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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 diphényliques polybromés dans des sédiments et des boues d'épuration — Méthode par extraction et chromatographie en phase gazeuse/spectrométrie de masse

Reference number
ISO 22032:2006(E)

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Foreword

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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.

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

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Introduction

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INTERNATIONAL STANDARD **ISO 22032:2006(E)**

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

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.

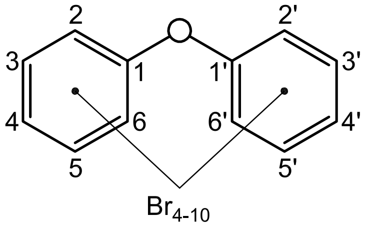

Figure 1 — Chemical formula of polybrominated diphenyl ethers

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Table 1 — PBDE congeners determined by this method

No.	Congener	Formula	Abbreviation [a]	Molar mass g/mol
1	2,2',4,4'-Tetrabromodiphenyl ether	$C_{12}H_6Br_4O$	BDE-47	485,795 0
2	2,2',4,4',5-Pentabromodiphenyl ether	$C_{12}H_5Br_5O$	BDE-99	564,691 1
3	2,2',4,4',6-Pentabromodiphenyl ether	$C_{12}H_5Br_5O$	BDE-100	564,691 1
4	2,2',4,4',5,6'-Hexabromodiphenyl ether	$C_{12}H_4Br_6O$	BDE-154	643,587 2
5	2,2',4,4',5,5'-Hexabromodiphenyl ether	$C_{12}H_4Br_6O$	BDE-153	643,587 2
6	2,2',3,4,4',5',6-Heptabromodiphenyl ether	$C_{12}H_3Br_7O$	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.

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5 Reagents and standards

Only use reagents with negligibly low concentrations of brominated diphenyl ethers compared with the concentration to be determined and verify by blank determinations. To prevent degradation, store standards in the dark at temperatures recommended by the manufacturer (calibration solutions should preferably be stored at approximately −18 °C).

5.1 Solvents for extraction, clean-up and preparation of stock solutions.

A variety of solvents may be used depending on the particular sample matrix to be analysed and the availability of commercial standard solution, e.g. toluene (C_7H_8), or acetone (propanone, C_3H_6O), or a mixture of acetone (propanone, C_3H_6O) and hexane (C_6H_{14}), or heptane (C_7H_{16}), or iso-octane (2,2,4-trimethylpentane, C_8H_{18}), or nonane (C_9H_{20}), or dichloromethane (CH_2Cl_2) for residual analysis.

5.2 Reference substances.

See Table 1. Solutions of reference substances are commercially available.

5.3 Internal standard substances.

Solutions of reference substances for use as internal standards for electron impact ionization (Table 2, substances 1 to 5) and for negative ion chemical ionization (Table 2, substances 6, 7 and 8) are commercially available.

Table 2 — Examples of internal standards

No	Name	Formula	Abbreviation	Molar mass g/mol
Internal standards for GCMS with electron impact ionization				
1	2,2’,4,4’-Tetrabromo[$^{13}C_{12}$]diphenyl ether	$^{13}C_{12}H_6Br_4O$	^{13}C-BDE-47	497,703 5
2	2,2’,4,4’,5-Pentabromo[$^{13}C_{12}$]diphenyl ether	$^{13}C_{12}H_5Br_5O$	^{13}C-BDE-99	576,599 5
3	2,2’,4,4’,5,5’-Hexabromo[$^{13}C_{12}$]diphenyl ether	$^{13}C_{12}H_4Br_6O$	^{13}C-BDE-153	655,495 5
4	2,2’,3,4,4’,5’,6-Heptabromo[$^{13}C_{12}$]diphenyl ether	$^{13}C_{12}H_3Br_7O$	^{13}C-BDE-183	734,391 6
5	Decabromo[$^{13}C_{12}$]diphenyl ether	$^{13}C_{12}Br_{10}O$	^{13}C-BDE-209	971,079 7
Internal standards for GCMS with negative ion chemical ionization [a]				
6	3,3’,4,4’-Tetrabromodiphenyl ether	$C_{12}H_6Br_4O$	BDE-77	485,795 0
7	2,2’,3,4,4’,5,6-Heptabromodiphenyl ether	$C_{12}H_3Br_7O$	BDE-181	722,483 2
8	Decabromo[$^{13}C_{12}$]diphenyl ether	$^{13}C_{12}Br_{10}O$	^{13}C-BDE-209	971,079 7
[a] Check for interferences when non-labelled PBDE is used as an internal standard. Other BDE congeners are suitable as internal standards, e.g. BDE-140.				

5.4 Sodium sulfate, anhydrous, Na_2SO_4, powdered.

5.5 Operating gases, for gas chromatography/mass spectrometry, of high purity and in accordance with manufacturer’s specifications.

5.6 Nitrogen, of high purity, at least 99,999 % by volume, for drying and for concentration by evaporation.

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5.7 Solutions of the single reference substances / internal standards.

Use commercially available solutions (may be in nonane, toluene or iso-octane) or prepare stock solutions, e.g. by dissolving 10 mg of each of the reference substances (5.2, 5.3) in toluene (5.1) in an amber, 10-ml volumetric flask and bring to volume (concentration: 1 mg/ml). Store at approximately –18 °C in the dark.

5.8 Multicomponent stock solution of reference substances.

Accurately transfer between 100 µl to 500 µl of each single standard solution (5.7) into an amber, 10-ml volumetric flask and bring to volume with the appropriate solvent, e.g. toluene, or nonane, or iso-octane (5.1). (Concentrations are between 10 µg/ml and 50 µg/ml per substance.)

5.9 Calibration solutions for multicomponent-multilevel calibration.

Prepare, e.g. seven calibration solutions with concentrations according to the detection capacity of the mass spectrometer. Combine the multicomponent stock solutions of reference substances (5.8), internal standards (5.10) and, if necessary, injection standard (5.12) to produce the solutions (e.g. shown in Table 5) by appropriate dilution with the appropriate solvent, e.g. toluene, or nonane, or iso-octane (5.1).

In order to avoid potential photodegradation, store the solutions in the dark. Check the concentrations of calibration solutions before use.

Use one of the calibration solutions to optimize the GC-MS system and to determine the retention times. As an alternative, determine and use relative retention times.

5.10 Stock solution of the internal standards.

Prepare a stock solution of the internal standards at an appropriate concentration in, e.g. toluene or iso-octane (2,2,4-trimethylpentane). Dilute this stock solution. See Table 5 for suggested concentrations of calibration solutions and sample extracts.

5.11 Clean-up material.

See Annex A.

5.12 Injection standard.

Use an injection standard, e.g. dibromooctafluorobiphenyl ($C_{12}Br_2F_8$), to determine recovery rates for the internal standard in each sample.

5.13 Baked sand.

Bake sand for at least 8 h at 400 °C.

6 Apparatus

Clean all glassware by rinsing with acetone (propanone) (5.1). Heating the glassware to 400 °C will reduce blanks. Recalibrate volumetric apparatus prior to use if heated.

6.1 Wide-necked bottle, 1 000 ml up to 5 000 ml capacity, for wet sediment or sludge.

6.2 Freeze drying apparatus.

6.3 Deep freezer.

6.4 Mortar and pestle, or **a grinding mill**.

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6.5 Drying ovens, capable of maintaining temperatures in the ranges of 100 °C to 400 °C for baking and storage of clean-up materials, for baking of glassware and for dry residue determination of samples.

6.6 Sieve shaker with appropriate sieve meshes (aperture size), e.g. 2 mm.

6.7 Desiccator.

6.8 Soxhlet extraction apparatus, consisting of round bottom flasks (e.g. 250 ml), Soxhlet extractors and Soxhlet thimbles (e.g. 27 mm × 100 mm), vertical condensers (e.g. 300 mm) and heating apparatus.

6.9 Evaporation device, e.g. rotary evaporator, turbo evaporator or vacuum concentration device.

6.10 Glass columns for chromatographic clean-up.

6.11 Volumetric cylinders, 250 ml and 500 ml.

6.12 Volumetric flasks, 1 ml, 2 ml, 10 ml, and 25 ml.

6.13 Pasteur pipettes, e.g. 2 ml.

6.14 Syringes, 2 µl, 5 µl, 10 µl and 50 µl, volume precision ± 2 %.

6.15 Sample vials.

Amber glass with fluoropolymer-lined screw-cap is most suitable.

6.16 Gas chromatograph, with either a splitless injection port or an on-column injection port coupled to a mass spectrometer (GC-MS) with electron impact or chemical ionization and appropriate reactant gas (e.g. CH_4).

6.17 Analytical column.

Fused silica column with non-polar low bleed separating phase (see Annex B for examples), e.g. inner diameter < 0,25 mm, length 15 m to at maximum 30 m (shorter columns for higher brominated congeners). A film thickness of 0,1 µm is recommended.

7 Sampling and sample pre-treatment

Take samples as specified in ISO 5667-13 in a bottle (6.1). Store and transport in the dark at approximately 4 °C. Pre-treat the samples immediately in the laboratory by homogenizing and freeze-drying. Grind the samples using apparatus (6.4) and sieve them using a sieve shaker (6.6) according to the analytical task.

8 Procedure

8.1 Extraction

Transfer a suitable mass, e.g. 5 g to 10 g, of the pre-treated, dry sample into a Soxhlet thimble. Depending on the expected concentration in the sample, add 100 µl to 1 000 µl of the internal standard solution (5.10), to the Soxhlet thimble. Place the thimble in the Soxhlet extractor. The various solvents given in 5.1 produce similar extraction efficiencies after a 16 h Soxhlet extraction. Certain sample matrices may require a more polar solvent to efficiently extract the PDBE congeners, e.g. a mixture of acetone (propanone) and hexane, or dichloromethane.

Other extraction techniques, e.g. accelerated solvent extraction, and shorter extraction times may be used after performing comparability exercise with a 16 h Soxhlet extraction.

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Fill the round-bottomed flask with, e.g. 100 ml of the solvent (5.1) and connect it to the Soxhlet extractor. The addition of boiling chips is useful. Lower the flask into the heating apparatus. Adjust the temperature of the heating device until the refluxing solvent reaches the lower part of the vertical condenser. Extract the sample for 16 h.

After the extraction is complete, concentrate the extract nearly to dryness using a suitable evaporation device (6.9).

Protect samples and extracts carefully from sunlight to avoid photodegradation of the decabromo diphenyl ether ($C_{12}Br_{10}O$).

Extraction of BDE-209 requires specific attention and, sometimes, longer extraction times than other PBDE congeners. Use toluene as an extraction solvent for extraction of BDE-209. During concentration, use toluene as a keeper.

8.2 Clean-up

Depending on the different sample matrices encountered, a variety of sample extract clean-up procedures may be suitable. Examples of clean-up procedures are given in Annex A. The recovery efficiency of all internal standards (see also 9.3 and 9.5) shall be 60 % or better. In most cases, use of a clean-up column given in A.1 followed by the procedure in A.2 is successful.

8.3 Measurement

Optimize the operating conditions of the GC-MS system, e.g. according to the manufacturer’s instructions. Examples of the gas chromatographic conditions are given in Annex B.

Prior to analysis, establish the operating conditions and verify the GC-MS system performance and the calibration for all analytes and their internal standards by analysis of a calibration standard.

Add the injection standard (5.12), if necessary, and analyse the sample with GC-MS.

Especially for the analysis of BDE-209, minimise the exposure of the samples to high temperatures for long periods of times during the injection and separation stages, because of the thermal degradation of BDE-209 at temperatures higher than 300 °C. Optimize the injection step, paying special attention to the peak height of BDE-209.

8.4 Blank determination

Analyse as a blank, a clean Soxhlet thimble in exactly the same way as the sample, but replacing the sample by the appropriate amount of baked sand (5.13). The concentration of PBDE in the blank should be negligible, compared with the concentrations of PBDEs to be determined.

8.5 Identification

Consider an analyte to be identified,

- if the retention time of the analyte in the mass chromatogram of the sample is the same as the retention time of the reference substance in the mass chromatogram of the calibration standard solution measured under identical experimental conditions (the deviation shall be below 1 %, and not exceeding 12 s), and
- if the ratio of the quantification and qualifier mass is within ± 10 % of the theoretical bromine isotope ratio.

See Table 3 for typical ions when negative ion chemical ionization detection used.

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Table 3 — Ions for negative ion chemical ionization detection

Compound	Ions for quantification	Ions for qualification
BDE-47	79	81
BDE-99	79	81
BDE-100	79	81
BDE-154	79	81
BDE-153	79	81
BDE-183	79	81
BDE-209	486,7 [a]	484,7
Internal standards		
BDE-77	79	81
BDE-181	79	81
^{13}C-BDE-209	494,7	496,7

[a] Do not use the ion 488,7 because of overlapping with a fragment ion from ^{13}C-BDE-209.

The following ions (Table 4) are proposed for electron impact ionization detection. The values in brackets are the percent ratio between the most intensive qualifier ion and the second qualifier ion. Use two representative masses with a recognizable bromine-pattern for identification of each compound. It is also possible to use more than one ion for quantification.

Table 4 — Ions for electron impact ionization detection

Compound	Ions for quantification	Ions for qualification
BDE-47	325,9	483,7 (69,5), 485,7 (100)
BDE-99	405,7	563,6 (100), 565,6 (98)
BDE-100	405,7	563,6 (100), 565,6 (98)
BDE-153	485,7	641,5 (100), 643,5 (73,4)
BDE-154	485,7	641,5 (100), 643,5 (73,4)
BDE-183	563,6	721,4 (100), 723,4 (93,6)
BDE-209	799,3 (100)	959,1 (25), 797,3 (81)
^{13}C-BDE-47	339,9	495,7 (69.5), 497,7 (100)
^{13}C-BDE-99	417,7	575,6 (100), 577,6 (98)
^{13}C-BDE-153	497,7	653,5 (100), 655,5 (73,4)
^{13}C-BDE-209	811,3 (100)	971,1 (25), 809,3 (81)

Check that the ions used are free from interferences caused by matrix components.

See Annex C for other ions that may be used and suggested time windows.

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9 Calibration

9.1 General

Modern mass spectrometric detection provides linear correlations between the concentrations of single substances and the corresponding responses over several decades of concentration. This facilitates an effective means of calibration. It is necessary to carry out the following steps.

9.2 Procedure steps

9.2.1 Evaluation of the range of the linear relationship

See Table 5 as an example of concentrations (5.9) for evaluating a linear range over several decades of concentration. The linear relationship should be assured with the concentration and response relationships as used in internal standardization (see 9.3). Plot, for example, the ratio values $y_i/y_{\text{is},i}$ (peak areas, peaks heights or integration units) for each substance i on the ordinate and the associated ratio of mass concentrations $\rho_i/\rho_{\text{is},i}$ on the abscissa. Check by the graphical representation of the calibration data as described in, e.g. ISO 8466-1.

Table 5 — Example concentrations in solutions for evaluating the linear range

Compound	Solutions						
	1	**2**	**3**	**4**	**5**	**6**	**7**
	ng/ml						
BDE-47	5	12,5	25	50	100	150	250
BDE-99	5	12,5	25	50	100	150	250
BDE-100	5	12,5	25	50	100	150	250
BDE-153	5	12,5	25	50	100	150	250
BDE-154	5	12,5	25	50	100	150	250
BDE-183	5	12,5	25	50	100	150	250
BDE-209	25	50	100	200	500	700	1 000
Internal standards for NCI							
BDE-77 – internal standard for BDE-47, BDE-99, BDE-100	100	100	100	100	100	100	100
BDE-181 – internal standard for BDE-153, BDE-154, BDE-183	100	100	100	100	100	100	100
^{13}C-BDE-209 ($^{13}C_{12}Br_{10}O$) – internal standard for BDE-209	500	500	500	500	500	500	500
Internal standards for EI							
^{13}C-BDE-47	200	200	200	200	200	200	200
^{13}C-BDE-99	200	200	200	200	200	200	200
^{13}C-BDE-153	200	200	200	200	200	200	200
^{13}C-BDE-183	400	400	400	400	400	400	400
^{13}C-BDE-209	500	500	500	500	500	500	500

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9.2.2 Two-point calibration over the total linear range

Use the mean values of obtained from multi-injections of two standards at different concentrations. Carry out a two-point calibration as described in 9.3. Check the validity of this calibration function which is dependent on the stability of the GC-MS system at least with each batch of samples. Use an independent standard or a reference material to check this.

9.2.3 Estimation of the accuracy of the calibration for the concentration of interest

For example, analyse, as a minimum in triplicate, an independent standard or a certified reference material as a sample, and calculate the results in accordance with the above calibration method. The standard deviation between the single results ρ_i and the known value ρ of the concentration in the standard is then calculated as follows:

$$s_\rho = \sqrt{\frac{\sum_{i=1}^{N}\left(\rho_i - \rho\right)^2}{N-1}} \tag{1}$$

where

s_ρ is a measure of the accuracy of the calibration;

N is the number of measurements.

9.2.4 Estimation of the limits of detection

Estimate the limits of detection according to documented procedures (for example, Eurachem Guide 1998[3], ISO/TR 13530[4], DIN 32645[2]). The limit of quantitation, also known as the limit of reporting corresponds, e.g. to the concentration for which $2s_\rho$ is less than, e.g. 1/3 of this concentration ρ.

9.2.5 Accuracy improvement

Where it is deemed necessary to improve the accuracy of the calibration for certain concentration ranges, carry out a multilevel calibration as specified in ISO 8466-1 over a range of no more than one decade.

9.3 Calibration with internal standards

The use of an internal standard for the determination of the concentration minimizes possible errors made during injection and from sample losses during sample pre-treatment steps, and differences in the final sample extract volumes and changes in recoveries caused by matrix effects. This calculation is usually available as an option in the quantitation programs of most manufacturers' data analysis software.

Additionally, it is possible to measure the recovery for the total procedure for each sample, if the values from the internal standard in the calibration solutions are compared to the values obtained from the extract. To achieve this, it is essential that the final volumes are identical. Alternatively, an injection standard can be used for the calculation of recoveries of internal standards (see 9.5).

See Table 5 for typical concentrations of reference compounds and internal standards in solutions for evaluating the linear range and for a listing of which internal standard to use for each PBDE compound. Adjust the concentrations according to the sensitivity of the equipment used and the range of determinations required.

Evaluate the linear range and, subsequently, set up a two-point calibration. Use the mean values of multi-injections, e.g. $\frac{y_i(1)}{y_{\text{is},i}(1)}$; $\frac{y_i(2)}{y_{\text{is},i}(2)}$; $\frac{y_i(3)}{y_{\text{is},i}(3)}$ at two concentration levels.

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Establish the linear function of the two pairs of values $\frac{y_i}{y_{\text{is},i}}$ and $\frac{\rho_i}{\rho_{\text{is},i}}$ of the measured series using the following Equation (2):

$$\frac{y_i}{y_{\text{is},i}} = a_i \frac{\rho_i}{\rho_{\text{is},i}} + b_i \tag{2}$$

where

y_i is the measured response of substance i; the unit depends on the evaluation, e.g. area value;

ρ_i is the mass concentration of substance i (external standard), in the working standard solution, in nanograms per millilitre, ng/ml;

a_i is the slope of the calibration function of substance i, the unit depends on the evaluation, e.g. area value × millilitres per nanogram, ml/ng;

b_i is the ordinate intercept of the calibration curve. The unit depends on the evaluation, e.g. area value;

$y_{\text{is},i}$ is the measured response of the internal standard for the substance i, the unit depends on the evaluation, e.g. area value;

$\rho_{\text{is},i}$ is the mass concentration of the internal standard, for the substance i, in nanograms per millilitre, ng/ml.

Estimate the accuracy of the calibration and limits of detection and quantification as described in 9.2.

9.4 Quantitation with the internal standard

Add a known amount of the internal standard (see Table 5) to the sample in the Soxhlet thimble prior to extraction. The mass concentration $\rho_{\text{is},i}$ in the final volume of extract shall be the same for calibration and sample measurement. Use the same solvent composition for the working standard solutions and the extracts.

Pre-treat and analyse the samples as described in 8.1 to 8.4. Inject identical volumes of the sample extracts as injected as calibration solutions.

Calculate the mass concentration $\rho_{i,\text{sample}}$ of the substance using Equation (3).

$$\rho_{i,\text{sample}} = \frac{\frac{y_{i,\text{sample}}}{y_{\text{is},i,\text{sample}}} - b_i}{a_i} \cdot \frac{m_{\text{is},i}}{m_{\text{sample}}} = \frac{\rho_{i,\text{sample extract}}}{\rho_{\text{is},i,\text{sample extract}}} \cdot \frac{m_{\text{is},i}}{m_{\text{sample}}} \tag{3}$$

where

$y_{i,\text{sample}}$ is the measured response, e.g. peak area, of the substance i in the sample extract;

$y_{\text{is},i,\text{sample}}$ is the measured response, e.g. peak area, of the internal standard, for substance i, of the sample;

$\rho_{i,\text{sample extract}}$ is the mass concentration of the substance i in the sample extract, in nanograms per millilitre, ng/ml; usually calculated by the software;

$\rho_{\text{is},i,\text{sample extract}}$ is the mass concentration of the internal standard in the sample extract, for substance i, in nanograms per millilitre, ng/ml; usually reported by the software;

$\rho_{i,\text{sample}}$ is the mass concentration of the substance i in the solid sample in micrograms per kilogram, µg/kg;

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$m_{\text{is},i}$	is the mass of the added internal standard substance, in micrograms, µg;
m_{sample}	the sample mass in kilograms, kg;
a_i	see Equation (2);
b_i	see Equation (2).

9.5 Determination of recoveries of the internal standards

It is possible to measure the recovery for single samples by comparing the values of the internal and the injection standards obtained in the calibration with that obtained from sample extracts. A control chart plot of the recoveries of the internal standards should indicate a 60 % recovery rate or better. Provided the relationship between the concentration and the response of the internal standard is linear and the intercept of this curve is negligible, the single recovery $A_{\text{is},i,\text{sample}}$ may be calculated according to Equation (4).

$$A_{\text{is},i,\text{sample}}(\%) = \frac{y_{\text{is},i,\text{sample}} \cdot y_{\text{is,inj,calibration}}}{y_{\text{is},i,\text{calibration}} \cdot y_{\text{is,inj,sample}}} \cdot 100 \qquad (4)$$

where

$A_{\text{is},i,\text{sample}}$	is the recovery of the internal standard, for the substance i; in percent, %;
$y_{\text{is},i,\text{sample}}$	is the measured response, e.g. peak area, of the internal standard, for substance i, in the sample;
$y_{\text{is},i,\text{calilbration}}$	is the measured response, e.g. peak area, of the internal standard, for substance i, in the calibration solution;
$y_{\text{is,inj,sample}}$	is the measured response, e.g. peak area, of the injection internal standard, in the sample;
$y_{\text{is,inj,calibration}}$	is the measured response, e.g. peak area, of the injection internal standard, in the calibration solution.

9.6 Results and reporting

Report results to two significant figures for the PBDE.

10 Test report

The test report shall specify the following:

a) a reference to this International Standard (ISO 22032:2006);

b) the identity of the sample;

c) the sample storage and pre-treatment;

d) the mesh size of the sieve applied

e) a complete description of the procedure;

f) the identification and quantification of single components;

g) an expression of results according to 9.6.

h) any deviation from this procedure and all circumstances that may have influenced the result.

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Annex A
(informative)

Clean-up procedures

A.1 Multi-layer column chromatography I (column size: 22 mm × 190 mm)

A.1.1 Reagents

A.1.1.1 **Silica 60**, 63 µm to 200 µm; bake at 250 °C for 12 h.

A.1.1.2 **Silica treated with silver nitrate**.

45 g of silica (A.1.1.1), 5 g of silver nitrate ($AgNO_3$) in 20 ml of water. Add the silver nitrate solution drop by drop and shake the mixture for 8 h. Bake it for 8 h at 120 °C. Store in amber glass bottles. The mixture is stable for approximately 1 month.

A.1.1.3 **Silica treated with sulfuric acid**.

56 g of silica (A.1.1.1), 44 g of sulfuric acid (H_2SO_4) (95 % to 97 %). Add the sulfuric acid drop by drop and shake the mixture for 8 h. Store in amber glass bottles. The mixture is stable for approximately 1 month.

A.1.1.4 **Silica treated with sodium hydroxide**.

33 g of silica (A.1.1.1), 17 g of sodium hydroxide (NaOH) (1 mol/l). Add the sodium hydroxide solution drop by drop and shake the mixture for 8 h. Store in amber glass bottles. The mixture is stable for approximately 1 month.

A.1.1.5 **Sodium sulfate** (Na_2SO_4), anhydrous; bake at 550 °C for 12 h.

A.1.1.6 **Ultrapure water**, water complying at least with Grade 1 of ISO 3696.

A.1.1.7 **Glass wool**.

A.1.2 Preparation of clean-up multi-layer column

Pack a glass chromatography column (ID × L, 22 mm × 190 mm) in the following sequence:

a) glass wool plug;

b) 2 g of silica (A.1.1.1);

c) 5 g of silica/sodium hydroxide (A.1.1.4); (this layer is focused on the removal of acid compounds);

d) 2 g of silica (A.1.1.1);

e) 10 g of silica/sulfuric acid (A.1.1.3); (this layer is focused on the removal of basic and aromatic compounds);

f) 2 g of silica (A.1.1.1);

g) 5 g of silica/silver nitrate (A.1.1.2); (this layer is focused on the removal of sulfur and sulfur-containing molecules);

h) 10 g of sodium sulfate (A.1.1.5); (this layer is focused on the removal of small amounts of water).

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A.1.3 Procedure

Condition the column with 50 ml of dichloromethane (CH_2Cl_2) and 50 ml of cyclohexane (C_6H_{12}).

Reduce the extract (8.1) to 2 ml and transfer to the column. Rinse the flask twice with 2 ml × 2 ml hexane (5.1).

For elution, use 50 ml of cyclohexane (C_6H_{12}) followed by 50 ml of cyclohexane:dichloromethane (volume fraction 80:20).

Add 100 µl injection standard (e.g. dibromooctafluorobiphenyl – $C_{12}Br_2F_8$-DBOFB) in toluene (5.1).

Combine the eluates and evaporate nearly to dryness using the keeper toluene.

A.2 Multi-layer column chromatography II (column size: 22 mm × 190 mm)

A.2.1 Reagents

A.2.1.1 Basic aluminium oxide (Al_2O_3) 90, activity Basic Super I, particle size 0,063 mm to 0,2 mm.

The aluminium oxide should be of maximum activity; avoid long storage or contact with humid air.

A.2.1.2 Sodium sulfate (Na_2SO_4), anhydrous, baked at 550 °C.

A.2.2 Procedure

Pack a glass chromatography column (ID × L, 22 mm × 190 mm) in the following sequence:

a) glass wool plug;

b) 1 cm to 2 cm of sodium sulfate (A.2.1.2);

c) 25 g of aluminium oxide (A.2.1.1); (this layer is focused on the removal of polar and acid compounds);

d) 1 cm to 2 cm of sodium sulfate (A.2.1.2).

Dilute the extract (8.1) or (A.1.3) to 10 ml with hexane (5.1) and pour it on the freshly prepared column.

Elute at first with a 150 ml mixture of hexane:dichloromethane 98:2, reject this eluate, it contains, e.g. hydrocarbons.

Secondly, elute the PBDE using 200 ml of a mixture of -hexane:dichloromethane 1:1.

Concentrate this eluate to a suitable final volume, e.g. approximately 100 µl, use toluene as a keeper.

Optimize the elution volumes when first applying the procedure and each time the properties of the aluminium oxide have changed.

A.3 Gel permeation chromatography (GPC)

A.3.1 General

This subclause gives an example of experimental conditions. This protocol is suitable for removing interferent compounds with a molecular size significantly different from PBDE, e.g. trigycerids and similar fatty compounds.

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A.3.2 Reagents

A.3.2.1 **Toluene** (C_7H_8), for residue analysis.

A.3.2.2 **Cyclohexane** (C_6H_{12}), for residue analysis.

A.3.2.3 **Ethyl acetate** ($C_4H_8O_2$), for residue analysis.

A.3.2.4 **Eluent:** cyclohexane-ethyl acetate (volume fraction 1:1).

A.3.2.5 **Bio-Beads S-X3** 1), 38 µm to 75 µm (200 mesh to 400 mesh).

A.3.2.6 **Syringe standard**, e.g. dibromooctafluorobiphenyl in toluene, 200 pg/µl.

A.3.3 Apparatus

A.3.3.1 **GPC apparatus**.

GPC clean-up system (with modular design):

- pump, sampling injector, sample rack;
- dilutor syringe for sample injection (10 ml), dilutor syringe for sample collection (5 ml), sample loop (5 ml);
- column: glass column (ID 24,4 mm, L 580 mm), variable length, filled with, e.g. 50 g Bio-Beads S-X3 (column bed approximately 350 mm).

A.3.3.2 **Evaporation apparatus**.

A.3.3.3 **Glassware**.

A.3.3.3.1 **Glass vial**, e.g. graduated with cap and PTFE septum (PTFE = polytetrafluoroethene);

A.3.3.3.2 **Volumetric flask** (amber), of capacity 5 ml;

A.3.3.3.3 **Concentration sample tube**, solvent capacity 500 ml,) with 0,5 ml stem;

A.3.3.3.4 **Microlitre syringe**, of capacity 100 µl;

A.3.3.3.5 **Pasteur pipettes**, 250 mm.

A.3.4 GPC clean-up procedure

Concentrate the extract obtained after column chromatography clean-up to a final volume of 0,5 ml using the concentration device. Evaporate the remaining solvent nearly to dryness under a gentle stream of nitrogen and dissolve immediately in a mixture of ethyl acetate:cyclohexane (volume fraction 1:1). Transfer into a 5 ml volumetric flask, bring to volume and then transfer completely into the glass vial. Inject 4 ml of this solution onto a column packed with the material (A.3.2.5) to clean up the extract.

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1) Bio-Beads S-X3 is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

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A.3.5 GPC clean-up conditions

Injection volume: 4 ml

Flow: 5 ml/min

Dump: 100 ml (10 min)

Collect: 125 ml (25 min)

Wash: 75 ml (15 min)

After preparing a new column for the GPC clean-up, verify the eluent volume for complete elution of the analytes of interest by analysing an appropriate standard solution and/or spiked sample extract. Recoveries of PBDEs should be $>$ 85 % and no interfering peak should appear in the gas chromatogram. If necessary, GPC conditions need to be modified to meet these requirements.

Collect the eluate in a concentration sample tube, add 0,5 ml of toluene and concentrate the extract to an approximate final volume of 0,5 ml.

Transfer the extract to a graduated vial and evaporate to dryness under a gentle stream of nitrogen. Dissolve the residue in 100 µl of syringe standard solution, transfer into an amber sample vial with insert and analyse using GCMS.

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Annex B
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Examples for separation conditions in gas chromatography and mass spectrometry

B.1 Example 1: GC-NCI-MS conditions

Injection:	Pulse splitless	50 psi (345 kPa) 1 min
Injector temperature:	300 °C	
Injection volume:	1 µl	
Transfer line temperature:	280 °C	
Ion source (NCI):	230 °C	CH_4
Quadrupole:	106 °C	
Resolution:	Low resolution, 1 mass unit	
Volume flow rate:	1,3 ml/min constant	
Carrier gas:	Helium	
Capillary column:	Length:	30 m
	Film thickness:	0,1 µm
	Inner diameter:	0,25 mm
Column material:	Medium polar, e.g. methylphenyl silicone phase DB5 MS 2)	
Temperature programme:	60 °C for 2 min, then 50 °C/min to 300 °C, then 30 °C/min to 330 °C for 10 min	

B.2 Example 2: GC-EI-MS conditions

Injection:	On column	35 kPa
Injector temperature:	110 °C	
Injection volume:	1 µl	
Transfer line temperature:	280 °C	
Ion source:	250 °C	
EI energy:	70 eV	

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2) DB5M5 is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

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Resolution:	Low resolution, 1 mass unit	
Carrier gas:	Helium	
Guard column:	Length:	2 m
	Inner diameter:	0,32 mm
Column material:	Uncoated, deactivated	
Capillary column:	Length:	15 m
	Film thickness:	0,1 µm
	Inner diameter:	0,25 mm
Column material:	Medium polar, e.g. methylphenyl silicone phase DB5 MS 2)	
Temperature programme:	110 °C for 0,2 min, then 30 °C/min to 200 °C, then 20 °C/min to 300 °C for 12,8 min	

B.3 Example 3: GC-NCI-MS conditions

Injection:	Pulse splitless	50 psi (354 kPa) 0,3 min
Injector temperature:	280 °C	
Injection volume:	1 µl	
Transfer line temperature:	300 °C	
Ion source:	230 °C	CH_4
Quadrupol:	106 °C	
Resolution:	Low resolution, 1 mass unit	
Volume flow rate:	0,9 ml/min constant	
Carrier gas:	Helium	
Capillary column:	Length:	15 m
	Film thickness:	0,1 µm
	Inner diameter:	0,25 mm
Column material:	DB1	
Temperature programme:	100 °C for 2 min, then 150 °C/min to 250 °C for 5 min, then 80 °C/min to 300 °C 2 min	

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B.4 Example 4: GC-EI-MS conditions

Injection:	PTV 3), splitless, pressure-pulse injection	
Injector temperature:	130 °C for 0,1 min, then 12 °C/s to 320 °C, then 320 °C for 5 min	
Injection volume:	2 µl	
Transfer line temperature:	320 °C	
Ion source:	210 °C	
Quadrupol:	106 °C	
Volume flow rate:	1,3 ml/min constant	
Carrier gas:	Helium	
Capillary column:	Length:	30 m
	Film thickness:	0,25 µm
	Inner diameter:	0,25 mm
Column material:	Restek-Rtx-CLPesticide 3)	
Temperature programme:	80 °C for 2 min, then 25 °C/min to 200 °C, then 5 °C/min to 320 °C for 13 min	

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3) Restek-Rtx-CLPesticide and PTV are examples of suitable products available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products.

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Annex C
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Typical ions and time windows for electron impact ionization detection

(GC conditions according to Example 2 in Annex B.)

Table C.1

Compound	Ion	Formula	Time window min	^{12}C	^{13}C	Intensity %
Tetrabromodiphenyl ether	$[M^{+}-2Br]$	$C_{12}H_6O^{79}Br_2$	4,00 to 8,50	323,879	335,919	51
	$[M^{+}-2Br]+2$	$C_{12}H_6O^{79}Br^{81}Br$		325,877	337,917	100
Pentabromodiphenyl ether	$[M^{+}-2Br]+2$	$C_{12}H_5O^{79}Br_2{}^{81}Br$	8,50 to 9,65	403,787	415,827	100
	$[M^{+}-2Br]+4$	$C_{12}H_5O^{79}Br^{81}Br_2$		405,785	417,825	98
Hexabromodiphenyl ether	$[M^{+}-2Br]+2$	$C_{12}H_4O^{79}Br_3{}^{81}Br$	9,65 to 11,70	481,698	493,738	68
	$[M^{+}-2Br]+4$	$C_{12}H_4O^{79}Br_2{}^{81}Br_2$		483,696	495,736	100
Heptabromodiphenyl ether	$[M^{+}-2Br]+4$	$C_{12}H_3O^{79}Br_3{}^{81}Br_2$	11,70 to 20,00	561,606	573,646	100
	$[M^{+}-2Br]+6$	$C_{12}H_3O^{79}Br_2{}^{81}Br_3$		563,604	575,644	98
BDE-209	$[M^{+}-2Br]+8$	$C_{12}O^{79}Br_4{}^{81}Br_4$	18,89	799,334	811,374	100
	$[M^{+}-2Br]+10$	$C_{12}O^{79}Br_3{}^{81}Br_5$		801,332	813,372	78

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Annex D
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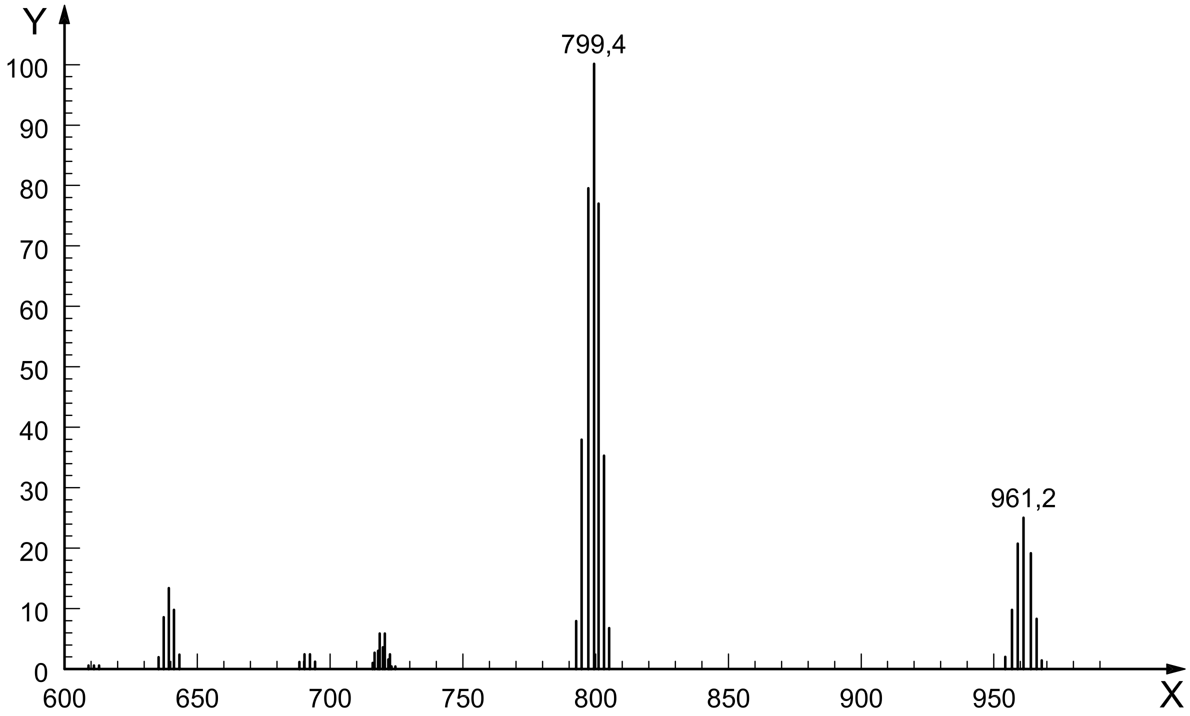
Examples of chromatograms and spectra



Key

X *m*/*z*

Y relative abundance

Figure D.1 — Spectrum of BDE-209 in electron impact ionization in the mass range from 600 *m*/*z* to 974 *m*/*z*

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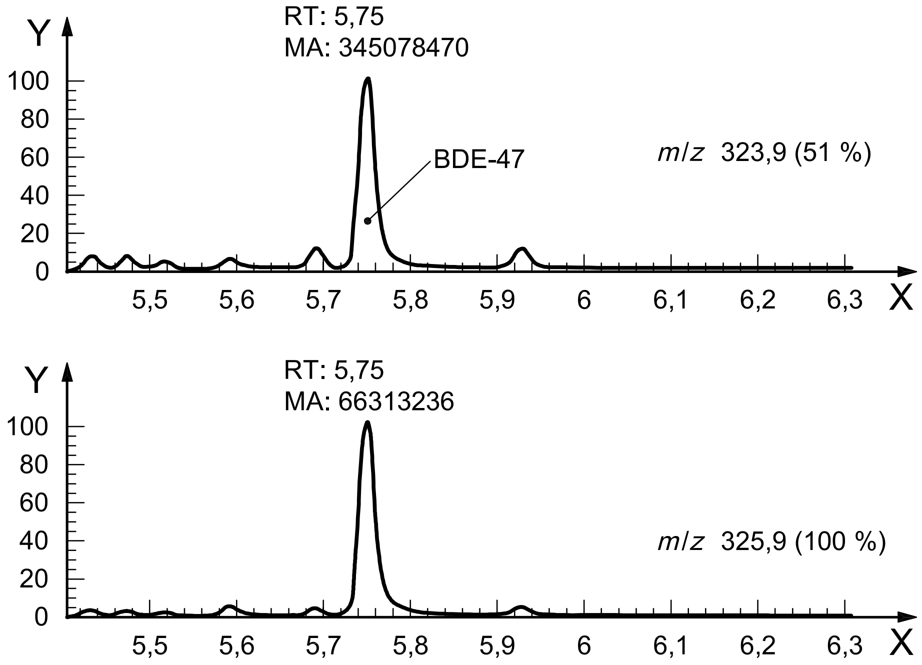
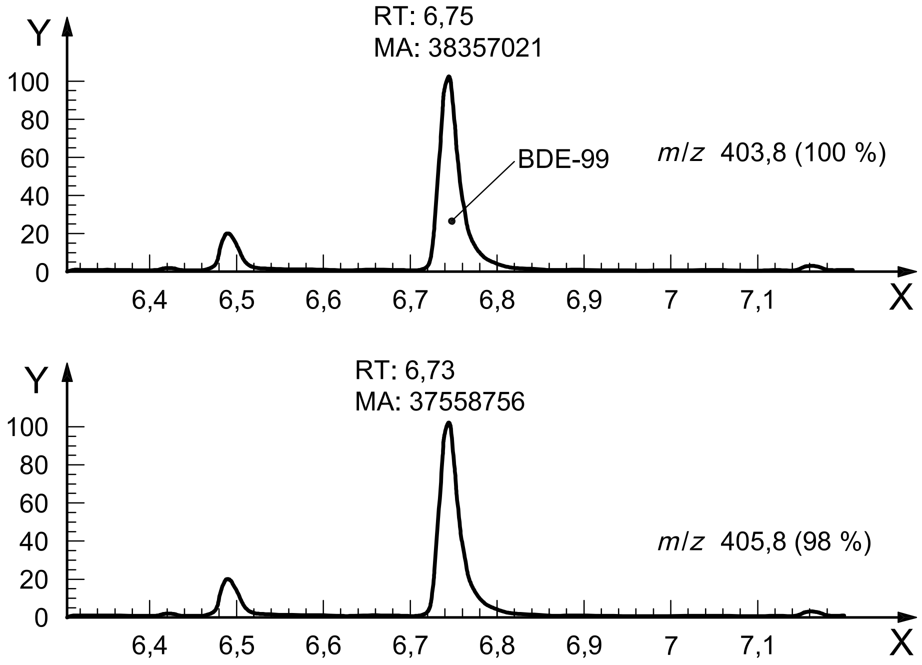


a) BDE-47 (506 pg); GC-MS conditions according to Example 2 in Annex B



b) BDE-100 (80,2 pg), BDE-99 (561 pg); GC-MS conditions according to Example 2 in Annex B

Figure D.2 — Chromatogram of a sewage sludge sample (*continued*)

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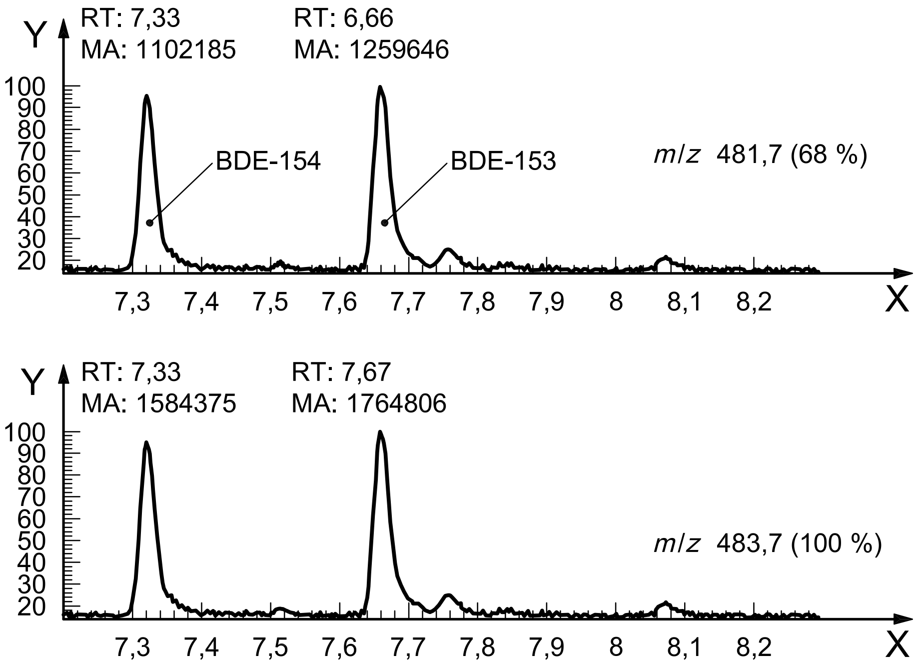
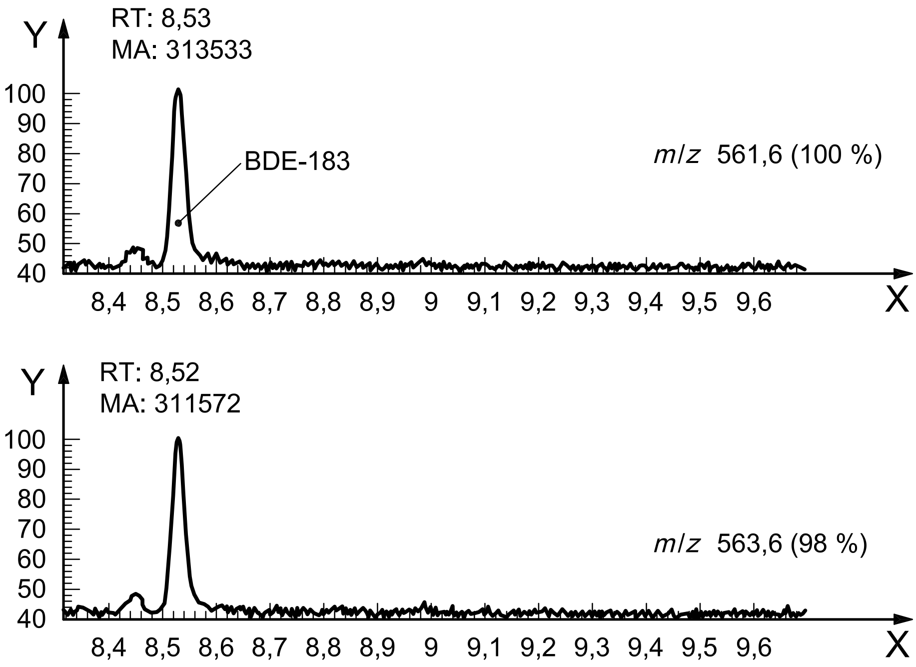


c) BDE-154 (39,4 pg), BDE 153 (53,4 pg); GC-MS conditions according to Example 2 in Annex B



d) BDE-183 (20,8 pg); GC-MS conditions according to Example 2 in Annex B

Figure D.2 — Chromatogram of a sewage sludge sample (*continued*)

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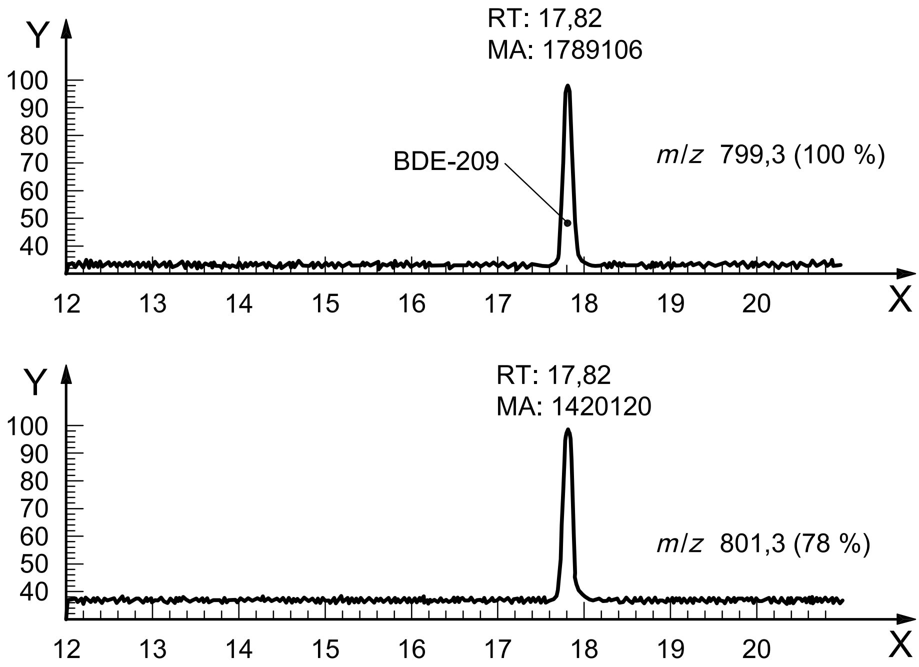

Key

X	time, min
Y	relative abundance
RT	retention time
MA	atomic mass

e) BDE-209 (10 259 pg); GC-MS conditions according to Example 2 in Annex B

Figure D.2 — Chromatogram of a sewage sludge sample

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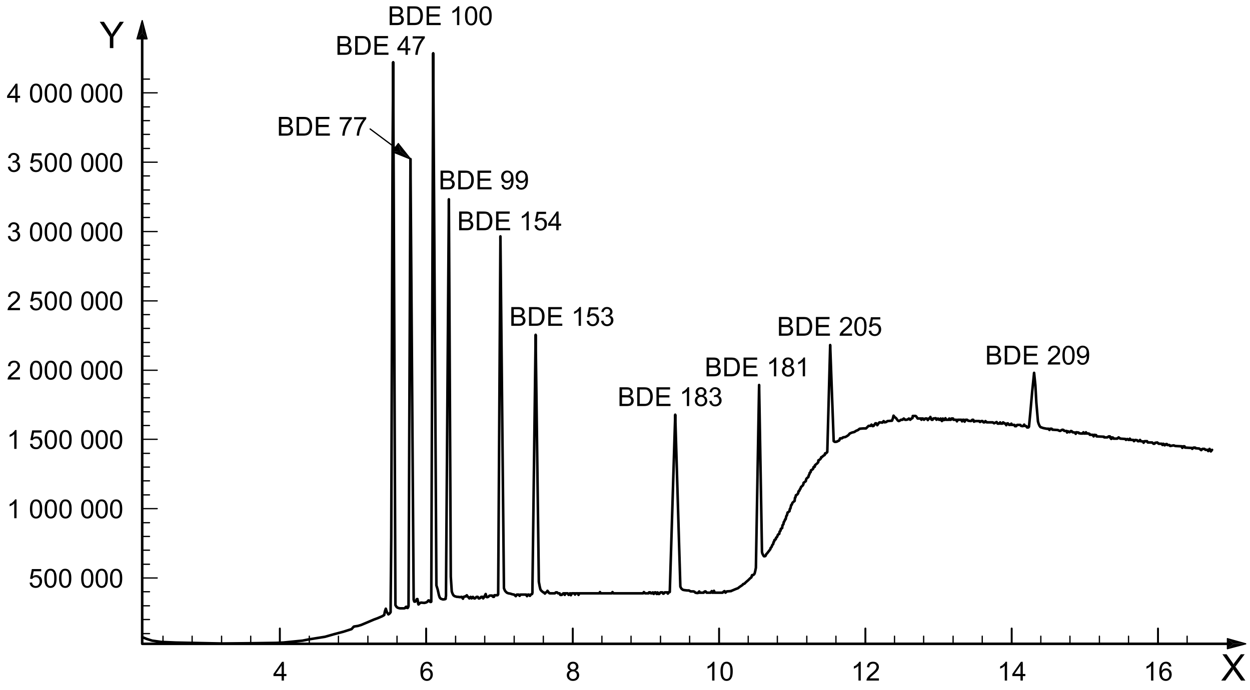

Key

X time, min

Y relative abundance

Figure D.3 — Chromatogram of a standard mix 500 pg/µl: TIC of the masses 79,05; 81,05; 160,9 and the specific masses 402,7 (BDE-100 and BDE-99) 482,7 (BDE-154 and BDE-153); 563,6 (BDE-183 and BDE-181); 641,5 (BDE-205); 488,6 (BDE-209); GC-MS conditions according to Example 3 in Annex B

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Annex E
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Precision data

Table E.1 — Precision data

Sample	**Matrix**	**Substance**	l	n	o %	$\bar{\bar{x}}$ µg/kg	s_R µg/kg	CV$_R$ %	s_r µg/kg	CV$_r$ %
1	Sediment	BDE-47	16	59	0,0	362	50,5	14,0	22,0	6,1
		BDE-100	16	57	0,0	93,3	28,96	31,0	8,38	9,0
		BDE-99	16	59	0,0	518	99,6	19,2	49,9	9,6
		BDE-154	16	57	0,0	39,2	9,11	23,2	2,46	6,3
		BDE-153	16	59	0,0	47,7	9,28	19,5	2,01	4,2
		BDE-183	16	58	1,7	2,48	0,425	17,1	0,195	7,8
		BDE-209	11	39	2,5	80,6	39,65	49,2	17,52	21,7
2	Sewage sludge	BDE-47	15	56	1,8	30,0	3,82	12,7	1,83	6,1
		BDE-100	15	53	3,6	6,39	1,230	19,3	0,659	10,3
		BDE-99	15	56	1,8	35,1	5,39	15,4	3,40	9,7
		BDE-154	15	55	0,0	2,94	0,952	32,4	0,475	16,2
		BDE-153	15	56	1,8	3,83	0,917	23,9	0,477	12,5
		BDE-183	15	57	0,0	3,59	0,813	22,6	0,539	15,0
		BDE-209	10	40	0,0	457	121,1	26,5	55,0	12,0

l is the number of laboratories;

n is the number of single results after elimination of outliers of the first kind (high deviation of a single value);

o is the percentage of outliers;

$\bar{\bar{x}}$ is the total mean after elimination of outliers;

s_R is the standard deviation between the laboratories;

CV$_R$ is the reproducibility coefficient of variation;

s_r is the standard deviation within the laboratories;

CV$_r$ is the repeatability coefficient of variation.

According to the standard, different clean-up methods (Annex A.1, A.2, A.3 and other) were applied. Toluene was used as the extraction solvent due to the requirements in respect to BDE-209.

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Bibliography

[1] EN 12880:2000, *Characterization of sludges — Determination of dry residue and water content*

[2] DIN 32645:1994, *Nachweis-, Erfassungs- und Bestimmungsgrenze*

[3] Eurachem Guide 1998, *The Fitness for Purpose of Analytical Methods*, ISBN 0-948926-12-0

[4] ISO/TR 13530 4), *Water quality — Guide to analytical quality control for water analysis*

[5] ISO 3696, *Water for analytical laboratory use — Specification and test methods*

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4) Under revision as ISO 13530.

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