stages of the whole method. Losses during extraction and clean-up can be detected and compensated by using these added congeners as internal standards for quantification together with recovery standards which are added just before the HRGC/HRMS analysis. However, due to possible differences in the binding and adsorption characteristics between the native PCDDs/PCDFs and the $^{13}C_{12}$-labelled congeners, which are added during analysis, complete substantiation of the extraction efficiency and compensation of losses during clean-up is not ensured. Therefore, in addition the applied methods shall be validated thoroughly. Examples of well-proven extraction and clean-up methods are given in Annex C.

The main purpose of the clean-up procedure of the raw sample extract is the removal of sample matrix components, which may overload the separation method, disturb the quantification or otherwise severely impact the performance of the identification and quantification method and to separate dioxin-like PCB from PCDD/F. Furthermore, an enrichment of the analytes in the final sample extract is achieved. Extraction procedures are usually based on soxhlet extraction of the < 2 mm fraction of the dry and ground or sieved solid sample. Sample clean-up is usually carried out by multi-column liquid chromatographic techniques using different adsorbents.

In principle any clean-up method can be used which recovers the analytes in sufficient quantities. Furthermore, the final sample extract shall not affect adversely the performance of the analytical system or the quantification step. However, all applied methods shall be tested thoroughly and shall pass a set of method validation requirements before they can be employed. In addition, the verification of the method performance for each single sample shall be part of the applied quality assurance protocol.

8.2 Extraction

The sample amount used for extraction may vary from 5 g to 50 g depending on the expected level of contamination.

The internal standard consisting of $^{13}C_{12}$-labelled congeners listed in Table 2 shall be added directly onto the

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sample before extraction.

The extraction procedure is carried out using soxhlet extraction with toluene. Duration of extraction should be adjusted according to kind and amount of sample used. The minimum requirement is 50 extraction cycles or approximately 12 h.

Other solvents or other methods like pressurized liquid extraction can also be used but shall be of proven equal performance.

Table 2 — ^{13}C labelled congeners included in the internal standard

^{13}C-spiking solution – Internal standard	
PCDD/F congeners	**PCB congeners**
2,3,7,8-$^{13}C_{12}$-TCDD	$^{13}C_{12}$-PCB 77
1,2,3,7,8-$^{13}C_{12}$-PeCDD	$^{13}C_{12}$-PCB 81
1,2,3,4,7,8-$^{13}C_{12}$-HxCDD	$^{13}C_{12}$-PCB 126
1,2,3,6,7,8-$^{13}C_{12}$-HxCDD	$^{13}C_{12}$-PCB 169
1,2,3,7,8,9-$^{13}C_{12}$-HxCDD	
1,2,3,4,6,7,8-$^{13}C_{12}$-HpCDD	$^{13}C_{12}$-PCB 105
$^{13}C_{12}$-OCDD	$^{13}C_{12}$-PCB 114
	$^{13}C_{12}$-PCB 118
2,3,7,8-$^{13}C_{12}$-TCDF	$^{13}C_{12}$-PCB 123
1,2,3,7,8-$^{13}C_{12}$-PeCDF	$^{13}C_{12}$-PCB 156
2,3,4,7,8-$^{13}C_{12}$-PeCDF	$^{13}C_{12}$-PCB 157
1,2,3,4,7,8-$^{13}C_{12}$-HxCDF	$^{13}C_{12}$-PCB 167
1,2,3,6,7,8-$^{13}C_{12}$-HxCDF	$^{13}C_{12}$-PCB 189
2,3,4,6,7,8-$^{13}C_{12}$-HxCDF	
1,2,3,7,8,9-$^{13}C_{12}$-HxCDF	
1,2,3,4,6,7,8-$^{13}C_{12}$-HpCDF	
1,2,3,4,7,8,9-$^{13}C_{12}$-HpCDF	
$^{13}C_{12}$-OCDF	

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8.3 Clean-up

8.3.1 General

Clean-up methods shall prepare the sample extract in an appropriate manner for the subsequent quantitative determination. Clean-up procedures shall concentrate PCDDs/Fs and dioxin-like PCBs in the extracts and to remove interfering matrix components present in the raw extract.

Proven clean-up procedures shall be used including usually two or more of the following techniques which can be combined in different orders. A detailed description of some of the procedures is given in Annex C.

Other methods can also be used but shall be of proven equal performance as the techniques described below.