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NORMAN

Network of reference laboratories and related organisations for monitoring and bio monitoring of emerging environmental pollutants

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Report on the 2nd inter-laboratory study C3-II

Analysis of decaBDE in dust and sediment

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1 Objective

The objective of this second interlaboratory study was to validate and harmonise the analytical methodology for the analysis of decaBDE in environmental samples, and to transfer the knowledge from expert to routine laboratories. Decabromodiphenyl ether (decaBDE), an emerging pollutant that belongs to the group of brominated flame retardants, seems to be an ideal example for this case study. On the one hand there is the need for monitoring according to the recently completed risk assessment (EUR 20402 EN) and on the other hand there is still a need for improvement in the analysis of decaBDE in many laboratories (de Boer & Cofino 2002, de Boer & Wells 2006). A sequential approach was followed, starting with expert laboratories that harmonised the method and identify the critical steps (first interlabotory study, Duffek, 2007), followed by the current second interlaboratory study to test and transfer the method to the routine level.

Based on the results of the first interlaboratory study the second round was organised with routine and some expert laboratories. Due to the difficulties in the analysis of decaBDE in environmental samples advice on critical steps for the analysis of decaBDE were given.

This report summaries the results of the 2nd interlaboratory study including statistical evaluation and a critical assessment of the results.

2 Participants

Ten laboratories from eight countries participated in this study.

a. Centre for Environment, Fisheries and Aquaculture Science, Burnham on Crouch, United Kingdom
b. Landersamt für Natur, Umwelt und Verbraucherschutz NRW, Düsseldorf, Germany
c. Unilever, London, United Kingdom
d. Waterdienst, Lelystad, The Netherlands
e. Institute for Environmental Studies, VU University, Amsterdam, The Netherlands
f. EMPA, Dübendorf, Switzerland
g. Toxicological Center, University of Antwerp, Wilrijk, Belgium
h. Institute of Chemical Technology, Prague, Czech Republic
i. Ministry of the Environment, Laboratory Services, Toronto, Ontario, Canada
j. Department of Environmental Science "G. Sarfatti", University of Siena, Siena, Italy

3 Methodology

In preparation of the second inter-laboratory study most participants followed an instruction meeting on 15th January 2008 (Amsterdam). This meeting was used to give advice on the analysis of decaBDE on the use of preferred conditions for extraction, clean-up, gas chromatography, control of blanks, and on further analytical improvements (see Annex I). The set-up and protocol (Annex II) of the second round was discussed as well.

Test samples

The inter-laboratory study took place between January and March 2008. An analytical standard solution, sediment, and a dust sample were provided before 21st of January 2008, including a questionnaire on experimental conditions, an instruction protocol and a standard form for reporting of results. The deadline for returning results and additional information was 15th March 2008.

A standard solution containing decaBDE in undisclosed concentration was distributed. This solution was prepared by diluting a certified standard solution of decaBDE in toluene purchased by Wellington Laboratories Inc. (Guelph, Ontario, Canada).

The sediment sample was from natural origin collected in the Elbe (Perlouc) in 2005, and was wet sieved (<63µm), freeze dried, and homogenized and provided by the EU project MODELKEY. The sample was stored at –80ºC. For the interlab study the samples were shipped and stored by the laboratories at room temperature.

The house dust reference material was from NIST (NIST 2585) recently certified for its PBDE content (Stapleton et al. 2006). The certified value of decaBDE is 2510 ± 90 ng/g dw. This reference material is a sterilized, freeze-dried and sieved (< 100 µm) house dust collected from vacuum cleaner bags from homes, motels, and hotels. It contains various polycyclic hydrocarbons, polychlorinated biphenyl congeners, chlorinated pesticides, and polybrominated diphenyl ether congeners. The participants did not know that they received the NIST 2585.

Sample treatment and analysis

The first interlaboratory showed that several methods for extraction and clean-up are appropriate for the determination of decaBDE in dust (Duffek, 2007). Obviously, the choice of the analytical method is less important than the experience of the laboratories and the careful control of critical factors like thermal and photochemical degradation of decaBDE as well as blanks. Analytical solutions to avoid these possible errors are described in the literature (Covaci et al. 2003, de Boer & Wells 2006). In view of all the critical factors in the analysis of decaBDE in environmental samples QA/QC measures are of utmost importance.

Laboratories were allowed to use their own method for the determination of DecaBDE considering the given advice. However, i) the use of $^{13}C_{12}$-labelled decaBDE as internal standard to compensate for the losses throughout the analytical procedure and for inter-injection fluctuations, ii) a short GC column (<15 m), and iii) testing of the GC-MS that no deterioration of decaBDE in the injection system and the column occurs, which all are critical factors, were obligatory.

For the final determination GC/MS operated in either electron ionisation (GC/EI-MS) or electron capture negative ionization (GC/ECNI-MS) mode was used. Four replicate analyses of each sample were requested. Because of known blank problems in decaBDE analysis, participants were asked to determine four independent blank replicates. All participants used isotope dilution technique for quantification. During the analysis of the test material the participants were also requested to record each single step of the whole procedure and any circumstances that might have influenced the results by filling out the provided questionnaire on experimental conditions.

Statistical evaluation

Statistical evaluation of the results submitted was carried out pursuant to the requirements of ISO 5725-2 using the software ProLab (quo data Ltd., Dresden, Germany). Data were checked for outliers according to Grubbs and Cochran.

Additionally, Z-scores were used in the evaluation of the interlaboratory results for each matrix. The QUASIMEME protocol for calculation of the Z-scores was followed. The relative deviation of the result obtained by the laboratory from the assigned value is regarded acceptable if less than 25% by QUASISMEME. For the standard solution a deviation of 10% was taken. The performance of the laboratory is acceptable if the difference between the mean value of decaBDE of the laboratory and the assigned value is equal or less than two. As assigned value for the standard solution 50.17 ± 2.5 ng/ml, and for the dust sample the certified NIST value of 2510 ± 90 ng/g dw was taken. For the sediment sample the mean value of all laboratories, with removal of outliers, was used (15 ± 1.7 ng/g dw). The

QUASIMEME programme recently used a new statistical method to evaluate the data; method of Cofino (Cofino et al., 2000; de Boer and Cofino, 2002). For this method the long-term uncertainties of the analyses of the target compounds are necessary, which are not available for this study.

4 Results and Discussion

Results and detailed method descriptions were received from 10 laboratories, one laboratory was unable to deliver results for the sediment sample, and one laboratory did not report the blank values. The received data are shown in Annex III.

Each participant used their own method, which was according to the instructions given. An overview of the different extraction and clean-up methods is provided in Figure 1. All participants added the internal standard, $^{13}C_{12}$-BDE209, before the extraction. Various extraction methods such as pressurised liquid extraction, Soxhlet, and ultrasonic extraction were applied using different solvents (toluene, hexane:aceton, dichloromethane). The amount of dust extracted varied between 0.1 and 0.3 g, and for the sediment between 1.5 and 7 g. A short GC column, 15 m or less, with an internal diameter of 0.25 mm was used by all laboratories. Four types of non-polar columns were used: Optima, DB-1, DB-5, and DB-XLB. GC injection types ranged from PTV to splittess with or without pulsed pressure, using a programmed injection temperature programme or a fixed injection temperature (<285ºC). Five laboratories used low resolution ECNI-MS, one high resolution ECNI-MS, three high resolution EI-MS, and one low resolution EI-MS.

A summary of the results for each matrix (average concentration and standard deviation) is shown in Fig. 2, 3, and 4 (sequence of laboratories is different from the sequence given in the section on participating laboratories). In general, the participants had no major difficulties with the analysis of decaBDE in the GC-test solution, dust and sediment samples. One laboratory reported difficulties with the analysis of sediment sample probably due to the presence of remaining sulphur in the final extract, and was unable to report concentrations. One laboratory reported relatively high blank values compared to the dust (10% of level) and sediment (36% of level) sample (Annex IV). Normally this laboratory would reanalyse the sample with a higher amount of sediment (50 g instead of 5 g). The other laboratories had decaBDE blank values lower than 1% of the dust level, and between 0.5% and 4% for the sediment sample. The average recovery of decaBDE from the dust sample for all laboratories was 109%, and 95% for the standard solution.

For the dust sample one individual within-laboratory outlier was found (one out of four results deviated significantly), for the sediment sample the mean of one laboratory deviated significantly from the total mean (between-laboratory outlier), and for the standard solution the mean of one laboratory significantly deviated from the total mean. The outliers were removed before statistical evaluation of the data according to ISO 5725. The performance characteristics are shown in table 1.

The repeatability (CV_r) was less than 12% for all samples. The reproducibility variation coefficient (CV_R) was 20% for the dust sample, 19% for the sediment, and 14% for the standard solution. The CV_R is higher than found in the first round intercomparison exercise where the CV was less than 10% both for the dust and standard solution. This is not surprising as in the first round only expert laboratories participated, while in the second round the level of experience with the analysis of decaBDE was very variable. Some of the laboratories recently established methods for the decaBDE analysis while others were more experienced. The results are very good compared to previous interlaboratory studies on decaBDE (e.g. de Boer & Cofino 2002, de Boer et al. 2005, de Boer & Wells 2006). In the International Laboratory Performance Study on the Analysis of Brominated Flame Retardants

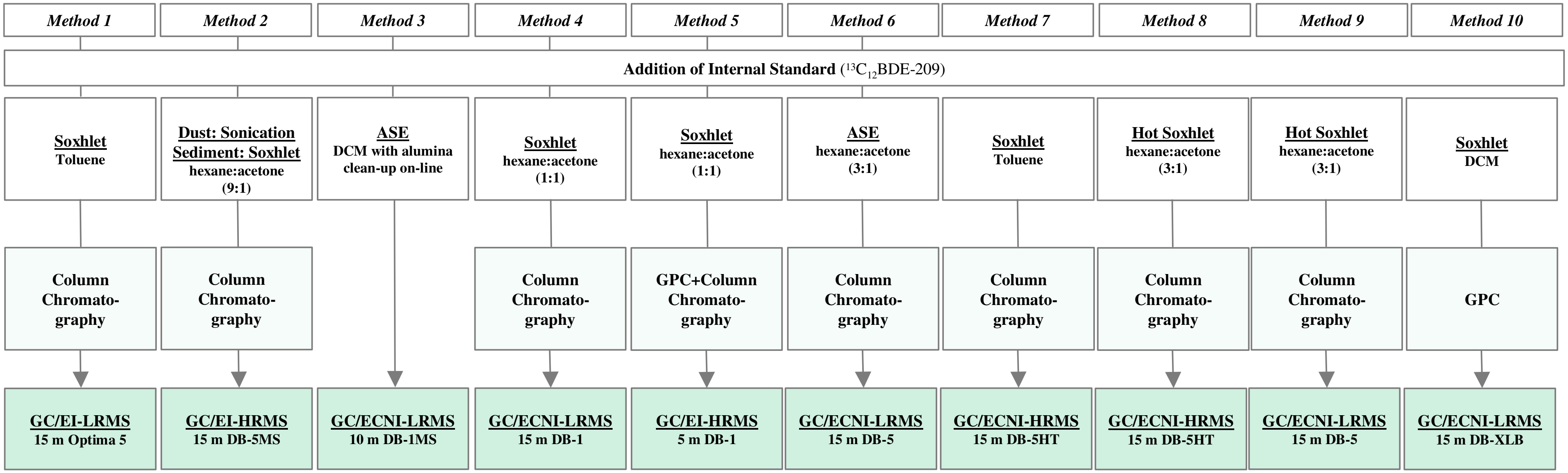

Figure 1: Procedures used for the determination of decaBDE in standard solution, dust (NIST 2585), sediment, and blank reported by ten laboratories.

in Environmental Samples organised by QUASIMEME last year (2007), a CV of 74% was found for decaBDE in marine sediment which was unsatisfactory (QUASIMEME, 2007). The concentration of decaBDE in this marine sediment (6 ng/g) was comparable to the concentration of our sediment (15 ng/g).

For the dust sample some laboratories found higher concentrations than the certified values, which was also observed in the first round exercise (Fig 3). However, the standard deviation of the certified values is rather low (CV 3.6% only); a more appropriate CV would be around 10%. The higher levels are not correlated to the extraction method, extraction solvent or detection technique; an explanation for this phenomenon cannot be given. In general, laboratories with higher levels of decaBDE in dust also found higher levels in sediment, as shown by the two-sample plot (Fig. 5). These higher levels are not correlated to the levels found in the standard solution (Fig 6, e.g. dust vs. standard solution). Differences in results for the standard solution between the laboratories were not correlated to the supplying company of the standards.

Table 1: Performance characteristics for the second NORMAN Inter-laboratory Study "Determination of decaBDE in sediment and dust".

Sample	**Substance**	***l***	***n***	**n_{AP} %**	$\bar{\bar{x}}$	**Lab mean S.D.**	**s_R**	**CV_R %**	**s_r**	**CV_r %**	**Recovery %**
Dust	BDE 209	10	39	2.5	2740	318	536	19.5	212	7.7	109
Sediment	BDE 209	9	32	11.1	15	1.7	2.9	19.4	1.8	11.9	
Solution	BDE 209	10	36	10	48	4.2	6.5	13.6	1.7	3.6	96

l	Number of laboratories
n	Number of single results
n_{AP}	Percentage of outliers
$\bar{\bar{x}}$	Total mean after elimination of outliers in µg/kg for the sediment and dust sample, and ng/ml for the test solution
s_R	Reproducibility standard deviation in µg/kg for the sediment and dust sample, and ng/ml for the test solution
CV_R	Reproducibility variation coefficient [%]
s_r	Repeatability standard deviation in µg/kg for the sediment and dust sample, and ng/ml for the test solution
CV_r	Repeatability variation coefficient [%]

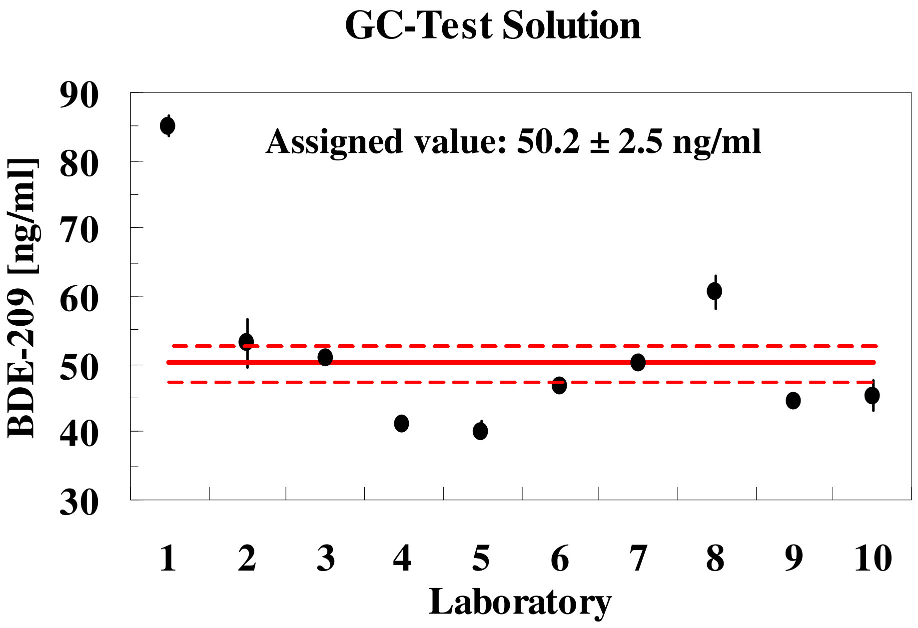

Figure 2: Means of four replicates and standard deviations of decaBDE concentrations in GC-test solution reported by ten laboratories (no elimination of outliers).

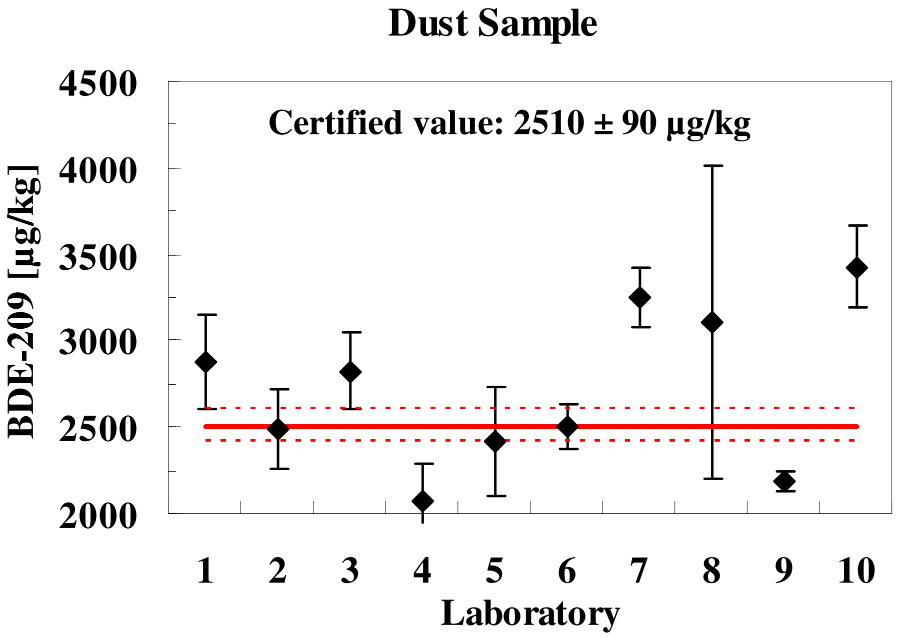

Figure 3: Means of four replicates and standard deviations of decaBDE concentrations in dust (NIST 2585) reported by ten laboratories (no elimination of outliers). Concentrations are corrected for blanks.

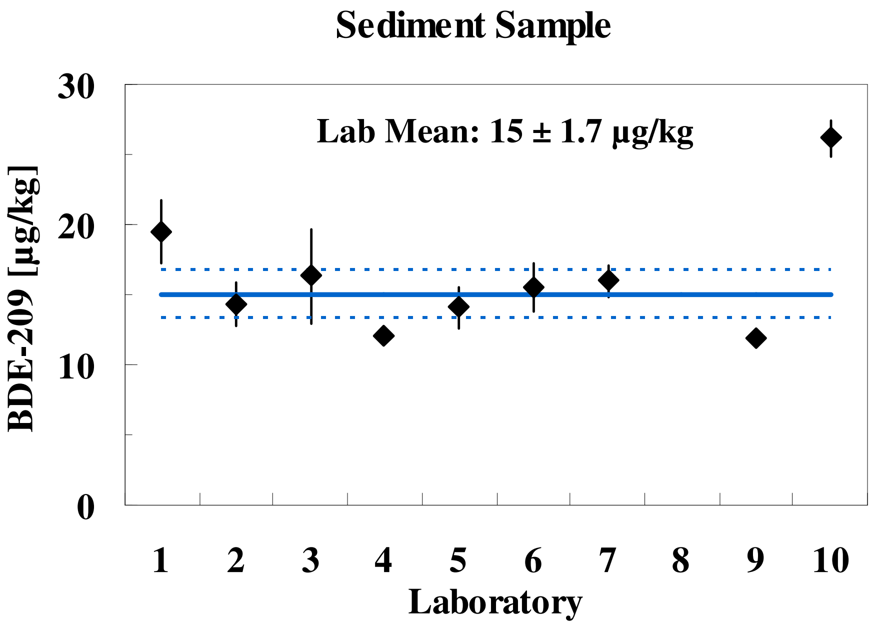

Figure 4: Means of four replicates and standard deviations of decaBDE concentrations in sediment reported by nine laboratories (no elimination of outliers). Concentrations are corrected for blanks.

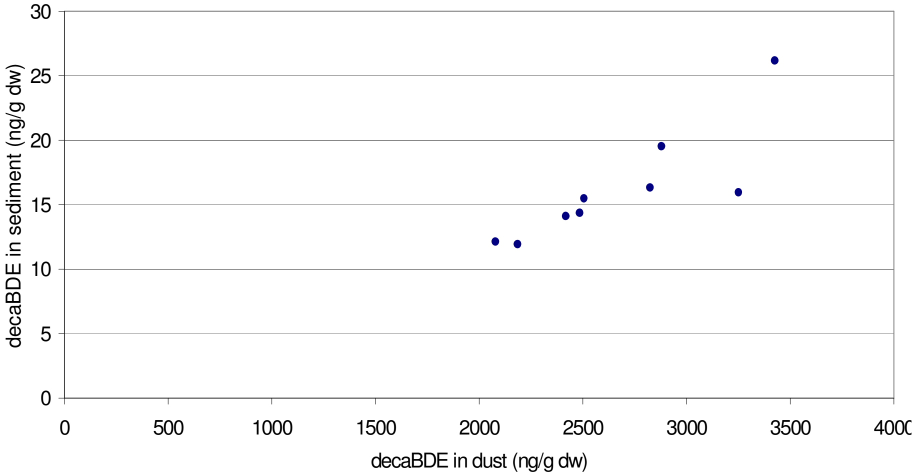

Figure 5: Two sample plot of concentrations of decaBDE in dust and sediment.

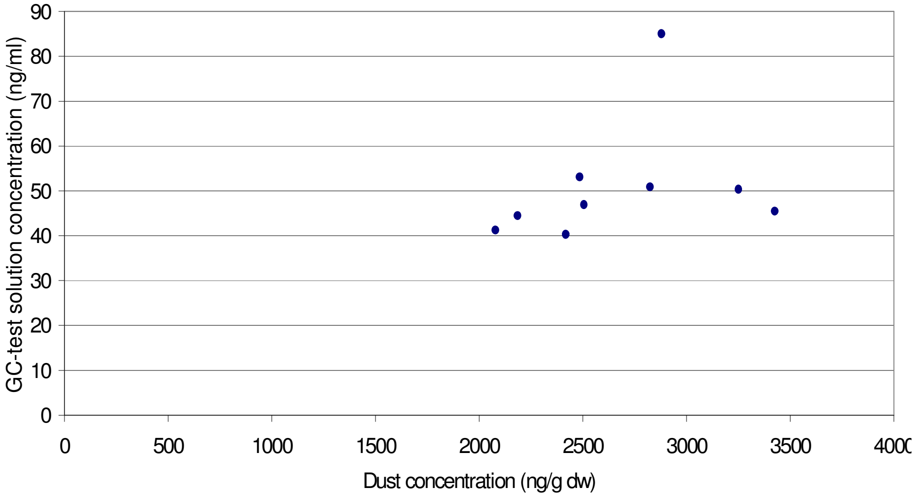

Figure 6: Two sample plot of concentrations of decaBDE in dust and standard solution.

Z-scores were calculated for the GC-test solution, dust and sediment samples (Table 2). The z-score provide information on the relative deviation of the result obtained by the laboratory from the assigned value. A z-score of 2 indicate that the result is 2 standard deviations higher than the assigned value; a negative z-score indicates the result is lower than the assigned value. The QUASIMEME criterion for acceptable results is a z-score from –2 to 2.

All laboratories had good z-scores (between –2 and 2) for both the standard solution and the matrices, except one laboratory for the standard solution and one laboratory for the sediment sample.

Table 2: Z-scores of the average results of the GC-test solution, dust and sediment sample.

Lab no.	GC-test solution	Dust	Sediment
1	7.0	0.6	1.2
2	0.6	-0.04	-0.2
3	0.1	0.5	0.4
4	-1.8	-0.7	-0.8
5	-2.0	-0.1	-0.3
6	-0.7	-0.01	0.1
7	0.0	1.2	0.3
8	2.1	0.9	
9	-1.1	-0.5	-0.8
10	-0.9	1.5	3.0

Conclusions

The method performance study showed that routine laboratories are able to analyse decaBDE in environmental samples with acceptable accuracy when using special attention to QA/QC. The choice of extraction and clean-up method was less critical than the control of critical factors such as thermal and photochemical degradation as well as blanks. The results indicate that knowledge from expert laboratories to routine laboratories can be transferred by means of proper training exercises as essential tool to discuss the critical factors for analysis, and using harmonised protocols by standardizing the critical factors.

5 References

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6 Annex

Annex I: Standard protocol and critical steps and factors for the analysis of decaBDE

Sixth Framework Programme

norman

Round 2 Interlaboratory Study - CASE Study 3 on DecaBDE in Sediment and Dust

Standard Protocol

Determination of BDE-209 in sediment and house dust

Principle

A proper test portion of the dried sediment or dust sample is extracted with an organic solvent by an appropriate extraction technique (e.g. soxhlet extraction, PLE, sonication, shaking). The obtained extract is concentrated and cleaned-up. Sample cleanup procedures may include sulphuric acid treatment, GPC, column chromatography on alumina, florisil or silica. For the sediment sample sulphur should be removed (e.g. active copper powder or TBA reagens). The purified extract is analyzed by high resolution capillary gas chromatography combined with mass spectrometry operated in the electron ionisation or electron capture negative ionisation mode. Quantification is conducted by the internal standard method using $^{13}C_{12}$ labelled Decabromodiphenyl ether as internal standard.

The International Standard ISO/DIS 22032 “Water quality - Determination of selected polybrominated diphenylethers in sediment and sewage sludge - Method using extraction and gas chromatography/mass spectrometry” is recommend as a guidance for the analysis of the test material. Special attention should be paid on following issues:

Problems	Solutions
Photodegradation under influence of direct sunlight	Use of UV filters at laboratory windows and at fluorescent lightings Use of amber glassware (or covered with e.g. aluminium foil)
Poor solubility	Check solubility in the organic solvent before preparing stock solutions or preparing highly concentrated extracts. BDE209 dissolves well in toluene. It should be avoided that the extracts would be evaporated until dryness, because decaBDE may not completely re-dissolve after that step. During concentration, use toluene as a keeper.
Blank problem (decaBDE may be present in dust in the laboratory or as contamination of the glassware)	The laboratory should be kept as clean as possible. Introduction of most types of packing materials in the lab should be avoided. All open glassware should be covered, e.g. by aluminium foil, to prevent dust particles to enter solutions or samples. Cleaning of glassware by heating at 420 °C and rinsing with

	toluene prior use Blank analysis should be carried out more frequently than usually. The treatment of the blanks should be identical to that of the sample (e.g. residence time at the bench). The use of a ^{13}C internal standard is highly recommended, and the sensitivity of the detector should be fully optimized.
Thermal degradation	Short (< 15 m) and narrow (< 0.25 mm) GC columns with thin films (0.1 µm), moderate injector (e.g. 275 °C) and column temperatures (< 300 °C), and short injector residence times, or cold injectors. Splitless injection is critical and can only be applied successfully when combined with pressure pulse or by using short splitless time. On column injection may a suitable alternative.

Annex II: Instruction protocol

Round 2 Interlaboratory Study - CASE Study 3 on DecaBDE in Sediment and Dust

Instruction Protocol

Materials

The **sediment** sample is a sieved (<63 µm), freeze dried and homogenised river sediment (Elbe, Germany). It contains polycyclic hydrocarbons, polychlorinated biphenyl congeners, chlorinated pesticides, and polybrominated diphenyl ether congeners. The bottle contains approximately 30 g of material. The sediment is low contaminated with BDE209 in the µg/kg range.

The **dust** sample is a sterilized, freeze dried and sieved (< 100 µm) house dust from vacuum cleaner bags collected from homes, motels, and hotels. It contains polycyclic hydrocarbons, polychlorinated biphenyl congeners, chlorinated pesticides, and polybrominated diphenyl ether congeners. The bottle contains approximately 4 g of material. The dust is high contaminated with BDE209 in the mg/kg range.

A **GC - test solution** of BDE-209, dissolved in toluene in undeclared concentration is provided to check for calibration errors. Its concentration is in the range of 25 to 100 ng/ml.

Homogeneity, stability and storage

The materials have been shown to be homogeneous and stable for the purpose of the test. The sediment and dust materials must be stored at temperatures between 15 °C to 30 °C away from direct sunlight. The GC-test solution should be stored at 4 °C protected from light.

Analysis

The materials are of naturally occurring river sediment and house dust and may contain constituents of unknown toxicities; therefore, caution and care should be exercised during its handling and use.

Prior to removal of subsamples for analysis, the contents of the bottle should be homogenised. The sediment and dust sample should be dried to a constant mass before weighing for analysis, to determine the concentration on a dry mass basis. Another possibility is the determination of the moisture content.

The samples should be analysed using routinely applied validated methods and procedures. Any appropriate extraction and cleanup method may be used. The use of **^{13}C-BDE-209** as an internal standard is **obligatory**. All measurements should be performed by using GC/MS operated in either electron impact (GC/EI-MS) or electron capture negative ionization (GC/ECNI-MS) mode. Some advice on how recognise and avoid possible sources of error is given in the Standard protocol (see enclosed). A short GC column of 15 m or less is **obligatory** to use for the analysis.

7 Replicates

Please determine **four** independent replicates with the **sediment** using a sample intake of 5 to 7 g of the sediment material.

Please determine **four** independent replicates with the **dust** using a sample intake of 0.1 to 0.5 g of the dust material.

The **GC - Test solution** should be analysed after having added an appropriate amount of the Internal Standard (^{13}C-BDE-209) which matches the expected concentration range (25 - 100 ng/ml). The prepared solution should be injected **four** times.

Because of the blank problem please determine **four** independent **blank** replicates. The treatment of the blanks should be identical to that of the sediment and dust sample (e.g. residence time at the bench).

8 Reporting of results

For the sediment and dust sample the results should be expressed on a dry weight basis (µg/kg). Results for the GC test solution should be given in ng/ml.

Please enter your analytical data and method characteristics using the provided files (provided by email). You should report all of the requested methods details by filling in the questionnaire.

The **results** of the sediment and dust and the GC – Test solution are to report by using Excel-file “Results of dust, sediment and GC test solution.xls” and Word-document “Experimental conditions.doc”. Please send the completed files to Pim Leonards by email (pim.leonards@ivm.vu.nl) until **15 March 2008.**

Please also send a typical **GC–chromatogram** of the sediment, dust sample and the GC-test solution with the drawn integration marks (either by email or in a printed version). Report results of blanks separately and do not include blanks in calculations!

Annex III: Submitted data of decaBDE in GC-test solution, dust, and sediment samples of the NORMAN interlab round 2 provided by the laboratories. Shown are the individual levels of four replicates. AVG: average concentration. SD: standard deviation. CV: coefficient of variation.

GC - Test Solution ng/ml

Lab no.	#1	#2	#3	#4	AVG	SD	CV (%)
1	84.9	82.9	86.6	85.7	85	1.6	1.9
2	57.63	52.46	52.81	49.42	53	3.4	6.4
3	50.45	51.86	50.70	50.55	51	0.7	1.3
4	41.61	41.18	41.15	41.06	41	0.2	0.6
5	42.0	39.6	39.4	40.2	40	1.2	2.9
6	46.04	47.19	47.79	46.64	47	0.7	1.6
7	50.43	51.61	49.18	50.15	50	1.0	2.0
8	59.33	61.15	58.31	63.89	61	2.4	4.0
9	44	45	45	44	45	0.6	1.3
10	47.39	44.81	42.52	47.22	45	2.3	5.1

Dust (with blank correction), ug/kg

Lab no.	#1	#2	#3	#4	AVG	SD	RSD (%)
1	2796	3009	2539	3173	2879	274	10
2	2656	2527	2154	2603	2485	227	9
3	2865	3082	2811	2537	2824	224	8
4	1852	2366	2050	2048	2079	213	10
5	2060	2347	2811	2453	2418	310	13
6	2434	2368	2666	2553	2505	132	5
7	3309	3443	3019	3232	3251	178	5
8	3681	1757	3503	3476	3104	903	29
9	2177	2115	2255	2195	2186	58	3
10	3138	3590	3329	3645	3426	236	7

Sediment (with blank correction), ug/kg

Lab no.	#1	#2	#3	#4	AVG	SD	RSD (%)
1	19.5	17.2	22.6	18.8	20	2.3	12
2	13.68	13.82	16.68	13.21	14	1.6	11
3	13.02	20.00	18.25	14.01	16	3.3	20
4	11.70	11.87	12.54	12.39	12	0.4	3
5	15.4	12	14.7	13.8	14	1.5	11
6	14.0	17.5	14.0	16.4	15	1.7	11
7	14.47	17.01	16.32	16.01	16	1.1	7
8							
9	12.2	12.1	11.4	12.0	12	0.4	3
10	27.26	26.01	24.43	26.95	26	1.3	5

Outlier

Type A: individual within-laboratory outlier	🟨
Type B: between-laboratory outlier due to significant deviation of the laboratory mean from the total mean	🟧

Annex IV: Data of blanks of decaBDE analysis. Shown is the average percentage contribution (AVG) of the blank value to the dust and sediment decaBDE levels based on four replicate analyses. AVG: average percentage. SD: standard deviation. CV: coefficient of variation.

Percentage blank value of decaBDE compared to dust sample.

Lab no.	AVG (%)	SD	CV (%)
1	10.3	0.9	9
2	0.1	0.0	14
3	0.02	0.0	8
4	0.3	0.1	18
5	0.3	0.1	57
6	0.07	0.0	11
7	0.008	0.0	6
8	0.6	0.7	124
9	0.4	0.2	41
10	not reported		

Percentage blank value of decaBDE compared to sediment sample.

Lab no.	AVG (%)	SD	CV (%)
1	36.2	13.8	38
2	1.2	0.1	10
3	3.3	0.6	20
4	2.3	0.4	16
5	0.9	0.4	41
6	0.5	0.1	12
7	1.6	0.1	7
8			
9	4.0	0.3	7
10	not reported		