
**Water quality — Determination of
selected polybrominated diphenyl ethers
in sediment and sewage sludge —
Method using extraction and gas
chromatography/mass spectrometry**

*Qualité de l'eau — Dosage d'une sélection d'éthers diphenyliques
polybromés dans des sédiments et des boues d'épuration — Méthode
par extraction et chromatographie en phase gazeuse/spectrométrie de
masse*



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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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

ISO 22032 was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*.

Introduction

The user should be aware that particular problems could require the specification of additional marginal conditions.

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

1 Scope

This International Standard specifies a method for the determination of selected polybrominated diphenyl ethers (PBDE) (see Figure 1 and Table 1) in sediment and sludge using gas chromatography/mass spectrometry (GC-MS) in the electron impact (EI) or negative ion chemical ionization (NCI) mode.

When using GC-EI-MS, the method is applicable to samples containing 0,05 µg/kg to 25 µg/kg of tetra- to octabromo congeners and 0,3 µg/kg to 100 µg/kg of decabromo diphenyl ether (BDE-209), respectively. Approximately ten times lower concentrations can be quantified when using GC-NCI-MS. The risk of misinterpretation of interfering substances is smaller with EI due to its higher specificity. It is also possible to analyse other brominated diphenyl ethers according to this International Standard, after verifying its applicability in each case.

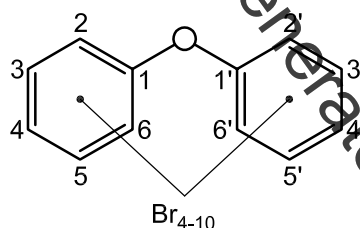


Figure 1 — Chemical formula of polybrominated diphenyl ethers

Table 1 — PBDE congeners determined by this method

No.	Congener	Formula	Abbreviation ^a	Molar mass g/mol
1	2,2',4,4'-Tetrabromodiphenyl ether	C ₁₂ H ₆ Br ₄ O	BDE-47	485,795 0
2	2,2',4,4',5-Pentabromodiphenyl ether	C ₁₂ H ₅ Br ₅ O	BDE-99	564,691 1
3	2,2',4,4',6-Pentabromodiphenyl ether	C ₁₂ H ₅ Br ₅ O	BDE-100	564,691 1
4	2,2',4,4',5,6'-Hexabromodiphenyl ether	C ₁₂ H ₄ Br ₆ O	BDE-154	643,587 2
5	2,2',4,4',5,5'-Hexabromodiphenyl ether	C ₁₂ H ₄ Br ₆ O	BDE-153	643,587 2
6	2,2',3,4,4',5'-Heptabromodiphenyl ether	C ₁₂ H ₃ Br ₇ O	BDE-183	722,483 2
7	Decabromodiphenyl ether	C ₁₂ Br ₁₀ O	BDE-209	959,171 4

^a Numbering analogous to IUPAC nomenclature for PCB.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 5667-13, *Water quality — Sampling — Part 13: Guidance on sampling of sludges from sewage and water-treatment works*

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

Extraction of brominated diphenyl ethers from the dried sample by an organic solvent. Clean-up of the extract by, e.g. preparative multi-layer silica gel column chromatography. After concentration, separation of the brominated diphenyl ethers by capillary gas chromatography and detection by either mass spectrometry in the selected ion monitoring mode using electron impact (EI), or negative ion chemical ionization (NCI). For determination of the concentration in the sample, an internal standard calibration over the total procedure is used.

4 Interferences

When applying GC-NCI-MS, 2,2',4,4',5,5'-hexabromobiphenyl (BB-153) and tetrabromobisphenol A can co-elute with BDE-154 and BDE-153, respectively, when using non-polar capillary columns and hence, interfere with the determination of the corresponding BDE congeners when monitoring the bromide ions $m/z = 79$ and $m/z = 81$. Moreover, naturally produced brominated compounds, such as halogenated bipyrrols and brominated phenoxyanisols, can be considered as potential interferences.

Sources of contamination are the following: brominated diphenyl ethers used as flame-retardants in organic polymers. Therefore, contact of the sample or the reagents with these organic polymers shall be avoided. Transportation paths include airborne dust, vial covers, pasteur pipette fillers and recycled paper.