Characterization of waste - Determination of selected polychlorinated biphenyls (PCB) in solid waste by gas chromatography with electron capture or mass spectrometric detection



EESTI STANDARDI EESSÕNA

NATIONAL FOREWORD

		eelset	This Estonian standard EVS-EN 15308:2016 consists of the English text of the European standard EN 15308:2016.
Standard on jõustur avaldamisega EVS Teata			This standard has been endorsed with a notification published in the official bulletin of the Estonian Centre for Standardisation.
Euroopa standardimisor Euroopa standardi kättesaadavaks 12.10.20	rahvuslikele liikr		Date of Availability of the European standard is 12.10.2016.
Standard on Standardikeskusest.	kättesaadav		The standard is available from the Estonian Centre for Standardisation.

Tagasisidet standardi sisu kohta on võimalik edastada, kasutades EVS-i veebilehel asuvat tagasiside vormi või saates e-kirja meiliaadressile <u>standardiosakond@evs.ee</u>.

ICS 13.030.10, 13.030.20, 13.080.10

Standardite reprodutseerimise ja levitamise õigus kuulub Eesti Standardikeskusele

Andmete paljundamine, taastekitamine, kopeerimine, salvestamine elektroonsesse süsteemi või edastamine ükskõik millises vormis või millisel teel ilma Eesti Standardikeskuse kirjaliku loata on keelatud.

Kui Teil on küsimusi standardite autorikaitse kohta, võtke palun ühendust Eesti Standardikeskusega: Aru 10, 10317 Tallinn, Eesti; koduleht <u>www.evs.ee</u>; telefon 605 5050; e-post <u>info@evs.ee</u>

The right to reproduce and distribute standards belongs to the Estonian Centre for Standardisation

No part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying, without a written permission from the Estonian Centre for Standardisation.

If you have any questions about copyright, please contact Estonian Centre for Standardisation:

Aru 10, 10317 Tallinn, Estonia; homepage www.evs.ee; phone +372 605 5050; e-mail info@evs.ee

EUROPEAN STANDARD

NORME EUROPÉENNE

EN 15308

EUROPÄISCHE NORM

October 2016

ICS 13.030.10; 13.030.20; 13.080.10

Supersedes EN 15308:2008

English Version

Characterization of waste - Determination of selected polychlorinated biphenyls (PCB) in solid waste by gas chromatography with electron capture or mass spectrometric detection

Caractérisation des déchets - Détermination de polychlorobiphényles (PCB) sélectionnés dans les déchets solides par chromatographie en phase gazeuse avec détection par capture d'électrons ou spectrométrie de masse Charakterisierung von Abfällen - Bestimmung ausgewählter polychlorierter Biphenyle (PCB) in festem Abfall unter Anwendung der Kapillar-Gaschromatographie mit Elektroneneinfang-Detektion oder massenspektrometrischer Detektion

This European Standard was approved by CEN on 21 September 2016.

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 CEN-CENELEC Management Centre 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 CEN-CENELEC Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and United Kingdom.



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

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

Contents Page European foreword......4 Introduction _____5 1 3 4 Reagents8 5 5.1 Reagents for extraction and drying......8 5.2 5.3 Reagents for gas chromatographic analysis......10 5.4 5.5 5.5.1 Internal and injection standards....... 10 5.5.2 Apparatus.......11 6 6.1 6.1.1 6.1.2 6.1.3 6.2 Gas chromatograph 12 6.3 Safety precautions 12 7 8 9 **10** 10.1 10.2 Drving ______ 13 10.3 11 11.1 11.2

	AgNO ₃ /silica clean-up	
	DMSO/n-hexane partitioning clean-up	
11.3.1		
	Gas chromatographic analysisGeneral	
	Optimizing the gas chromatograph	
	Detectors	
	Check on method performance	
	GC-MS determination	
11.4.6	GC-ECD determination	21
12	Calculation	22
13	Test report	22
Annex	A (informative) Performance characteristics	23
A.1	General	
A.2	Type of samples and sample preparation	
A.3	Homogeneity and stability	
A.4	ExtractionClean-up	
A.5 A.6	Detection	
A.7	PCB standard solution	
Annex	B (informative) Examples of GC-MS chromatograms of a calibration standard solution and a contaminated soil sample	29
Annex	C (informative) Examples of GC-ECD chromatograms of a calibration standard solution and a cable shredder sample	36
Annex	D (normative) Calculation method for the estimation of total PCB content	39
	E (informative) Summary of general requirements and recommendations	
Bibliog	graphy	49
		2

European foreword

This document (EN 15308:2016) has been prepared by Technical Committee CEN/TC 444 "Test methods for the characterization of solid matrices", the secretariat of which is held by NEN.

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 April 2017, and conflicting national standards shall be withdrawn at the latest by April 2017.

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

This document supersedes EN 15308:2008.

According to the CEN-CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, 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.

Introduction

Polychlorinated biphenyls (PCB) have been widely used as additives in industrial applications where chemical stability has been required. This stability on the other hand creates environmental problems when PCBs are eventually released into the environment. Since some of these PCB compounds are highly toxic, their presence in the environment (air, water, soil, sediment and waste) is regularly monitored and controlled. At present determination of PCB is carried out in these matrices in most of the routine laboratories following the preceding steps for sampling, pretreatment, extraction, clean-up by measurement of specific PCB by means of gas chromatography in combination with mass spectrometric detection (GC-MS) or gas chromatography with electron capture detector (GC-ECD).

Taking into account the different matrices and possible interfering compounds, this European Standard does not contain one single possible way of working. Several choices are possible, in particular relating to clean-up. Detection with both Mass Spectrometry and Electron Capture is possible. Two different extraction procedures and nine clean-up procedures are described. The use of internal and injection standards is described in order to have an internal check on choice of the extraction and clean-up procedure. This European Standard has been validated on seven solid waste which are typically bris, aste w. contaminated with PCB (building debris, cable shredder, contaminated soil, electronic waste, sealant waste, shredder light fraction and waste wood). Validation data are given in Annex A (informative).

1 Scope

This European Standard specifies a method for quantitative determination of seven polychlorinated biphenyl congeners (PCB-28, PCB-52, PCB-101, PCB-118, PCB-138, PCB-153 and PCB-180) in solid waste using high-resolution gas chromatography with electron capture or mass spectrometric detection. The basic content of this standard is identical to that of the Horizontal PCB-standard and is therefore also applicable to soil, sludge and treated bio-waste. The detection and the quantification limits in this method are dependent on sample intake, the level of interferences as well as instrumental limitations. Under the conditions specified in this standard, minimum amounts of individual PCB congeners equal or above 0,01 mg/kg dry matter can typically be determined with no interferences present.

NOTE For the analysis of PCB in insulating liquids, petroleum products, used oils and aqueous samples is referred to EN 61619, EN 12766-1 and EN ISO 6468 respectively.

The method may be applied to the analysis of other PCB congeners not specified in the scope, but its suitability should be proven by proper in-house validation experiments.

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 12766-1, Petroleum products and used oils - Determination of PCBs and related products - Part 1: Separation and determination of selected PCB congeners by gas chromatography (GC) using an electron capture detector (ECD)

EN 12766-2, Petroleum products and used oils - Determination of PCBs and related products - Part 2: Calculation of polychlorinated biphenyl (PCB) content

EN 14346, Characterization of waste - Calculation of dry matter by determination of dry residue or water content

3 Terms and definitions

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

3.1

polychlorinated biphenyl

PCB

biphenyl substituted by one to ten chlorine atoms

3.2

congener

member of the same kind, class or group of chemicals

Note 1 to entry: Any one of the two hundred and nine individual PCB.

Note 2 to entry: The IUPAC congener numbers are for easy identification; they do not represent the order of chromatographic elution.