

VEE KVALITEET. MITMETE POLÜBROOMITUD
DIFENÜÜLEETRITE (PBDE) MÄÄRAMINE VEE
KOGUPROOVIDES, KASUTADES TAHKE FAASI
EKSTRAKTSIOONI (SPE) JA GAASIKROMATOGRAAFIAT
MASSISPEKTROMEETRILISE DETEKTEERIMISEGA
(GC-MS)

Water quality - Determination of selected
polybrominated diphenyl ether (PBDE) in whole water
samples - Method using solid phase extraction (SPE)
with SPE-disks combined with gas chromatography -
mass spectrometry (GC-MS)

EESTI STANDARDI EESSÕNA

NATIONAL FOREWORD

See Eesti standard EVS-EN 16694:2015 sisaldab Euroopa standardi EN 16694:2015 ingliskeelset teksti.	This Estonian standard EVS-EN 16694:2015 consists of the English text of the European standard EN 16694:2015.
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English Version

Water quality - Determination of selected polybrominated diphenyl ether (PBDE) in whole water samples - Method using solid phase extraction (SPE) with SPE-disks combined with gas chromatography - mass spectrometry (GC-MS)

Qualité de l'eau - Dosage du pentabromodiphényléther (PBDE) dans des échantillons d'eau totale - Méthode par extraction en phase solide (SPE) avec disques SPE, avec couplage chromatographie en phase gazeuse - spectrométrie de masse (CG-SM)

Wasserbeschaffenheit - Bestimmung von ausgewählten polybromierten Diphenylethern (PBDE) in Gesamtwasserproben - Verfahren mittels Festphasenextraktion (SPE) mit SPE-Disks in Verbindung mit Gaschromatographie - Massenspektrometrie (GC-MS)

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European foreword

This document (EN 16694:2015) has been prepared by Technical Committee CEN/TC 230 “Water analysis”, 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of an identical text or by endorsement, at the latest by March 2016, and conflicting national standards shall be withdrawn at the latest by March 2016.

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Introduction

WARNING — Persons using this European Standard should be familiar with usual laboratory practice. This European Standard 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 European Standard be carried out by suitably trained staff.

Polybrominated diphenyl ethers (PBDE) are priority substances listed in Annex X of the EU Water Framework Directive (WFD, Directive 2000/60/EC) for which environmental quality standards (EQS) have been set at EU level for inland waters as well as other surface waters to protect the aquatic environment against chemical pollution (Directive 2008/105/EC). With the exception of metals, the EQS are expressed as total concentrations in the whole water sample. Furthermore, analytical methods used in WFD monitoring need to meet certain requirements as regards the minimum limit of quantification and the maximum tolerable measurement uncertainty (Directive 2009/90/EC). So far, there is no standardized method available for the determination of PBDE in whole water samples fulfilling those requirements. Hence, the European Commission mandated CEN to develop or improve standards in support of the implementation of the monitoring requirements of WFD.

Directive 2008/105/EC has been amended by Directive 2013/39/EU, however this standard has been developed for the analysis of PBDE as listed in Annex A of Directive 2008/105/EC.

The priority substances list in Annex X of the WFD includes technical pentabromodiphenyl ether, which is regarded as a mixture of the congeners BDE-28, BDE-47, BDE-99, BDE-100, BDE-153 and BDE-154. The annual average environmental quality standard (AA-EQS) for pentabromodiphenyl ether in inland surface waters is 0,5 ng/l and is defined for the whole water sample including suspended particulate matter (SPM) present in the sample. Compounds such as PBDE strongly adsorb to environmental solids resulting in a fraction bound to particles, which may be substantial. The aim of the method is to extract whole water samples in one single step to determine the dissolved as well as the particle bound PBDE fraction. Identification and quantification of BDE congeners at trace level concentrations require both highly sensitive chromatographic equipment and effective enrichment steps and awareness of the potential impact of blanks.

1 Scope

This European Standard specifies a method for the determination of six selected polybrominated diphenyl ethers (PBDE) listed in Table 1, representative for technical brominated diphenyl ethers (BDE) in water samples in mass concentrations $\geq 0,025$ ng/l for each individual congener. The method uses solid-phase extraction with SPE-disks in combination with gas chromatography-mass spectrometry (GC-MS). It is applicable to the analysis of PBDE in surface water containing suspended particulate matter (SPM) up to 500 mg/l (whole water samples), drinking water and groundwater. The limit of quantification (LOQ) was determined according to ISO/TS 13530, on the basis of replicate determinations of the procedural blank, carried out under reproducibility conditions.

This method may be used for the analysis of other BDE congeners not listed in Table 1 or other types of water. However, it is important to verify its applicability before use.

Table 1 — Polybrominated diphenyl ethers (PBDE) determined by this method

Congener	Abbreviation ^a	Formula	Molar mass g/mol	CAS RN ^b
2,4,4'-Tribromodiphenyl ether	BDE-28	C ₁₂ H ₇ Br ₃ O	406,8954	41318-75-6
2,2',4,4'-Tetrabromodiphenyl ether	BDE-47	C ₁₂ H ₆ Br ₄ O	485,7950	5436-43-1
2,2',4,4',5-Pentabromodiphenyl ether	BDE-99	C ₁₂ H ₅ Br ₅ O	564,6911	60348-60-9
2,2',4,4',6-Pentabromodiphenyl ether	BDE-100	C ₁₂ H ₅ Br ₅ O	564,6911	189084-64-8
2,2',4,4',5,6'-Hexabromodiphenyl ether	BDE-154	C ₁₂ H ₄ Br ₆ O	643,5872	68631-49-2
2,2',4,4',5,5'-Hexabromodiphenyl ether	BDE-153	C ₁₂ H ₄ Br ₆ O	643,5872	207122-15-4

NOTE EC numbers are not applicable for PBDE congeners.

^a Numbering analog to IUPAC nomenclature for PCB

^b CAS RN: Chemical Abstracts Service Registry Number

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 ISO 3696, *Water for analytical laboratory use - Specification and test methods (ISO 3696)*

EN ISO 5667-1, *Water quality - Sampling - Part 1: Guidance on the design of sampling programmes and sampling techniques (ISO 5667-1)*

EN ISO 5667-3, *Water quality - Sampling - Part 3: Preservation and handling of water samples (ISO 5667-3)*

ISO 8466-1, *Water quality — Calibration and evaluation of analytical methods and estimation of performance characteristics — Part 1: Statistical evaluation of the linear calibration function*

3 Principle

A mixture of suitable internal standards (¹³C₁₂-labelled BDE or fluorinated BDE) is added to a 1 l water sample, which is then extracted using a solid-phase extraction disk (SPE-disk). The disk is eluted with a