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BSI Standards Publication

Characterization of waste — Determination of brominated flame retardants (BFR) in solid waste

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National foreword

This British Standard is the UK implementation of EN 16377:2013.

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A list of organizations represented on this committee can be obtained on request to its secretary.

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Characterization of waste - Determination of brominated flame retardants (BFR) in solid waste

Caractérisation des déchets - Détermination des retardateurs de flamme bromés (BFR) dans les déchets solides

Charakterisierung von Abfällen - Bestimmung bromierter Flammschutzmittel (BFR) in Feststoffabfall

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

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Foreword

This document (EN 16377:2013) has been prepared by Technical Committee CEN/TC 292 “Characterization of waste”, 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of an identical text or by endorsement, at the latest by March 2014, and conflicting national standards shall be withdrawn at the latest by March 2014.

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.

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Introduction

WARNING — Persons using this European Standard should be familiar with common 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 European Standard specifies a method for the determination of selected polybrominated flame retardants (BFR), chemically known as polybrominated diphenylethers (BDE), in waste materials using gas chromatography/mass spectrometry (GC-MS) in the electron impact (EI) ionisation mode (GC-EI-MS).

When applying GC-EI-MS, the method is applicable to samples containing 100 µg/kg to 5 000 µg/kg of tetra- to octabromodiphenylether congeners and 100 µg/kg to 10 000 µg/kg of decabromo diphenylether (see Table 1). It is also possible to analyse other brominated flame retardants applying the method described in this European Standard, provided the method's applicability has been proven.

Table 1 — Brominated flame retardants determined by this method

No.	Congener	Formula	Abbreviation[a]	Molar mass g/mol
1	2,2',4,4'-Tetrabromodiphenylether	$C_{12}H_6Br_4O$	BDE-47	485,795 0
2	2,2',4,4',5-Pentabromodiphenylether	$C_{12}H_5Br_5O$	BDE-99	564,691 1
3	2,2',4,4',6-Pentabromodiphenylether	$C_{12}H_5Br_5O$	BDE-100	564,691 1
4	2,2',4,4',5,6'-Hexabromodiphenylether	$C_{12}H_4Br_6O$	BDE-154	643,587 2
5	2,2',4,4',5,5'-Hexabromodiphenylether	$C_{12}H_4Br_6O$	BDE-153	643,587 2
6	2,2',3,4,4',5',6-Heptabromodiphenylether	$C_{12}H_3Br_7O$	BDE-183	722,483 2
7	Decabromodiphenylether	$C_{12}Br_{10}O$	BDE-209	959,171 4

[a] Numbering for the BDE according to IUPAC nomenclature for PCB.

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 14346, *Characterization of waste — Calculation of dry matter by determination of dry residue or water content*

EN 15002, *Characterization of waste — Preparation of test portions from the laboratory sample*

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

Brominated diphenylethers (BDE) are extracted from the dried sample using an organic solvent. In case of high levels of plastic waste matrices, cryo-grinding is required in order to provide particle sizes that allow the complete extraction of the analytes. Appropriate extraction techniques are soxhlet, sonication or pressurised fluid extraction. The obtained extracts are concentrated and cleaned up by column chromatography and Gel Permeation Chromatography (GPC).

Following the concentration and clean-up process, the brominated diphenylethers are separated by capillary gas chromatography and detected by mass spectrometry in the selected ion monitoring mode using electron impact ionisation (EI). Quantification is carried out by the internal standard method.

4 Interference

Naturally produced brominated compounds, such as halogenated bipyrrols or brominated phenoxyanisols can be considered as potential sources of interference.

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

5 Reagents and standards

Only use reagents with negligibly low concentrations of brominated diphenylethers (BDE), 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 (4 °C in the case of BDE). Temperatures < 4 °C may lead to precipitation.

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 solutions. Toluene (C_7H_8), or acetone (C_3H_6O), or a mixture of acetone (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}), cyclohexane (C_6H_{12}) or dichloromethane (CH_2Cl_2) for residual analysis.

Toluene is highly recommended, especially when the volume of the solvent is reduced to a minimum.

5.2 Standards

5.2.1 Calibration standards

BDE listed in Table 1 are used for calibration. Solutions of the calibration substances are commercially available.

5.2.2 Internal standards

Solutions of reference substances for use as internal standards for electron impact ionisation (Table 2) are commercially available.

Table 2 — Examples of internal standards for GC-EI-MS analysis

No.	Name	Formula	Abbreviation	Molar mass g/mol
1	2,2',4,4'-Tetrabromo[$^{13}C_{12}$]diphenylether	$^{13}C_{12}H_6Br_4O$	^{13}C-BDE-47	497,703 5
2	2,2',4,4',5-Pentabromo[$^{13}C_{12}$]diphenylether	$^{13}C_{12}H_5Br_5O$	^{13}C-BDE-99	576,599 5
3	2,2',4,4',5,5'-Hexabromo[$^{13}C_{12}$]diphenylether	$^{13}C_{12}H_4Br_6O$	^{13}C-BDE-153	655,495 5
4	2,2',3,4,4',5',6-Heptabromo[$^{13}C_{12}$]diphenylether	$^{13}C_{12}H_3Br_7O$	^{13}C-BDE-183	734,391 6
5	Decabromo[$^{13}C_{12}$]diphenylether	$^{13}C_{12}Br_{10}O$	^{13}C-BDE-209	971,079 7

5.2.3 Injection standard

To determine recovery rates for the internal standard in each sample e.g. dibromooctafluorobiphenyl ($C_{12}Br_2F_8$) is used as an injection standard.

5.2.4 Solutions of the single standards / internal standards

Use commercially available solutions or prepare stock solutions by dissolving 10 mg of each of the reference substances in toluene (5.2.1 and 5.2.2) in an amber 10 ml volumetric flask and bring to volume, resulting in a final concentration of 1 mg/ml. Store all solutions at approximately 4 °C in the dark to avoid potential photodegradation.

5.2.5 Multicomponent stock solution of standards

Accurately transfer between 100 µl to 500 µl of each single standard solution (5.2.4) into an amber 10 ml volumetric flask and bring to volume, resulting in final concentrations between 10 µg/ml and 50 µg/ml per substance.

5.2.6 Multicomponent stock solution of internal standards

Prepare a stock solution of the internal standards at an appropriate concentration in toluene (e.g. 2 µg/ml).

5.2.7 Calibration solutions

Prepare, e.g. seven, calibration solutions with concentrations according to the detection capacity of the mass spectrometer. Combine the multi-component stock solutions of standards (5.2.5), internal standards (5.2.6) and, if necessary, injection standard (5.2.3) to produce the solutions e.g. shown in Table 4 by appropriate dilution with toluene.

5.3 Sodium sulphate, anhydrous, Na_2SO_4, powdered.

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

5.5 Liquid nitrogen, to cool waste matrices for cryo-grinding.

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

5.7 Clean-up material, according to Annex A.

5.8 Baked sand and fine gravel

Bake sand and fine gravel (0,7 mm to 12 mm) for at least 8 h at 400 °C.

6 Equipment

6.1 Customary laboratory glassware

Clean all glassware by rinsing with acetone (5.1). Heating the glassware to 400 °C will reduce blanks. Heated volumetric apparatuses will require recalibration prior to use.

6.2 Freeze drying apparatus

6.3 Deep freezer

6.4 Mortar and pestle, or a grinding mill

6.5 Drying oven, capable of maintaining temperatures in the range of 100 °C to 550 °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 Extraction apparatuses

6.8.1 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, heating apparatus.

6.8.2 Sonication apparatus

Sonication should be applied for at least 3 h in a temperature controlled sonicator.

6.8.3 Pressurised fluid extraction apparatus

Consisting of extraction cells which can be heated to 150 °C at static pressures up to 10 MPa. The device should be programmable regarding the temperature, duration and number of extraction cycles. The cells need to be flushed with extraction solvent.

6.9 Evaporation device, such as rotary evaporator, turboevaporator 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, programmable temperature vaporiser (PTV) or an on-column injection port coupled to a mass spectrometer with electron impact ionisation (GC-EI-MS).

6.17 Analytical column

Fused silica column with non-polar low bleed separating phase, providing thermal stability up to 400 °C; e.g. inner diameter < 0,25 mm, length 15 m, film thickness of 0,1 µm is recommended. See Annex B for examples.

7 Sample pretreatment

Prepare the laboratory sample as specified in EN 15002. Store and transport in the dark at approximately 4 °C. Pretreat the samples in the laboratory by homogenising and freeze-drying. Grind the samples (6.4) and sieve (6.6) according to the requirements of the analytical task.

Waste matrixes may have to be ground carefully before being extracted. In order to avoid analyte losses and decomposition of matrix components, cryo-grinding with liquid nitrogen is recommended. Hereby, the sample is cooled in liquid nitrogen before grinding. Use a mill that allows cooling with liquid nitrogen during grinding (especially in case of plastic matrices).

8 Procedure

8.1 Blank determination

Regardless of the selected extraction procedure, perform a blank determination using the same amount of reagents that are used for the pretreatment, extraction, clean-up and analysis of a sample. Analyse the blank immediately prior to the analysis of the samples to prove that samples are free of contaminants.

The concentrations of the blank should be negligible, compared with the concentrations of BDEs to be determined.

8.2 Extraction

8.2.1 General

^{13}C-labelled compounds as internal standards are expensive and would need to be added in extremely high amounts in order to match the concentrations of the analytes to be expected in plastic containing waste matrices (much higher than in case of sludge and sediments, for example). Furthermore, the analytes are bound within the matrix particles and have to diffuse into the extraction solvents while ^{13}C-labelled standard compounds added prior to extraction are dissolved directly. Thus, the idea of compensating for incomplete extraction by addition of ^{13}C-labelled standard compounds is obsolete. These two reasons suggest ^{13}C-labelled standard compounds should be added after extraction and before clean-up (8.3). If ^{13}C-labelled compounds are added to the sample before extraction an internal standard solution containing for example 2 µg/ml of each labelled compound is recommended (ISTD I). If ^{13}C-labelled compounds are added after extraction and before clean-up to an aliquot of the whole extract, an internal standard solution containing for example 0,2 µg/ml of each labelled compound is recommended (ISTD II).

Protect samples and extracts carefully from sunlight to avoid photodegradation of the higher brominated analytes.

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

8.2.2 Soxhlet extraction

Transfer a suitable mass, e.g. 1 g to 3 g, of the pretreated, dry sample into a Soxhlet thimble. Depending on the expected concentration in the sample, optionally add between 200 µl and 800 µl of the internal standard solution (ISTD I, 5.2.4), to the Soxhlet thimble. Place the thimble in the Soxhlet extractor.

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 recommended. 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, reduce the extract volume to below 50 ml using a suitable evaporation device (6.9) and make up to 100 ml in a volumetric flask.

Other extraction techniques, e.g. accelerated solvent extraction, and shorter extraction times may be used after performing a comparability exercise with a 16 h Soxhlet extraction. In case of waste matrices, especially those containing portions of plastic with technically incorporated BDEs and/or decabromodiphenylethane (DBDPE), sonication and pressurised fluid extraction are likely to be successful and lead to shorter extraction times. Solvents other than toluene, especially solvents of higher polarity, are likely to extract more matrix components which might complicate subsequent clean-up. The use of highly polar solvents should be carefully tested for comparability especially regarding the effectiveness of the clean-up procedure.

8.2.3 Sonication

Alternatively transfer a suitable mass, e.g. 1 g to 3 g of the pretreated, dry sample into a sealable glass container (50 ml), optionally add between 200 µl and 800 µl of the internal standard solution (5.2.4) and then 20 ml of toluene and sonicate the closed vial for 3 h. Collect the solvent and repeat this procedure with another portion of toluene (20 ml). Pool the toluene extracts and fill up to 50 ml in a volumetric flask.

8.2.4 Pressurised fluid extraction

Alternatively transfer a suitable mass, e.g. 1 g to 3 g, of the pretreated, dry sample into an extraction cell. Optionally add between 200 µl and 800 µl of the internal standard solution (5.2.3). Fill the void volume with baked sand or fine gravel (see 5.8). To avoid clogging of the frit it may be useful to mix the sample with sand or fine gravel before filling into the cell. The following conditions for one extraction cycle were seen to be successful: heating period of the cell 5 min at 100 °C and 140 bar; static extraction period 5 min (solvent: toluene; flush: 60 %; purge: 60 s). It is recommended that two additional cycles be performed. The total extract volume of about 40 ml is to be made up to a volume of 50 ml in a volumetric flask.

8.3 Clean-up

For clean-up, a suitable aliquot of the extract (8.2), e.g. 1/10, is taken and if the internal standard was not added before extraction (ISTD I, see 8.2) add now the isotope standards (e.g. 250 µl of ISTD II, see 8.2). The extract is evaporated under reduced pressure to a volume of 0,5 ml. Evaporation is not done to complete dryness because polymers from the matrix once precipitated as dry film might not re-dissolve and lead to analyte loss.

Depending on the different sample matrices encountered, a variety of extract clean-up procedures may be suitable. Examples of clean-up procedures are given in Annex A. The suitability of the chosen clean-up procedure can be checked for each sample on the basis of the recovery of the internal standards.

NOTE In most cases, using a clean-up given in A.1 followed by the procedure in A.3 and A.2 is successful.

8.4 Measurement

Optimise 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 (5.2.3), 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 time during the injection and separation stages, because of the thermal degradation of BDE-209 at temperatures higher than 300 °C. Optimise the injection step, paying special attention to the peak height of BDE-209.

In case of GC-MS with electron impact ionisation BDE-209 can exhibit an exponential increase of detector response factor with increasing concentrations of the calibration standards. Therefore, it is necessary to use ^{13}C-labelled BDE-209 as an internal standard to obtain a linear range of the ratio native/labelled compound over a reasonable concentration range.

8.5 Identification

Consider an analyte 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 ± 25 % of the theoretical bromine isotope ratio.

The following ions (Table 3) are proposed for electron impact ionisation 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 recognisable bromine-pattern for identification of each compound. It is also possible to use more than one ion for quantification.

Table 3 — Ions for electron impact ionisation detection

Compound	**Ions for quantification** *m/z*	**Ions for qualification** *m/z* (%)
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	799,4	959,4 (15), 797,4 (100)
^{13}C-BDE-47	339,9	495,7 (69,5), 497,7 (100)
^{13}C-DBE-99	417,7	575,6 (100), 577,6 (98)
^{13}C-DBE-153	497,7	653,5 (100), 655,5 (73,4)
^{13}C-DBE-209	811,4	809,4 (76), 811,4 (100)

Ensure that ions are free of interferences caused by matrix components.

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

EN ISO 22892 may also be consulted to assist in identification of BDE.

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 4 as an example of concentrations for evaluating a linear range over several decades of concentration. The linear relationship should be ensured with the concentration- and response relationships as used in internal standardization (see 9.3).

Plot, for example, the ratio values $\frac{y_i}{y_{is,i}}$ (peak areas, peaks heights or integration units) for each substance *i* on the ordinate and the associated ratio of mass concentrations $\frac{\rho_i}{\rho_{is,i}}$ on the abscissa.

Check the graphical representation of the calibration data with descriptions in ISO 8466-1.

The MS response in case of BDE-209 may be not linear; the respective ^{13}C-labelled compound compensates this phenomenon.

9.2.2 Two point calibration over the total linear range

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.

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 according to Formula (1):

$$S_\rho = \sqrt{\frac{\sum_{i=1}^{N}(\rho_1-\rho)^2}{N}} \quad (1)$$

where

s_ρ is a measure of the accuracy of the calibration;

ρ_i is the measured mass concentration of the certified reference standard;

ρ is the known mass concentration of the certified reference standard;

N is the number of measurements.

9.2.4 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.

Table 4 — Example concentrations in solutions for evaluating the linear range

Compound	**Solution 1** ng/ml	**Solution 2** ng/ml	**Solution 3** ng/ml	**Solution 4** ng/ml	**Solution 5** ng/ml	**Solution 6** ng/ml	**Solution 7** ng/ml
BDE-47	10	50	250	500	750	1 500	2 500
BDE-99	10	50	250	500	750	1 500	2 500
BDE-100	10	50	250	500	750	1 500	2 500
BDE-153	10	50	250	500	750	1 500	2 500
BDE-154	10	50	250	500	750	1 500	2 500
BDE-183	10	50	250	500	750	1 500	2 500
HBCD	10	50	250	500	750	1 500	2 500
Internal standards for EI							
^{13}C-BDE-47	500	500	500	500	500	500	500
^{13}C-BDE-99	500	500	500	500	500	500	500
^{13}C-BDE-153	500	500	500	500	500	500	500
^{13}C-BDE-183	500	500	500	500	500	500	500
^{13}C-HBCD	500	500	500	500	500	500	500

9.3 Calibration with internal standards

The use of an internal standard for the determination of the concentration minimises both possible errors made during injection and by sample losses during sample pretreatment steps, and furthermore, 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 quantification 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 of 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 (9.5).

See Table 4 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 BDE 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. $\frac{y_i(1)}{y_{is,i}(1)}$; $\frac{y_i(2)}{y_{is,i}(2)}$; $\frac{y_i(3)}{y_{is,i}(3)}$ at two concentration levels.

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

$$\frac{y_i}{y_{is,i}} = a_i \frac{\rho_i}{\rho_{is,i}} + b_i \qquad (2)$$

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;

a_i is the slope of the calibration function of substance *i,* the unit depends on the evaluation;

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

$y_{is,i}$ is the measured response of the internal standard for the substance *i*, the unit depends on the evaluation, for example, area value;

$\rho_{is,i}$ 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 Quantification with the internal standard

The mass concentration of the internal standard $\rho_{is,i}$ 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.

Inject identical volumes of the sample extracts as injected as calibration solutions.

Calculate the mass concentration $\rho_{i,sample}$ of the substance using Formula (3):

$$\rho_{i,\,sample} = \frac{\frac{y_{i,\,sample}}{y_{is,\,i,\,sample}} - b_i}{a_i} \cdot \frac{m_{is,\,i,\,sample} \cdot 100}{m_{sample} \cdot d_m} = \frac{\rho_{i,\,sample\ extract}}{\rho_{is,\,i,\,sample\ extract}} \cdot \frac{m_{is,\,i,\,sample} \cdot 100}{m_{sample} \cdot d_m} \quad (3)$$

where

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

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

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

$\rho_{is,i,sample\ extract}$ 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;

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

$m_{is,i\ sample}$ is the mass of the added internal standard substance, in micrograms, µg;

m_{sample} is the sample mass in kilogram, kg;

a_i see Formula (2);

b_i see Formula (2);

d_m is the dry matter content of the sample, determined according to EN 14346.

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. 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 $A_{is,i,sample}$ is calculated according to Formula (4).

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

where

$A_{is,i,sample}$ is the recovery of the internal standard, for the substance *i*; in percent, %;

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

$y_{is,i,calilbration}$ is the measured response, e.g. peak area, of the internal standard, for substance *i*, in the calibration solution;

$y_{is,inj,\,sample}$ is the measured response, e.g. peak area, of the injection internal standard, in the sample;

$y_{is,inj,calibration}$ 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 BDE.

NOTE Formula (3) gives the mass concentration $\rho_{i,sample}$ in µg/kg as described in EN ISO 22032. As the concentrations are often much higher in waste materials, reporting of results in mg/kg might be more appropriate.

Two significant figures would be, for example: 21 mg/kg; 2,1 mg/kg; 0,21 mg/kg.

10 Test report

The test report shall include at least the following information:

a) reference to this European Standard (EN 16377);

b) identity of the sample;

c) sample storage and pretreatment;

d) mesh size of the sieve applied in sieving or grinding;

e) complete description of the procedure;

f) identification and quantification of single components;

g) expression of results according to 9.6.

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

Annex A
(normative)

Clean-up procedures

A.1 Multi-layer column chromatography I (Al_2O_3), column size: 22 mm × 190 mm

A.1.1 Reagents

A.1.1.1 Neutral aluminium oxide (Al_2O_3) 90, activity neutral 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.1.1.2 Sodium sulphate, Na2SO4, anhydrous, baked at 550 °C.

A.1.2 Preparation of clean-up column

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

— BOTTOM: glass wool plug;

— 1 cm to 2 cm sodium sulphate (A.1.1.2);

— 25 g aluminium oxide (A.1.1.1); (this layer is focussed on the removal of polar and acid compounds);

— TOP: 1 cm to 2 cm sodium sulphate (A.1.1.2).

A.1.3 Procedure

— dilute the extract (8.2) or (A.2.3) 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, since it contains e.g. hydrocarbons;

— secondly, elute the BDE using 200 ml of a mixture of hexane:dichloromethane (1:1);

— concentrate this eluate to a final volume, e.g. 0,5 ml, use toluene as a keeper.

Optimise the elution volumes when first applying the procedure and also each time the properties of the aluminium oxide (A.1.1.1) have changed.

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

A.2.1 Reagents

A.2.1.1 Silica 60, 63 µm to 200 µm; bake at 250°C for 12 h.

A.2.1.2 Silica treated with silver nitrate

45 g of silica (A.2.1.1), 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.2.1.3 Silica treated with sulfuric acid

56 g of silica (A.2.1.1), 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.2.1.4 Silica treated with sodium hydroxide

33 g of silica (A.2.1.1), 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.2.1.5 Sodium sulphate, Na_2SO_4, anhydrous. Bake the sodium sulfate at 550°C for 12 h.

A.2.1.6 Ultrapure water

A.2.1.7 Glass wool

A.2.2 Preparation of clean-up multi-layer column

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

— BOTTOM: glass wool plug;

— 2 g of silica (A.2.1.1);

— 5 g of silica/silver nitrate (A.2.1.2) to remove sulfur and sulfur-containing molecules;

— 2 g of silica (A.2.1.1);

— 5 g of silica/sodium hydroxide (A.2.1.4) to remove acid compounds;

— 2 g of silica (A.2.1.1);

— 10 g of silica/sulfuric acid (A.2.1.3) to remove basic and aromatic compounds;

— TOP: 10 g of sodium sulphate (A.2.1.5) to remove small amounts of water.

If this silica multilayer column is applied after the multi-layer column (A.1, see recommendation in 8.3), it is recommended to reduce the volume to 1/10 of the above mentioned amounts, i.e. 0,2 g of silica gel; 0,5 g of silica gel/NaOH; 0,2 g of silica gel; 1,0 g of silica gel/sulfuric acid; 0,2 g of silica gel; 0,5 g of silica gel/silver nitrate; 1,0 g of sodium sulphate.

A.2.3 Procedure

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

Reduce the extract (8.2 or A.1.3) to 2 ml and transfer to the column. Rinse the flask twice with 2 x 2 ml hexane (5.1).

Elution: Use 50 ml of cyclohexane (5.1) followed by 50 ml of cyclohexane:dichloromethane (80:20).

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

If the volume of this column is reduced to 1/10 (A.2.2), reduce the extract (A.1.3) to 200 µl and reduce eluents to 5 ml of cyclohexane (C_6H_{12}) followed by 5 ml of cyclohexane:dichloromethane (80:20).

If this silica multilayer is not applied after column A.1 (see 8.2) and GPC (see 8.2 and A.3), it is recommended to evaporate the extract (8.2) to not less than about 0,5 ml. Otherwise co-extracted polymers might precipitate and complicate further clean-up steps.

A.3 Gel permeation chromatography (GPC)

A.3.1 General

This protocol exemplifies experimental conditions suitable for removing interfering compounds with a molecular size significantly different from BDE, e.g. polymers extracted from the waste matrix.

A.3.2 GPC apparatus

Automatic 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 × L, 24,4 mm × 580 mm), variable length, filled with, e.g. 50 g Bio-Beads®[1)] S-X3 (column bed approximately 350 mm).

Alternatively, a chromatography glass column (ID × L, 35 cm × 60 cm) may be used to perform a manual GPC (A.3.7).

A.3.3 Evaporation apparatus

A.3.4 Glassware

— Glass vial, e.g. graduated with cap and PTFE septum (PTFE = polytetrafluoroethylene);

— 5 ml volumetric flask (amber);

— Concentration sample tube, solvent capacity 500 ml) with 0,5 ml stem;

— Microlitre syringe 100 µl;

— Pasteur pipettes (e.g. 2 ml).

A.3.5 Reagents

A.3.5.1 **Toluene**, (C_7H_8) for residue analysis.

A.3.5.2 **Cyclohexane**, (C_6H_{12}) for residue analysis.

A.3.5.3 **Ethyl acetate**, ($C_4H_8O_2$), for residue analysis.

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1) Bio-Beads® is the trademark of a product supplied by Bio-Rad Laboratories, Inc. This information is given for the convenience of users of this European Standard and does not constitute an endorsement by CEN of the product named. Equivalent products may be used if they can be shown to lead to the same results.

A.3.5.4 **Eluent**, Cyclohexane:Ethyl acetate (1:1).

A.3.5.5 **Bio-Beads®[1)] S-X3,** 38 µm to 75 µm (200 mesh to 400 mesh).

A.3.5.6 **Syringe standard**, e.g. dibromooctafluorobiphenyl in toluene, 200 pg/µl.

A.3.6 Automatic GPC clean-up procedure

A.3.6.1 General

Concentrate the extract obtained after column chromatography clean-up (A.1) to a final volume of 0,5 ml using the concentration device. Dissolve immediately in a mixture of ethyl acetate:cyclohexane (1:1). For automatic GPC, 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.3.5.5) to clean-up the extract. A total of about 300 ml is needed for one clean-up.

A.3.6.2 Automatic GPC clean-up conditions

Injection volume:	4 ml
Flow:	5 ml/min of eluent (A.3.5.4)
Dump:	100 ml (0 min to 20 min)
Collect:	150 ml (20 min to 50 min)
Wash:	50 ml (50 min to 60 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 BDE 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. Proceed with further clean-up (e.g. A.2).

A.3.7 Manual GPC clean-up

Alternatively to automatic GPC (A.3.6), a manual GPC clean-up can be performed using a glass column (A.3.2) filled with a suspension of 50 g of Bio-Beads®[1)] S-X3 (A.3.5.5) in the eluent (A.3.5.4). Cover the Bio-Beads®[1)] material bubble-free with ethyl acetate:cyclohexane 1:1, allow settling of the material for 2 h and condition with 0,5 l of eluent (A.3.5.4) prior to first use.

Concentrate the extract obtained after column chromatography clean-up (A.1) to a final volume of 0,5 ml using the concentration device. Dissolve immediately in 2 ml of ethyl acetate:cyclohexane (1:1) and transfer onto the chromatography glass column. Then, 300 ml of the eluent (A.3.5.4) are put onto the column. Adjust an eluent flow of approximately 5 ml/min.

Discard: 100 ml

Collect: 200 ml

Annex B
(informative)

Examples of separation conditions in gas chromatography and mass spectrometry

Example 1: 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	
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:	e.g. DB5-MS[2)] (5 % Phenyl) – 95 % Methylpolysiloxane)	
Temperature programme:	110 °C (0,2 min) → 30 °C/min to 200 °C → 20 °C/min to 340 °C (12,8 min)	

2) DB-5 and ZB-5 are examples of suitable products available commercially. This information is given for the convenience of users of this European Standard and does not constitute an endorsement by CEN of these products.

Example 2: GC-EI-MS conditions

Injection:	PTV, splitless, pressure-pulse injection	
Injector temperature:	90 °C (0,1 min), 4 °C/s to 360 °C (15 min)	
Injection volume:	2 µl	
Transfer line temperature:	300 °C	
Ion source:	280 °C	
EI energy:	70 eV	
Resolution:	low, 1 mass unit	
Acquisition mode:	SIM	
Quadrupol:	150 °C	
Flow rate:	1,0 ml/min constant	
Carrier gas:	helium	
Capillary column:	length:	15 m
	inner diameter:	0,25 mm
	film thickness:	0,1 µm
Column material:	e.g. ZB5-MS[2)] (5 % Phenyl) – 95 % Methylpolysiloxane)	
Temperature programme:	80 °C (1 min) → 20 °C/min to 340 °C (5 min)	

Annex C
(informative)

Typical ions and time windows for electron impact ionisation detection

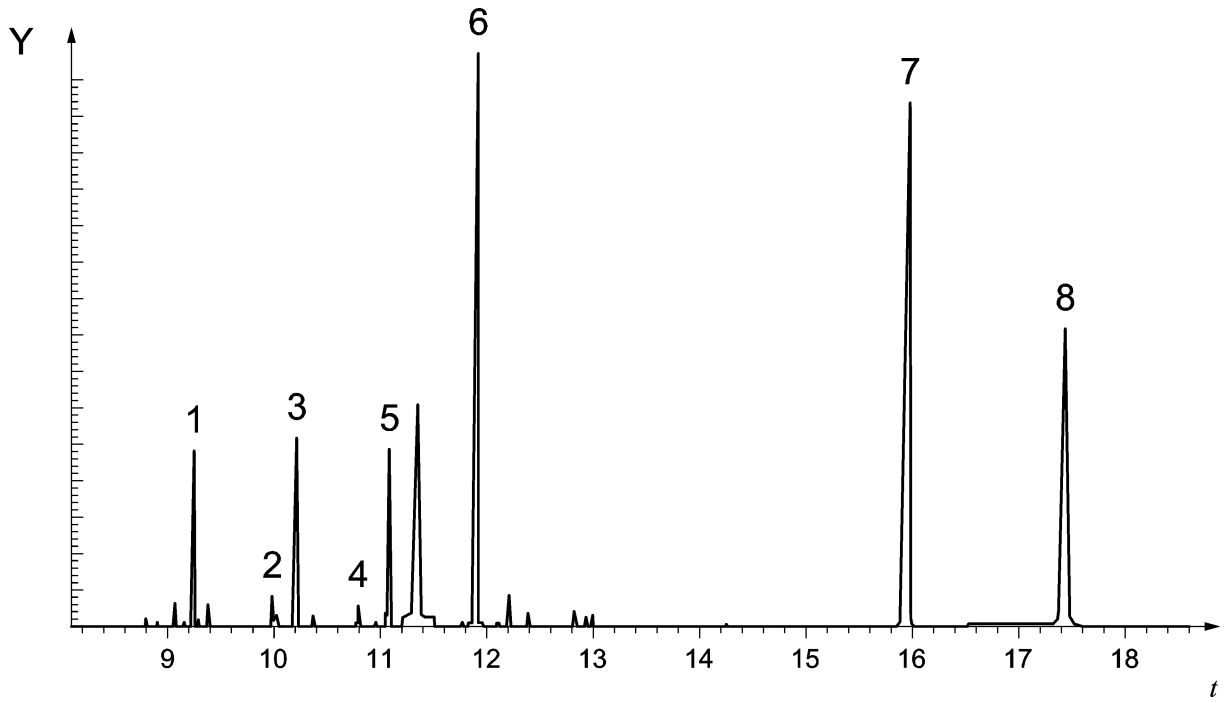
Table C.1 gives typical ions and time windows for the compounds analysed with GC-EI-MS with conditions according to Example 2 in Annex B:

Table C.1 — Typical ions and time windows for electron impact ionisation detection

Compound	Ion	Formula	Time window min	^{12}C *m/z*	^{13}C *m/z*	Intensity %
Tetrabromodiphenylether	$[M^+-2Br]$	$C_{12}H_6O^{79}Br_2$	8,00 to 9,50	323,879	335,919	51
	$[M^+-2Br]+2$	$C_{12}H_6O^{79}Br^{81}Br$		325,877	337,917	100
Pentabromodiphenylether	$[M^+-2Br]+2$	$C_{12}H_5O^{79}Br_2{}^{81}Br$	9,70 to 10,40	403,787	415,827	100
	$[M^+-2Br]+4$	$C_{12}H_5O^{79}Br^{81}Br_2$		405,785	417,825	98
Hexabromodiphenylether	$[M^+-2Br]+2$	$C_{12}H_4O^{79}Br_3{}^{81}Br$	10,60 to 11,50	481,698	493,738	68
	$[M^+-2Br]+4$	$C_{12}H_4O^{79}Br_2{}^{81}Br_2$		483,696	495,736	100
Heptabromodiphenylether	$[M^+-2Br]+4$	$C_{12}H_3O^{79}Br_3{}^{81}Br_2$	11,60 to 12,00	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	$[M^+-2Br]+8$	$C_{12}O^{79}Br_4{}^{81}Br_4$	15,50 to 16,50	799,334	811,374	100
	$[M^+-2Br]+10$	$C_{12}O^{79}Br_3{}^{81}Br_5$		801,332	813,372	78

Annex D
(informative)

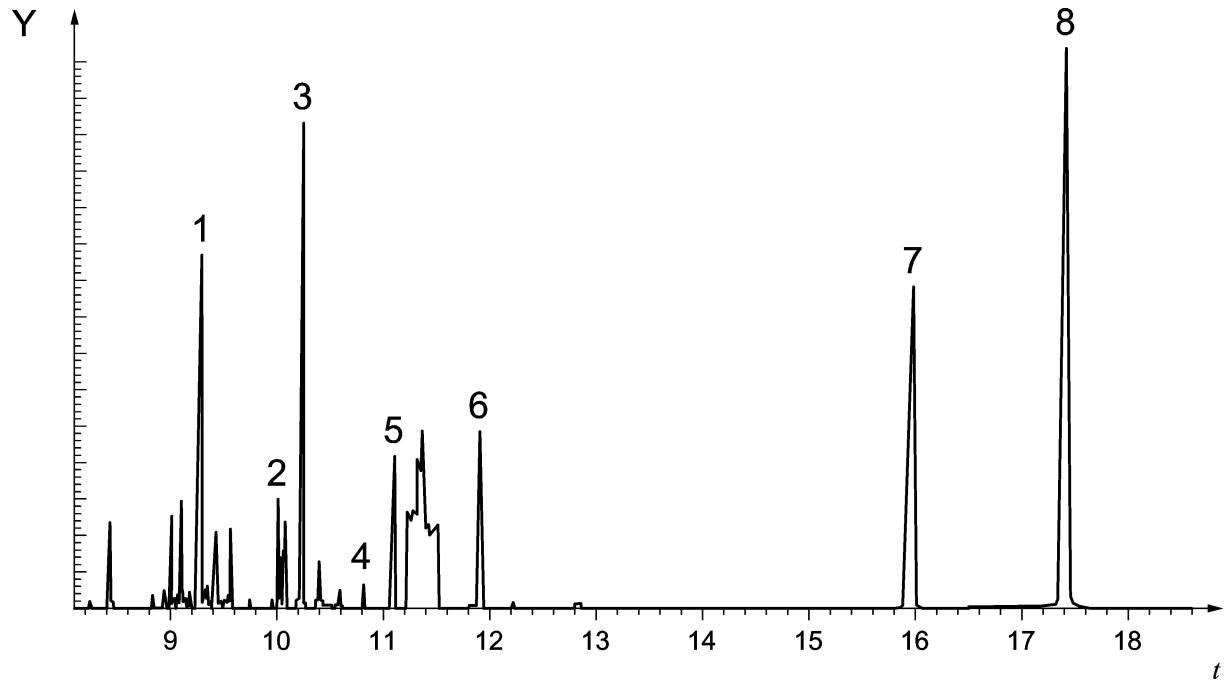
Examples of chromatograms



Key

Y	relative abundance		
t	time in minutes		
1	BDE/MBDE 47	5	BDE/MBDE 153
2	BDE/MBDE 100	6	BDE/MBDE 183
3	BDE/MBDE 99	7	BDE/MBDE 209
4	BDE/MBDE 154	8	DBDPE/MDBDPE

Figure D.1 — GC-MS-Chromatogram of an electronic waste sample after clean-up (both columns and GPC), showing the BDE congeners and DBDPE [decabromodiphenylethane]



Key

Y	relative abundance		
t	time in min		
1	BDE/MBDE 47	5	BDE/MBDE 153
2	BDE/MBDE 100	6	BDE/MBDE 183
3	BDE/MBDE 99	7	BDE/MBDE 209
4	BDE/MBDE 154	8	DBDPE/MDBDPE

Figure D.2 — GC-MS-Chromatogram of a shredder light fraction sample after clean-up (both columns and GPC), showing the BDE congeners and DBDPE [decabromodiphenylethane]

Annex E
(informative)

Precision data

Table E.1 — Validation data

	Matrix	Substance	*l*	*n*	n_{AP}	$\bar{\bar{x}}$ mg/kg	s_R mg/kg	CV_R %	s_r mg/kg	CV_r %
1	Filter dust	BDE-47	12	46	17,3	0,925	0,355	38,44	0,067	7,27
1		BDE-100	10	38	21,0	0,176	0,083	46,92	0,018	10,44
1		BDE-99	12	46	8,7	1,329	0,488	36,69	0,142	10,68
1		BDE-154	10	38	10,5	0,115	0,038	33,27	0,012	10,45
1		BDE-153	11	42	19,0	0,277	0,064	28,35	0,019	8,54
1		BDE-183	12	46	21,7	0,59	0,25	42,33	0,058	9,89
1		BDE-209	9	35	20,0	19,21	10,85	56,46	1,01	5,26
2	Shredder light fraction	BDE-47	12	48	0	1,074	0,477	44,43	0,297	27,65
2		BDE-100	9	36	0	0,157	0,078	49,58	0,029	18,8
2		BDE-99	12	48	0	1,572	0,726	46,16	0,444	28,26
2		BDE-154	11	44	18,2	0,124	0,042	33,74	0,032	25,97
2		BDE-153	11	44	9,1	0,401	0,160	39,89	0,111	27,66
2		BDE-183	12	48	16,7	1,173	0,627	53,49	0,51	43,51
2		BDE-209	9	36	22,2	13,37	8,40	62,82	3,81	28,47
3	Shredder light fraction	BDE-47	12	46	26,0	2,548	0,95	37,29	0,142	5,58
3		BDE-100	10	38	21,1	0,228	0,135	59,12	0,018	7,81
3		BDE-99	12	46	8,7	3,337	1,167	34,98	0,449	13,44
3		BDE-154	11	42	0	0,362	0,253	77,41	0,069	21,12
3		BDE-153	12	46	0	1,02	0,506	49,59	0,257	25,21
3		BDE-183	12	46	8,7	3,129	1,704	54,46	1,004	32,09
3		BDE-209	9	33	10,0	65,02	39,15	60,21	12,52	19,26
5	Electro waste	BDE-47	12	45	8,9	4,032	2,053	50,93	0,84	20,82
5		BDE-100	10	37	21,6	0,413	0,214	51,85	0,038	9,22
5		BDE-99	12	45	17,8	5,441	2,665	48,97	0,772	14,18
5		BDE-154	12	45	26,7	1,304	0,281	21,59	0,211	16,15
5		BDE-153	12	45	8,9	7,599	4,043	53,2	0,69	9,08
5		BDE-183	12	45	17,8	45,42	22,98	50,59	3,95	8,69
5		BDE-209	9	34	8,8	338,6	281,5	83,1	52,8	15,6

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);
n_{AP} is the percental rate of outliers (eliminated single results);

$\overline{\overline{x}}$	is the total mean after elimination of outliers;
s_R	is the standard deviation between the laboratories;
CV_R	is the reproducibility variation coefficient;
s_r	is the standard deviation within the laboratories;
CV_r	is the repeatability variation coefficient.

Annex F
(informative)

Summary of general requirements and recommendations

The purpose of this summary is to support the organisation of sampling and sample pre-treatment processes. The information given should be helpful for preparing a sampling plan.

Requirements (Table F.1) not mentioned in the normative part of this document should be considered as recommendations.

Table F.1 — General requirements and recommendations

Matrix restrictions	Solid waste
Typical working range	Above 0,1 mg/kg
Sampling instrument	Stainless steel equipment; avoid contact to plastics
Pretreatment of sample container	Clean and dry
Material of sample container	Glass, stainless steel
Transport conditions	Ambient temperature or lower
Preservation	No
Storage conditions	About 20 °C (ambient temperature) or lower
Drying procedure	Air drying at maximum 40 °C or freeze drying (depending on material)
Particle size reduction	Crushing, cutting, grinding (depending of material)
Particle size	Less than 2 mm recommended (plastics: 0,5 mm)
Laboratory sample	About 1 kg depending on homogeneity and particle size of material
Test portion	About 20 g

Bibliography

[1] EN ISO 22032, *Water quality — Determination of selected polybrominated diphenyl ethers in sediment and sewage sludge — Method using extraction and gas chromatography/mass spectrometry (ISO 22032)*

[2] EN ISO 22892, *Soil quality — Guidelines for the identification of target compounds by gas chromatography and mass spectrometry (ISO 22892)*

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