BAM-U019: Polychlorinated Biphenyls in Soil

Certification Report

ausverkauft / out of stock

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Contents

List of Abbreviations

BIPM	International Bureau of Weights and Measures
CCQM	Consultative Committee for the Amount of Substance – Metrology in Chemistry
CIPM	International Committee for Weights and Measures
CIPM MRA	Mutual Recognition Arrangement of CIPM members

ECD	Electron capture detector
FBE	Fluidised bed extraction
GC	Gas chromatography
PCB	Polychlorinated biphenyls
MS	Mass spectrometry
PFE	Pressurised fluid extraction
PTFE	Polytetrafluoroethylene

1 Introduction

1.1 PCB congeners of interest and analytical procedures

Polychlorinated Biphenyls (PCB) belong to the persistent organic pollutants to be monitored by field laboratories on contaminated sites. The quantification of PCBs in soil using gas chromatography with electron capture detection (GC-ECD) has been standardised at first [1] and the alternative application of GC-MS for this purpose is widely used by routine laboratories and has been standardised meanwhile [2].

BAM-U019 is certified for the extractable mass fractions of the congeners PCB-28, PCB-53, PCB-101, PCB-118, PCB-138, PCB-153 and PCB-180. Among the 209 theoretically possible congeners, these are the most often analysed ones and they are listed in respective standards such as [1, 2]. Besides, the material is certified for the extractable mass fractions of PCB-44, PCB-149 and PCB-170 which are only listed in [3].

1.2 Strategy of the certification project

A real world material taken from a contaminated site was preliminarily checked for its appropriateness regarding the sum of contents of the target compounds PCB-28, PCB-53, PCB-101, PCB-138, PCB-153 and PCB-180 being lower than 10 mg/kg as stated in [4]. This certification exercise was conducted at BAM. Nine external field laboratories which were chosen on basis of their proficiency in PCB analyses proven in the framework of 15th regular PT round “Contaminated sites” of BAM took part in this exercise. Besides, two different labs of BAM were among the participants. The calibration and measurement capability (CMC) of BAM with regard to PCB analyses has internationally been recognised by way of key comparisons [5] of the Consultative Committee for Amount of Substance – Metrology in Chemistry (CCQM) of the International Bureau of Weights and Measures (BIPM).

Both analytical methods, GC-MS and GC-ECD, were used for quantification of PCB contents and a variety of different extraction procedures was applied as well. Traceability was established using calibration standard solutions certified for the contents of these congeners and in case of GC-MS partly ^{13}C-labelled PCBs were applied as internal standards (see clause 5.3.3).

2 Candidate material

The sandy soil was sampled from a contaminated site within a garden colony in the surroundings of Berlin, Germany. The specific location displayed an aged contamination originating from unknown sources. After drying to constant weight, the bulk material was classified by means of an automatic sieving station and a total amount of 34 kg of the fraction < 125 µm was collected. Thereafter, this material was homogenised by means of a 120 L stainless steel barrel in a drum hoop mixer (J. Engelsmann AG, Ludwigshafen; Germany). The barrel was equipped with a mixing insert inside to improve the mixing intensity.

After final homogenisation by means of a spinning riffler of Retsch GmbH, Haan, Germany, applying the so called “cross riffling” procedure [6] a total of 320 units were bottled in 100 mL amber screw-capped glass bottles containing (105.9 ± 1.0) g each and units were numbered in the order of leaving the bottling process. The screw caps equipped with PTFE inserts were tightly closed and sealed with shrinking foil. All units were stored at -20 °C directly after bottling. Table 1 comprises the matrix characterisation of the candidate material.

Table 1: Matrix characterisation of BAM-U019

Parameter	Value	Method
Particle size range (µm)	< 125	Sieving
Water content (%)	1.22 ± 0.07	*Karl Fischer* titration
Drying loss (%)	1.26 ± 0.10	Gravimetry after drying to constant mass at 105 °C (ISO 11465:1993)
CHN-Analysis (%)	C: 2.25 ± 0.12; H: 0.34 ± 0.02; N: 0.17 ± 0.01	Combustion

3 Homogeneity study

The procedure employed for this study was pressurized fluid extraction (PFE) with acetone/n-hexane (1:1) followed by GC-MS with PCB 209 as internal standard. Sixteen bottles were selected equidistantly from the whole batch of 320 units in the order of bottling. All 16 units were extracted once respectively under repeatability conditions on three consecutive days. The sample intake of 5 g for one determination was chosen on basis of earlier experience with similar materials used in proficiency testing and the recommendations of the standard procedure in [1]. All extracts were analysed under repeatability conditions after randomisation. ANNEX A.1 contains the individual measurement results and table 2 reveals the synopsis of the 1-way analysis of variance (ANOVA).

Table 2: Results of the 1-way ANOVA of the homogeneity study

Congener	MS_{within} [b] ($mg^2\ kg^{-2}$)	$MS_{between}$ [a] ($mg^2\ kg^{-2}$)	F_{obs} [c]	F_{crit} [d]	u_{bb} [e] ($mg\ kg^{-1}$)
PCB 28	0.002	0.005	2.006	1.99	0.028
PCB 44	0.017	0.035	2.050	1.99	0.077
PCB 52	0.039	0.090	2.319	1.99	0.131
PCB 101	0.039	0.075	1.923	1.99	0.109
PCB 118	0.027	0.062	2.336	1.99	0.109
PCB 138	0.011	0.021	1.903	1.99	0.057
PCB 149	0.006	0.011	1.845	1.99	0.041
PCB 153	0.008	0.019	2.293	1.99	0.059
PCB 170	5.73 e-5	8.67 e-5	1.512	1.99	0.010
PCB 180	9.34 e-4	2.61 e-3	2.789	1.99	0.024

[a] *Mean of squared deviations within bottles (from 1-way ANOVA)*
[b] *Mean of squared deviations between bottles (from 1-way ANOVA)*
[c] *Observed F-value:* $MS_{between}/MS_{within}$
[d] *Critical F-value at the 95 % level of confidence*
[e] *Standard uncertainty between the bottles: Estimate of inhomogeneity contribution to the total uncertainty of the respective property value according to [7].*

The estimates of inhomogeneity contributions u_{bb} potentially hidden by the measurement uncertainty and to be included in the total uncertainty were estimated according to ISO Guide 35 [7] as the maximum of the values obtained from Eq. (1) and (2).

$$u_{bb} = \sqrt{\frac{MS_{among} - MS_{within}}{n}} \quad (1) \qquad u_{bb} = \sqrt{\frac{s_{method}}{n}} \sqrt[4]{\frac{2}{N(n-1)}} \quad (2)$$

where:

s_{method} = Method variability $(= \sqrt{MS_{within}}\ )$
n = Number of replicate determinations per sample
N = Number of bottles analysed

4 Stability study

4.1 Initial stability study

From earlier experience with soil based CRM containing organic analytes a temperature-driven deterioration of the PCB content was to be expected also in this case. Selected units of the candidate material were submitted to a so-called isochronous [8] accelerated ageing at temperatures between +4 and+ 60 °C over periods of 1-12 months as shown in Table 3. After respective periods of ageing time, individual units were stored at (–20 ± 5) °C. All units were analysed for PCB contents using the procedure under repeatability conditions together with reference samples which had been kept at (–20 ± 5) °C since bottling. For the individual data see ANNEX A.2.

Table 3: Accelerated ageing of exposed units, exposition temperatures and periods

Ageing time [Months]	+ 4 °C	+ 22 °C	+ 40 °C	+ 60 °C	Remark
1	x	x	x	x	*initial study*
3	x	x	x	x	*initial study*
6	x	x	x	x	*initial study*
12	x	x	x		*initial study*
24	x	x			*post certification monitoring*
36	x	x			*post certification monitoring*

Data evaluation and expiry date estimation strictly follow the procedures as comprehensively described in [9]: From semi-logarithmic plots of measured single values over time, effective deterioration rates were determined and tested against an Arrhenius model describing the dependence on temperature of the deterioration rates. All PCBs congeners matched the model sufficiently well in order to derive worst case estimates for the shelf lives of the material. Table 4 contains activation energies and the contribution for the long term stability u_{lts} that was included in the uncertainty budget of the certified values (see 5.3.3) to provide the validity of the certified values estimated from the model for at least three years.

Table 4: Long term stability contribution u_{lts} to the uncertainty of the certified values and activation energies derived from the Arrhenius model

PCB congener	u_{lts} (%)	ΔE_a (kJ mol^{-1})
PCB 28	10.59	21.4
PCB 44	4.34	21.1
PCB 52	8.86	21.0
PCB 101	10.12	19.0
PCB 118	13.64	18.7
PCB 138	12.40	17.8
PCB 149	6.71	18.3
PCB 153	11.57	17.7
PCB 170	9.13	14.8
PCB 180	6.56	15.3

Figure 1 shows the dependence of the logarithm of the effective deterioration rate k_eff on the inverse temperature by way of example for PCB 44 and PCB 153.

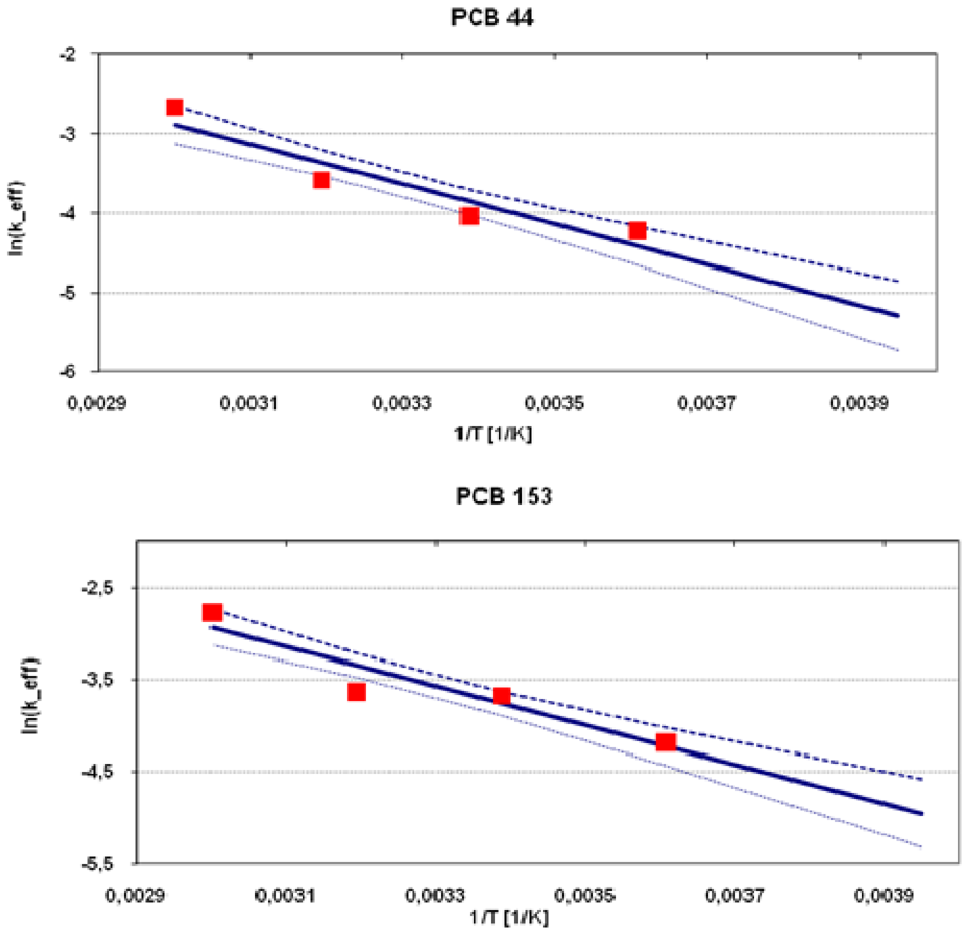

Fig. 1: Effective deterioration rate versus inverse temperature for PCB 44 and PCB 153.

The expiry term estimates as summarised in table 4 were calculated using the value at the upper confidence bound of the regressed line as a worst-case estimate for the rate of deterioration to be expected.

The shelf lives at a storage temperature of (-20 ± 5) °C are relatively short for the PCB congeners which were expected to be relatively stable. Any exposure to room or even higher temperatures may further reduce the time of validity of certified PCB contents of BAM-U019. Therefore, the expiry date of **one year after dispatch from BAM** is established. Transportation/delivery time should be kept at the possible minimum and any exposure to heat or strong light should be avoided.

4.2 Post-certification stability monitoring

The first rough estimation of stability will be updated by further measurements of units stored at +4 °C and +20 °C over the period of availability of the material. The post-certification measurements will be conducted according to the information given in table 3.

5 Characterisation study

5.1 Selection of participants and methods

Nine external field laboratories which were chosen on basis of their proficiency in PCB analyses demonstrated within the framework of 15th regular PT round “Contaminated sites” of BAM took part in this exercise. Besides, two different labs of BAM were among the participants since the calibration and measurement capability (CMC) of BAM with regard to PCB analyses has internationally been recognised by way of key comparisons [5] of the Consultative Committee for Amount of Substance – Metrology in Chemistry (CCQM) of the International Bureau of Weights and Measures (BIPM). The methods employed covered both methods GC-MS and GC-ECD as well as modern and classic extraction procedures. The equivalence of Soxhlet extraction, pressurised fluid extraction (PFE), sonication, shaking, fluidised bed extraction, and Twisselmann extraction were seen to yield equivalent recoveries of halogenated organics including PCB in numerous investigations.

The following laboratories participated in the certification study and are sorted alphabetically. This order is different from that used in tables 5 and 6 below.

BAM, working group 1.21	12489	Berlin
BAM, working group 1.22	12489	Berlin
BIOLAB Umweltanalysen GmbH	38112	Braunschweig
Chemisches Labor Dr. Wirts + Partner	30559	Hannover
EUROFINS-AUA GmbH	09633	Halsbrücke
Gesellschaft für Analytik und Umweltberatung mbH	64625	Bensheim
Labor Dr. Döring	28357	Bremen
PORST & PARTNER	90762	Fürth
UABG GmbH	10365	Berlin
UIS Umweltinstitut	70469	Stuttgart
WESSLING Laboratorien GmbH	30625	Hannover

5.2 Design of the study

Two units of the candidate material were to be analysed by each laboratory in triple each. Each laboratory was to prepare its independent calibration from appropriate PCB mix solutions available from commercial suppliers with certified PCB contents. Traceability of these contents given by manufacturers like Ehrenstorfer and LGC Promochem to the SI units was shown at BAM before by comparison of these standards with a certified solution PCB solution provided by IRMM (BCR 365).

Most of the laboratories using GC-MS applied some ^{13}C-labelled PCBs as internal standards for recovery. Other labelled species (e. g. PAHs) were used as well when universal analytical procedures for both PCBs and PAHs were applied by some of the field laboratories using GC-MS as well. Laboratories using GC-ECD applied PCB 209 or Aldrin for this purpose.

Extraction methods and solvents to be used by each laboratory are listed in Table 5. Details are given in the Annex (A.3). Results for the contents of PCBs were to be reported on basis of total mass intake, no dry mass determinations were asked for.

Table 5: Extraction and determination methods

Laboratory	Analytical method	Extraction method	Extraction solvent
C01	GC-ECD	pressurised fluid extraction	n-hexane
C02	GC-IDMS	pressurised fluid extraction	n-hexane; acetone
C03	GC-IDMS	shaking	n-hexane; acetone
C04	GC-MS	Soxhlet	n-hexane
C05	GC-IDMS	Twisselmann	petroleum ether; acetone
C06	GC-MS	shaking	n-heptane; acetone
C07	GC-ECD	sonication	iso-hexane; acetone
C08	GC-MS	shaking	petroleum ether; acetone
C09	GC-ECD	Soxhlet	n-hexane
C10	GC-IDMS	pressurised fluid extraction	n-hexane; acetone
C11	GC-ECD	pressurised fluid extraction	cyclohexane

5.3 Evaluation of results and certified values

The results of the certification study are listed comprehensively in table 6 and were evaluated in accordance with ISO Guide 35 [7]. For all measurement data see ANNEX A.3. The computer software SoftCRM [10] was partially used for statistical tests and data treatment.

5.3.1 Statistical evaluation

Though the participants in the intercomparison were carefully selected on basis of proven proficiency, a certain scatter of results was to be expected from experience. Thus, there was no good reason for assuming that the single values measured by the different laboratories would belong to a common mother distribution. This was confirmed by the statistical analysis within which the following statistical parameters were calculated and are summarised in table 7:

- *the mean of laboratory means*
- *the standard deviation of the distribution of laboratory means, and the standard deviation of the mean of laboratory means*
- *the confidence interval of the mean of laboratory means at the 0.05 significance level*

and the following statistical tests were carried out (at significance levels of 0.05 and 0.01):

- *Cochran test for the identification of outliers with respect to laboratory variance*
- *Grubbs test for the identification of outliers with respect to the mean*
- *Dixon and Nalimov test for the verification of possible outlier indications*
- *Kolmogorov-Smirnov Test (Lilliefors version) for the normality test*
- *Test for skewness and kurtosis*

The results of the above calculations and tests for a data evaluation based upon the laboratory means are given in table 7 ct. The main features are as follows:

- *Scheffé-Test: Data sets differ significantly.*
- *Bartlett-Test: Variances are mostly inhomogeneous (at the significance level of 0.01).*
- *Cochran-Test: Several outliers were detected (significance level 0.05 and 0.01).*
- *Dixon-, Grubbs- und Nalimov-Test: Several outliers were detected (significance level 0.01).*

Table 6: Data sets received from the participating laboratories (mean ± standard deviation)

	C01	C02	C03	C04	C05	C06	C07	C08	C09	C10	C11
	(mg kg^{-1})	(mg kg^{-1})	(mg kg^{-1})	(mg kg^{-1})	(mg kg^{-1})	(mg kg^{-1})	(mg kg^{-1})	(mg kg^{-1})	(mg kg^{-1})	(mg kg^{-1})	(mg kg^{-1})
PCB 28	0.452 ± 0.117	0.503 ± 0.040	0.466 ± 0.046	0.667 ± 0.015	0.462 ± 0.026	0.568 ± 0.019	0.408 ± 0.004	0.519 ± 0.020	0.524 ± 0.012	0.524 ± 0.040	0.535 ± 0.045
PCB 44		2.31 ± 0.29	1.85 ± 0.10	2.27 ± 0.06	1.89 ± 0.04			2.10 ± 0.09		1.73 ± 0.13	2.08 ± 0.17
PCB 52	2.20 ± 0.55	3.32 ± 0.23	3.08 ± 0.13	3.19 ± 0.08	2.98 ± 0.07	3.04 ± 0.12	2.63 ± 0.04	3.37 ± 0.28	2.80 ± 0.14	2.72 ± 0.22	3.19 ± 0.27
PCB 101	2.21 ± 0.59	3.04 ± 0.27	3.22 ± 0.11	3.52 ± 0.09	2.47 ± 0.05	2.86 ± 0.05	3.04 ± 0.04	3.66 ± 0.17	2.51 ± 0.08	2.60 ± 0.19	2.67 ± 0.22
PCB 118	1.96 ± 0.52	2.70 ± 0.20	2.51 ± 0.09	2.87 ± 0.07	2.46 ± 0.09	2.50 ± 0.06	2.73 ± 0.06	2.51 ± 0.23	2.67 ± 0.12	2.29 ± 0.15	2.59 ± 0.22
PCB 138	1.28 ± 0.31	1.79 ± 0.22	2.08 ± 0.07	2.02 ± 0.05	1.76 ± 0.04	2.06 ± 0.06	1.63 ± 0.03	2.31 ± 0.05	1.82 ± 0.05	1.49 ± 0.11	1.64 ± 0.13
PCB 149		1.39 ± 0.14	1.12 ± 0.05	1.22 ± 0.01	1.06 ± 0.02		1.20 ± 0.01	1.15 ± 0.03		1.01 ± 0.08	1.41 ± 0.11
PCB 153	0.93 ± 0.22	1.80 ± 0.13	1.32 ± 0.04	1.46 ± 0.03	1.35 ± 0.03	1.52 ± 0.06	1.30 ± 0.02	1.50 ± 0.03	1.35 ± 0.07	1.31 ± 0.11	1.37 ± 0.10
PCB 170		0.275 ± 0.025	0.272 ± 0.010	0.351 ± 0.012	0.267 ± 0.005			0.267 ± 0.018		0.207 ± 0.013	0.213 ± 0.017
PCB 180	0.289 ± 0.035	0.413 ± 0.042	0.363 ± 0.010	0.447 ± 0.011	0.375 ± 0.008	0.389 ± 0.018	0.401 ± 0.024	0.403 ± 0.020	0.380 ± 0.012	0.398 ± 0.041	0.363 ± 0.025

The means of laboratory means after elimination of the outliers were taken as the best estimates w_{char} for the values to be certified, and the standard deviations of the mean of laboratory means were taken as the uncertainty contributions u_{char} from characterisation by this intercomparison exercise.

Table 7: Values and statistical parameters from the intercomparison

Compound	w_{char}[a] (mg/kg)	s[b] (mg/kg)	u_{char}[c] (mg/kg)	CI[d] (mg/kg)	Pooling	Data sets
PCB 28	0.496	0.048	0.015	0.034	no	10
PCB 44	2.035	0.218	0.082	0.202	no	7
PCB 52	2.956	0.344	0.104	0.231	no	11
PCB 101	2.891	0.454	0.137	0.305	no	11
PCB 118	2.583	0.164	0.052	0.118	no	10
PCB 138	1.807	0.296	0.089	0.199	no	11
PCB 149	1.196	0.143	0.051	0.120	no	8
PCB 153	1.381	0.209	0.063	0.141	no	11
PCB 170	0.2645	0.0475	0.0180	0.0439	no	7
PCB 180	0.393	0.026	0.008	0.018	no	10

[a] *mean of laboratory means*
[b] *standard deviation of the population of laboratory means*
[c] *standard uncertainty of w_{char}*
[d] *confidence interval*

Table 7 continued

Compound	Scheffè[f]	Bartlett[g] 0.01	Cochran[h] 0.01	Grubbs[h] 0.01(0.05)	Dixon[h] 0.01(0.05)	Nalimov[h] 0.01(0.05)	Gauss[i] 0.05	Deleted laboratories
PCB 28	no	Inhom	C01	-(C04)	-(C04)	C4(C04)	yes	C04
PCB 44	no	Inhom	C01	-(-)	-(-)	-(C01)	yes	
PCB 52	no	Inhom	C01	-(-)	-(-)	-(-)	yes	
PCB 101	no	Inhom	C01	-(-)	-(-)	-(-)	yes	
PCB 118	no	Hom	C01	-(C01)	-(C01)	C01(C01)	yes	C01
PCB 138	no	Inhom	C01,C02	-(-)	-(-)	-(-)	yes	
PCB 149	no	Inhom	C02,C011	-(-)	-(-)	-(-)	yes	
PCB 153	no	Inhom	C01,C011	-(-)	-(C01,C02)	-(C02)	no	
PCB 170	no	Hom	-	-(-)	-(-)	-(C04)	yes	
PCB 180	no	Inhom	C01	-(C01)	-(C01)	-(C05)	yes	C01

[f] *Scheffé test for two-by-two data set compatibility*
[g] *Bartlett test for homogeneity of laboratory variances (significance level 0.01)*
[h] *Outlier tests: outlying laboratory are indicated, significance level 0.01 (0.05 in brackets)*
[I] *Normal at a significance level of 0.05*

5.3.2 Traceability

In order to ensure traceability of the extractable contents of the investigated PCB, commercially available solutions of the respective PCBs were used by the participants as calibration standards. In an earlier work it could be shown at BAM, by means of an intercalibration experiment, that the commercial standard PCB solutions are consistent with the certified reference material BCR-365.

5.3.3 Certified values and combined uncertainties

As mentioned in clause 5.3.1, the means of laboratory means were taken as the best estimates w_{char} for the values to be certified. The standard uncertainties of the mean of laboratory means were taken as the uncertainty contributions u_{char} from characterisation.
Besides this uncertainty of characterisation u_{char}, a possibly undetected inhomogeneity u_{bb}, and the estimated deterioration of the PCB content over time u_{lts} contribute to the combined uncertainty according to

$$u_{c,r}^2 = u_{char,r}^2 + u_{bb,r}^2 + u_{lts,r}^2$$

where the index r refers to the corresponding relative uncertainties. The uncertainties of purity of the calibration standards were negligible in comparison with the three major contributions.

The final values are given in table 8 where the expansion factor for the expanded uncertainties U is $k = 2$. The value and the expanded uncertainty are rounded according to the recommendations of the GUM [11] and are given with respect to raw sample mass in table 9. The water content of BAM-U019 was seen as remaining stable if the material is handled according to the instructions given in the certificate (see also clause 6).

Table 8: Certified mass fractions of PCBs in BAM-U019 (before rounding)

Compound	w_{char} **(mg kg^{-1})**	u_{char} **(mg kg^{-1})**	u_{bb} **(mg kg^{-1})**	u_{lts} **(mg kg^{-1})**	u_c **(mg kg^{-1})**	U (k = 2) **(mg kg^{-1})**
PCB 28	0.496	0.02817	0.015179	0.052535	0.0615133	0.1230
PCB 44	2.035	0.07686	0.082396	0.088403	0.1432190	0.2864
PCB 52	2.956	0.13071	0.103720	0.261778	0.3104362	0.6209
PCB 101	2.891	0.10923	0.136886	0.292686	0.3410777	0.6822
PCB 118	2.583	0.10873	0.051861	0.352419	0.3724392	0.7449
PCB 138	1.807	0.05698	0.089247	0.224023	0.2477868	0.4956
PCB 149	1.196	0.04122	0.050558	0.080292	0.1034504	0.2069
PCB 153	1.381	0.05899	0.063016	0.159843	0.1816603	0.3633
PCB 170	0.2645	0.0098922	0.017953	0.024157	0.03168156	0.0634
PCB 180	0.393	0.0236	0.00822192	0.0257699	0.03589774	0.07180

Table 9: Certified mass fractions of PCBs in BAM-U019 (after rounding)

Compound	**Mass fraction in (mg kg^{-1})**	U **(mg kg^{-1})**
PCB 28[3)]	0.50	0.13
PCB 44	2.04	0.29
PCB 52[3)]	2.96	0.62
PCB 101[3)]	2.90	0.68
PCB 118	2.58	0.75
PCB 138[3)]	1.81	0.50
PCB 149	1.20	0.21
PCB 153[3)]	1.38	0.37
PCB 170	0.264	0.063
PCB 180[3)]	0.39	0.08

[3)] PCB target congeners which are relevant for risk assessment according to Council Directive 96/59/EC [4]

6 Information on the proper use of BAM-U019

6.1 Shelf life

From the initial stability study a preliminary shelf life of three years at storage temperatures not higher than -20 °C is estimated. Since the dispatch to the end user may occur at any time during this period the certified properties will be valid for 12 months beginning with the dispatch of the material from BAM. The validity of this information will be maintained by post-certification stability monitoring.

6.2 Transport, storage and use

The stability of the content of PCBs allows the dispatch of the material at ambient temperature. On receiving, it is to be stored at (–20 ± 5) °C. Before withdrawing a sub-sample the bottle has to have reached ambient temperature. Thereafter, the bottle must be closed tightly and stored at (–20 ± 5) °C. The water content remains stable when the material is treated as described.

6.3 Safety instructions

The soil material was not sterilised, however, it is supposed to not exhibit any biological activity due to having been dried to constant weight. No hazardous effect is to be expected when the material is used under conditions usually adopted for the analysis of environmental matrices moderately contaminated with halocarbons like PCBs.

It is strongly recommended to handle and dispose of the reference material in accordance with the guidelines for hazardous materials legally in force at the site of end use and disposal.

6.4 Legal notice

Neither the BAM Federal Institute for Materials Research and Testing nor any person acting on their behalf make any warranty or representation, express or implied, that the use of any information, material, apparatus, method or process disclosed in this document may not infringe privately owned rights, or assume any liability with respect to the use of, or damages resulting from the use of any information, material, apparatus, method or process disclosed in this document.

7 References

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[3] EN 12766-1:2000: Petroleum products and used oils - Determination of PCBs and related products - Part 1: Separation and determination of selected PCB congeners by gas chromatography (GC) using an electron capture detector (ECD)

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ANNEX: Measurement results

A.1 Homogeneity study

The measurements for the homogeneity and stability study were both performed under repeatability conditions with a randomised order of extracts and using GC-MS after PFE. It should be noted that in case of testing the homogeneity the accuracy of the determined values is of minor importance as long as the precision of the measurements is sufficient. Therefore, the calibration was chosen in a way to allow a rapid and robust performance of the extended measurement series. As a result, the determined PCB contents differ from those values obtained during the certification measurements.

PCB 28				PCB 44			
Bottle	Replicate			Bottle	Replicate		
	1 (mg kg^{-1})	2 (mg kg^{-1})	3 (mg kg^{-1})		1 (mg kg^{-1})	2 (mg kg^{-1})	3 (mg kg^{-1})
4	0.588	0.484	0.456	4	1.921	1.681	1.598
24	0.496	0.493	0.461	24	1.602	1.582	1.608
44	0.541	0.544	0.588	44	1.844	1.763	1.823
64	0.621	0.519	0.569	64	2.043	1.735	1.820
84	0.509	0.504	0.514	84	1.748	1.654	1.692
104	0.510	0.483	0.464	104	1.692	1.593	1.585
124	0.476	0.435	0.497	124	1.568	1.540	1.730
144	0.385	0.440	0.516	144	1.378	1.425	1.742
164	0.396	0.352	0.536	164	1.405	1.278	1.703
184	0.455	0.400	0.574	184	1.545	1.387	1.815
204	0.515	0.522	0.512	204	1.689	1.652	1.686
224	0.513	0.498	0.518	224	1.838	1.528	1.652
244	0.454	0.471	0.481	244	1.539	1.692	1.705
264	0.462	0.461	0.421	264	1.600	1.584	1.527
284	0.374	0.443	0.513	284	1.366	1.478	1.657
304	0.448	0.497	0.466	304	1.542	1.711	1.574

Homogeneity study

PCB 52			
Bottle	Replicate		
	1 (mg kg^{-1})	2 (mg kg^{-1})	3 (mg kg^{-1})
4	3.044	2.665	2.473
24	2.519	2.474	2.445
44	2.892	2.723	2.831
64	3.180	2.643	2.875
84	2.727	2.556	2.592
104	2.650	2.500	2.494
124	2.461	2.415	2.649
144	2.155	2.252	2.656
164	2.152	1.962	2.609
184	2.414	2.200	2.867
204	2.659	2.615	2.583
224	2.838	2.498	2.606
244	2.377	2.600	2.663
264	2.498	2.476	2.362
284	2.126	2.286	2.560
304	2.426	2.679	2.440

PCB 101			
Bottle	Replicate		
	1 (mg kg^{-1})	2 (mg kg^{-1})	3 (mg kg^{-1})
4	2.838	2.574	2.415
24	2.381	2.401	2.409
44	2.755	2.647	2.669
64	3.041	2.607	2.748
84	2.676	2.534	2.532
104	2.600	2.407	2.337
124	2.426	2.320	2.625
144	2.037	2.143	2.685
164	2.099	1.952	2.585
184	2.340	2.056	2.715
204	2.531	2.475	2.523
224	2.736	2.398	2.490
244	2.272	2.495	2.583
264	2.439	2.412	2.242
284	2.081	2.156	2.501
304	2.400	2.566	2.333

PCB 118			
Bottle	Replicate		
	1 (mg kg^{-1})	2 (mg kg^{-1})	3 (mg kg^{-1})
4	2.439	2.260	2.203
24	2.082	2.160	2.169
44	2.437	2.369	2.395
64	2.712	2.322	2.416
84	2.245	2.206	2.265
104	2.146	2.076	2.116
124	1.986	2.083	2.333
144	1.838	1.920	2.302
164	1.729	1.753	2.293
184	2.067	1.909	2.424
204	2.222	2.175	2.256
224	2.415	2.074	2.181
244	1.997	2.251	2.270
264	2.022	2.103	2.007
284	1.870	1.964	2.189
304	2.131	2.307	2.093

PCB 149			
Bottle	Replicate		
	1 (mg kg^{-1})	2 (mg kg^{-1})	3 (mg kg^{-1})
4	1.121	0.999	0.929
24	0.957	0.907	0.985
44	1.097	1.000	1.086
64	1.171	1.001	1.028
84	0.999	0.981	0.966
104	0.994	0.908	0.914
124	0.916	0.876	1.007
144	0.827	0.832	1.019
164	0.801	0.775	1.008
184	0.883	0.839	1.072
204	0.977	0.954	0.962
224	1.066	0.920	0.935
244	0.851	0.966	0.986
264	0.944	0.903	0.856
284	0.786	0.838	0.943
304	0.917	1.009	0.904

Homogeneity study

PCB 153				PCB 138			
Bottle	Replicate			Bottle	Replicate		
	1 (mg kg^{-1})	2 (mg kg^{-1})	3 (mg kg^{-1})		1 (mg kg^{-1})	2 (mg kg^{-1})	3 (mg kg^{-1})
4	1.423	1.266	1.229	4	1.571	1.501	1.429
24	1.193	1.193	1.213	24	1.358	1.364	1.437
44	1.438	1.332	1.337	44	1.641	1.502	1.547
64	1.523	1.308	1.339	64	1.610	1.512	1.551
84	1.279	1.277	1.244	84	1.550	1.442	1.430
104	1.286	1.188	1.177	104	1.535	1.321	1.351
124	1.199	1.170	1.288	124	1.377	1.358	1.502
144	1.059	1.082	1.312	144	1.225	1.241	1.538
164	1.056	0.994	1.290	164	1.212	1.146	1.475
184	1.189	1.060	1.353	184	1.337	1.211	1.595
204	1.272	1.228	1.240	204	1.466	1.420	1.425
224	1.378	1.221	1.238	224	1.572	1.408	1.443
244	1.122	1.256	1.244	244	1.298	1.454	1.440
264	1.210	1.202	1.121	264	1.389	1.369	1.296
284	1.066	1.085	1.204	284	1.239	1.251	1.404
304	1.194	1.303	1.149	304	1.356	1.511	1.353

PCB 170				PCB 180			
Bottle	Replicate			Bottle	Replicate		
	1 (mg kg^{-1})	2 (mg kg^{-1})	3 (mg kg^{-1})		1 (mg kg^{-1})	2 (mg kg^{-1})	3 (mg kg^{-1})
4	0.222	0.20	0.20	4	0.456	0.396	0.389
24	0.206	0.19	0.17	24	0.347	0.357	0.345
44	0.225	0.20	0.20	44	0.409	0.422	0.414
64	0.253	0.20	0.22	64	0.454	0.407	0.393
84	0.213	0.18	0.19	84	0.363	0.393	0.350
104	0.208	0.21	0.17	104	0.378	0.343	0.357
124	0.175	0.20	0.18	124	0.351	0.375	0.382
144	0.165	0.16	0.20	144	0.312	0.324	0.396
164	0.145	0.16	0.25	164	0.299	0.288	0.387
184	0.170	0.14	0.20	184	0.368	0.324	0.388
204	0.190	0.17	0.19	204	0.361	0.368	0.349
224	0.214	0.20	0.21	224	0.435	0.354	0.382
244	0.161	0.21	0.16	244	0.325	0.362	0.364
264	0.169	0.18	0.18	264	0.340	0.360	0.326
284	0.159	0.15	0.18	284	0.333	0.301	0.345
304	0.174	0.21	0.13	304	0.333	0.412	0.330

A.2 Stability study

The measurements for the stability study were both performed under repeatability conditions with a randomised order of extracts and using GC-MS after PFE.

PCB 28 Bottle	t (Months)	T [°C]	-1 (mg kg^{-1})	-2 (mg kg^{-1})	-3 (mg kg^{-1})	Mean (mg kg^{-1})	s (mg kg^{-1})
45	ref	-20	0.6318	0.6137	0.5782	0.6079	0.0273
46	ref	-20	0.5537	0.5688	0.6284	0.5836	0.0395
53	1	4	0.6604	0.6006	0.6142	0.6251	0.0313
54	3	4	0.5566	0.5495	0.4900	0.5320	0.0366
55	6	4	0.5160	0.5355	0.5177	0.5231	0.0108
56	12	4	0.5048	0.4826	0.4823	0.4899	0.0129
62	1	22	0.5151	0.5600	0.5554	0.5435	0.0247
63	3	22	0.4844	0.5034	0.5356	0.5078	0.0258
65	6	22	0.4505	0.4284	0.4644	0.4478	0.0181
66	12	22	0.4514	0.4033	0.4618	0.4389	0.0312
73	1	40	0.5298	0.5492	0.5585	0.5458	0.0146
74	3	40	0.5738	0.4392	0.5657	0.5262	0.0755
75	6	40	0.4136	0.4617	0.4637	0.4463	0.0284
76	12	40	0.3999	0.4054	0.4001	0.4018	0.0031
77	1	60	0.4785	0.4385	0.4436	0.4535	0.0217
78	3	60	0.4387	0.4041	0.4005	0.4144	0.0211
79	6	60	0.2900	0.3186	0.3303	0.3130	0.0207

PCB 44 Bottle	t [Months]	T [°C]	-1 (mg kg^{-1})	-2 (mg kg^{-1})	-3 (mg kg^{-1})	Mean (mg kg^{-1})	s (mg kg^{-1})
45	ref	-20	2.0729	2.0609	1.9322	2.0220	0.0780
46	ref	-20	1.8408	1.9059	2.0902	1.9457	0.1294
53	1	4	2.0906	1.9224	2.0461	2.0197	0.0871
54	3	4	1.8598	1.7722	1.5920	1.7413	0.1365
55	6	4	1.6922	1.7602	1.6819	1.7114	0.0425
56	12	4	1.7042	1.5387	1.6351	1.6260	0.0831
62	1	22	1.6815	1.8736	1.8092	1.7881	0.0977
63	3	22	1.5300	1.6400	1.7897	1.6532	0.1304
65	6	22	1.5599	1.3937	1.5655	1.5064	0.0976
66	12	22	1.5348	1.3203	1.5443	1.4665	0.1267
73	1	40	1.6724	1.8435	1.8916	1.8025	0.1152
74	3	40	1.7927	1.4536	1.8699	1.7054	0.2215
75	6	40	1.3813	1.4989	1.5285	1.4696	0.0778
76	12	40	1.3213	1.3414	1.3161	1.3263	0.0133
77	1	60	1.6185	1.4230	1.4800	1.5072	0.1006
78	3	60	1.4344	1.3292	1.3501	1.3712	0.0557
79	6	60	1.0192	1.1389	1.1655	1.1079	0.0779

Stability study

PCB 52 Bottle	t (Months)	T [°C]	-1 (mg kg^{-1})	-2 (mg kg^{-1})	-3 (mg kg^{-1})	Mean (mg kg^{-1})	s (mg kg^{-1})
45	ref	-20	3.3602	3.3608	3.1454	3.2888	0.1242
46	ref	-20	2.9167	3.0283	3.3732	3.1061	0.2380
53	1	4	3.3889	3.0529	3.3346	3.2588	0.1804
54	3	4	2.9789	2.8123	2.5818	2.7910	0.1994
55	6	4	2.6975	2.8226	2.7143	2.7448	0.0679
56	12	4	2.7465	2.4880	2.6207	2.6184	0.1293
62	1	22	2.6898	3.0208	2.8816	2.8640	0.1662
63	3	22	2.4229	2.6526	2.8931	2.6562	0.2351
65	6	22	2.4193	2.2298	2.5225	2.3905	0.1485
66	12	22	2.5021	2.1004	2.5148	2.3724	0.2356
73	1	40	2.6569	2.9668	3.0831	2.9023	0.2203
74	3	40	2.8489	2.3123	3.0404	2.7339	0.3774
75	6	40	2.2175	2.3837	2.4679	2.3564	0.1274
76	12	40	2.1428	2.1503	2.1307	2.1413	0.0099
77	1	60	2.5842	2.2669	2.4312	2.4274	0.1587
78	3	60	2.3036	2.1552	2.1658	2.2082	0.0828
79	6	60	1.6532	1.8398	1.9130	1.8020	0.1339

Stability study

PCB 101 Bottle	t [Months]	T [°C]	-1 (mg kg^{-1})	-2 (mg kg^{-1})	-3 (mg kg^{-1})	Mean (mg kg^{-1})	s (mg kg^{-1})
45	ref	-20	3.2328	3.2087	3.0917	3.1777	0.0755
46	ref	-20	2.9137	2.9252	3.2534	3.0308	0.1929
53	1	4	3.3329	3.0578	3.2567	3.2158	0.1421
54	3	4	2.9955	2.7953	2.4393	2.7434	0.2817
55	6	4	2.7136	2.8349	2.6515	2.7333	0.0933
56	12	4	2.6743	2.4490	2.4992	2.5408	0.1183
62	1	22	2.7136	2.9366	2.9070	2.8524	0.1211
63	3	22	2.5120	2.6086	2.7350	2.6185	0.1118
65	6	22	2.4182	2.1350	2.4776	2.3436	0.1831
66	12	22	2.3313	2.0601	2.3040	2.2318	0.1493
73	1	40	2.8142	2.8589	2.9829	2.8854	0.0874
74	3	40	2.9577	2.2650	2.8434	2.6887	0.3714
75	6	40	2.2063	2.3154	2.3179	2.2799	0.0637
76	12	40	2.0563	2.1020	2.0765	2.0783	0.0229
77	1	60	2.6295	2.2406	2.2122	2.3608	0.2332
78	3	60	2.4176	2.0647	2.1902	2.2242	0.1789
79	6	60	1.6323	1.8161	1.8311	1.7598	0.1107

Stability study

PCB 118 Bottle	t [Months]	T [°C]	-1 (mg kg^{-1})	-2 (mg kg^{-1})	-3 (mg kg^{-1})	Mean (mg kg^{-1})	s (mg kg^{-1})
45	ref	-20	3.0932	3.0682	2.8557	3.0057	0.1305
46	ref	-20	2.8682	2.8718	3.1475	2.9625	0.1602
53	1	4	3.3551	3.0991	3.0427	3.1657	0.1665
54	3	4	2.8123	2.8711	2.4052	2.6962	0.2537
55	6	4	2.6990	2.7562	2.4878	2.6476	0.1414
56	12	4	2.5603	2.3854	2.4412	2.4623	0.0893
62	1	22	2.6455	2.9149	2.7706	2.7770	0.1348
63	3	22	2.5937	2.5496	2.6231	2.5888	0.0370
65	6	22	2.2600	2.0745	2.2780	2.2042	0.1127
66	12	22	2.2315	2.0572	2.2330	2.1739	0.1010
73	1	40	2.7601	2.7906	2.7222	2.7576	0.0342
74	3	40	2.9500	2.3493	2.7795	2.6930	0.3096
75	6	40	2.0672	2.3084	2.2772	2.2176	0.1312
76	12	40	2.0823	2.1063	2.0925	2.0937	0.0120
77	1	60	2.4949	2.2750	2.1639	2.3113	0.1684
78	3	60	2.3533	2.0446	2.0807	2.1595	0.1688
79	6	60	1.5409	1.6991	1.7477	1.6626	0.1081

Stability study

PCB 138 Bottle	t [Months]	T [°C]	-1 (mg kg^{-1})	-2 (mg kg^{-1})	-3 (mg kg^{-1})	Mean (mg kg^{-1})	s (mg kg^{-1})
45	ref	-20	1.8234	1.8400	1.7112	1.7915	0.0701
46	ref	-20	1.7146	1.7303	1.8943	1.7797	0.0995
53	1	4	2.0021	1.8439	1.8302	1.8921	0.0956
54	3	4	1.6614	1.7107	1.4606	1.6109	0.1325
55	6	4	1.6153	1.6738	1.4713	1.5868	0.1042
56	12	4	1.5400	1.4507	1.4611	1.4840	0.0488
62	1	22	1.5596	1.7256	1.6397	1.6416	0.0830
63	3	22	1.5600	1.5444	1.5741	1.5595	0.0149
65	6	22	1.3517	1.2484	1.3496	1.3166	0.0590
66	12	22	1.3287	1.2377	1.3323	1.2996	0.0536
73	1	40	1.6441	1.6512	1.6183	1.6378	0.0173
74	3	40	1.7329	1.3778	1.6304	1.5803	0.1828
75	6	40	1.2257	1.3698	1.3395	1.3116	0.0760
76	12	40	1.2261	1.2501	1.2800	1.2521	0.0270
77	1	60	1.4607	1.3751	1.2854	1.3737	0.0877
78	3	60	1.3779	1.2171	1.2286	1.2745	0.0897
79	6	60	0.9524	1.0650	1.1022	1.0398	0.0780

Stability study

PCB 149 Bottle	t [Months]	T [℃]	-1 (mg kg^{-1})	-2 (mg kg^{-1})	-3 (mg kg^{-1})	Mean (mg kg^{-1})	s (mg kg^{-1})
45	ref	-20	1.3050	1.2833	1.2143	1.2675	0.0473
46	ref	-20	1.2172	1.2031	1.3273	1.2492	0.0680
53	1	4	1.3517	1.2623	1.2865	1.3002	0.0462
54	3	4	1.1849	1.1479	1.0038	1.1122	0.0957
55	6	4	1.1214	1.1666	1.0384	1.1088	0.0650
56	12	4	1.0963	1.0066	1.0165	1.0398	0.0492
62	1	22	1.0720	1.1970	1.1482	1.1391	0.0630
63	3	22	1.0474	1.0611	1.1122	1.0736	0.0342
65	6	22	0.9465	0.8694	0.9639	0.9266	0.0503
66	12	22	0.9426	0.8322	0.9331	0.9026	0.0612
73	1	40	1.1119	1.1514	1.1433	1.1355	0.0209
74	3	40	1.1715	0.9332	1.1645	1.0897	0.1356
75	6	40	0.8513	0.9387	0.9467	0.9122	0.0529
76	12	40	0.8340	0.8496	0.8451	0.8429	0.0080
77	1	60	1.0169	0.9323	0.8885	0.9459	0.0653
78	3	60	0.9494	0.8367	0.8566	0.8809	0.0602
79	6	60	0.6879	0.7559	0.7701	0.7380	0.0439

Stability study

PCB 153 Bottle	t [Months]	T [°C]	-1 (mg kg^{-1})	-2 (mg kg^{-1})	-3 (mg kg^{-1})	Mean (mg kg^{-1})	s (mg kg^{-1})
45	ref	-20	1.6629	1.5084	1.5112	1.5608	0.0884
46	ref	-20	1.5155	1.5214	1.6966	1.5778	0.1029
53	1	4	1.7476	1.6152	1.5980	1.6536	0.0819
54	3	4	1.4619	1.4691	1.2698	1.4002	0.1130
55	6	4	1.4236	1.4898	1.3004	1.4046	0.0961
56	12	4	1.3764	1.2736	1.2918	1.3140	0.0549
62	1	22	1.3671	1.8271	1.3990	1.5311	0.2569
63	3	22	1.3667	1.5251	1.4458	1.4459	0.0792
65	6	22	1.1867	1.1049	1.2022	1.1646	0.0523
66	12	22	1.1805	1.0930	1.1921	1.1552	0.0542
73	1	40	1.4413	1.4548	1.4546	1.4502	0.0078
74	3	40	1.5222	1.2066	1.4562	1.3950	0.1665
75	6	40	1.0677	1.2031	1.1987	1.1565	0.0769
76	12	40	1.0812	1.0985	1.1076	1.0958	0.0134
77	1	60	1.2827	1.2089	1.1337	1.2084	0.0745
78	3	60	1.2373	1.0674	1.0828	1.1292	0.0940
79	6	60	0.8541	0.9573	0.9837	0.9317	0.0685

Stability study

PCB 170 Bottle	t [Months]	T [°C]	-1 (mg kg^{-1})	-2 (mg kg^{-1})	-3 (mg kg^{-1})	Mean (mg kg^{-1})	s (mg kg^{-1})
45	ref	-20	0.3314	0.3303	0.3185	0.3268	0.0072
46	ref	-20	0.3360	0.3294	0.3563	0.3406	0.0140
53	1	4	0.3545	0.3389	0.3324	0.3420	0.0113
54	3	4	0.3045	0.3168	0.2772	0.2995	0.0202
55	6	4	0.3049	0.3021	0.2824	0.2964	0.0123
56	12	4	0.2852	0.2766	0.2827	0.2815	0.0044
62	1	22	0.2955	0.3114	0.2970	0.3013	0.0088
63	3	22	0.2883	0.2842	0.2965	0.2897	0.0063
65	6	22	0.2599	0.2457	0.2578	0.2545	0.0077
66	12	22	0.2620	0.2456	0.2580	0.2552	0.0085
73	1	40	0.3005	0.3033	0.2983	0.3007	0.0025
74	3	40	0.3227	0.2664	0.3062	0.2984	0.0289
75	6	40	0.2411	0.2649	0.2569	0.2543	0.0121
76	12	40	0.2504	0.2537	0.2503	0.2515	0.0020
77	1	60	0.2669	0.2671	0.2505	0.2615	0.0095
78	3	60	0.2646	0.2407	0.2470	0.2508	0.0124
79	6	60	0.2114	0.2259	0.2329	0.2234	0.0110

Stability study

PCB 180 Bottle	t [Months]	T [°C]	-1 (mg kg^{-1})	-2 (mg kg^{-1})	-3 (mg kg^{-1})	Mean (mg kg^{-1})	s (mg kg^{-1})
45	ref	-20	0.4925	0.4948	0.4708	0.4861	0.0132
46	ref	-20	0.4819	0.4907	0.5278	0.5001	0.0244
53	1	4	0.5367	0.5055	0.4984	0.5135	0.0204
54	3	4	0.4523	0.4648	0.4099	0.4423	0.0288
55	6	4	0.4505	0.4550	0.4144	0.4400	0.0222
56	12	4	0.4210	0.4068	0.4134	0.4137	0.0071
62	1	22	0.4354	0.4698	0.4495	0.4516	0.0173
63	3	22	0.4382	0.4261	0.4422	0.4355	0.0084
65	6	22	0.3817	0.3580	0.3809	0.3735	0.0135
66	12	22	0.3743	0.3554	0.3829	0.3708	0.0141
73	1	40	0.4504	0.4519	0.4377	0.4467	0.0078
74	3	40	0.4665	0.3953	0.4509	0.4376	0.0374
75	6	40	0.3472	0.3865	0.3758	0.3698	0.0203
76	12	40	0.3569	0.3682	0.3666	0.3639	0.0062
77	1	60	0.3983	0.3881	0.3643	0.3836	0.0174
78	3	60	0.3895	0.3525	0.3569	0.3663	0.0202
79	6	60	0.2933	0.3239	0.3407	0.3193	0.0240

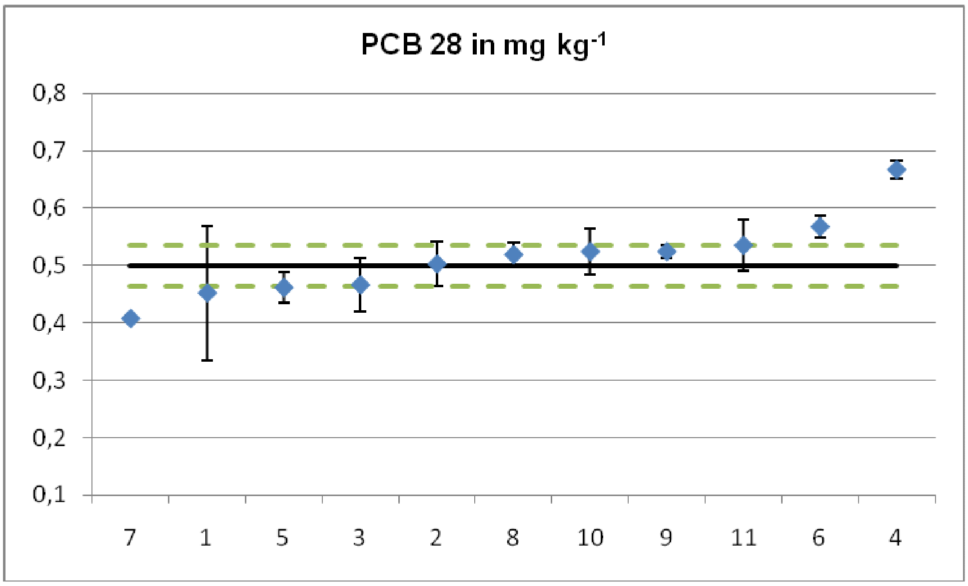
A.3 Characterisation study

Analytical methods performed by the participants

No.	Detection	Column [m]	Extraction method	Internal Standard	Calibration standard
1	ECD	60	ASE Hexane	PCB 209 LGC Standards	PCB Mix 3 in Hexane Ehrenstorfer
2	MS	30	ASE Acetone/ Hexane	^{13}C-labelled PCB31 and 138 Wellington	PCB Mix 19 Ehrenstorfer
3	MS	30	Shaking Acetone/ hexane	PAK-Dx Mix 4 Neochema	PCB Mix 1 and PCB Mix 19 Neochema
4	MS	30	Soxhlet Hexane	PCB 209 LGC Standards	PCB Mix in n-Hexane Neochema
5	MS	30	Twisselmann Acetone/ petrolether	d10-Anthracene Dr. Ehrenstorfer	PCB solution in isooctan (from 3 PCB Mixes) Dr. Ehrenstorfer
6	MS	30	Shaking Acetone/ heptane	Decafluorbiphenyl Sigma-Aldrich	PCB-Mix Ehrensdorfer
7	ECD	60	Sonication Acetone/ i-hexane	PCB - Mix 12 in iso-Octane Dr. Ehrenstorfer	PCB Mix 8 , PCB Mix 149, PCB Mix 118 Dr. Ehrenstorfer
8	MS	30	Shaking Acetone/ petrolether	PCB-Mix 6 (PCB 28, 52, 101, 138, 152, 180, each 10 mg/L) Neochema	PCB-Mix 19 (#-Nr. 20031900), 14 PCBs each 10 mg/L Dr. Ehrenstorfer
9	ECD	60	Soxhlet Hexane	Aldrin Injection standard	PCB MIX 3, each 10 ng/µl Dr. Ehrenstorfer GmbH
10	MS	50	ASE Acetone/ hexane	PCB 28, 52, 101, 153, 138, 180 (^{13}C-labelled) Cambridge Isotope Laboratory	15 PCB Congener Standard Solution each 10 µg/ml in n-Hexane LGC Standards
11	ECD	50	ASE Cyclohexane	PCB 209 LGC Standards	NE-USL 100, 15 PCB in Hexane, 10 µg/ml each LGC Standards

Characterisation study: **PCB 28** in mg kg^{-1}

Lab	1	2	3	4	5	6	Mean	s
1	0.349	0.497	0.332	0.488	0.401	0.647	0.452	0.117
2	0.495	0.525	0.550	0.432	0.501	0.513	0.503	0.040
3	0.410	0.499	0.512	0.414	0.505	0.458	0.466	0.046
4	0.655	0.676	0.656	0.660	0.663	0.694	0.667	0.015
5	0.410	0.460	0.470	0.480	0.480	0.470	0.462	0.026
6	0.580	0.550	0.590	0.573	0.541	0.572	0.568	0.019
7	0.413	0.409	0.401	0.406	0.410	0.407	0.408	0.004
8	0.501	0.535	0.511	0.497	0.548	0.522	0.519	0.020
9	0.519	0.530	0.513	0.518	0.520	0.546	0.524	0.012
10	0.588	0.496	0.541	0.484	0.493	0.544	0.524	0.040
11	0.559	0.605	0.552	0.484	0.505	0.506	0.535	0.045

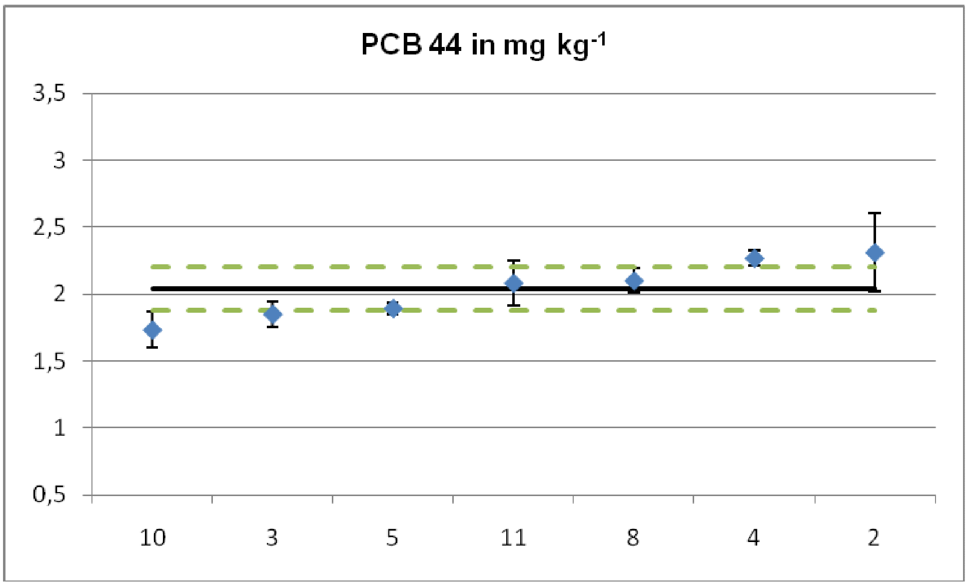


mean of laboratory means (m)

m ± 2 u_{char}

Characterisation study: **PCB 44** in mg kg^{-1}

Lab	1	2	3	4	5	6	Mean	s
1	2.250	2.550	2.680	1.870	2.140	2.390	2.31	0.292
2	1.830	1.940	1.800	1.800	1.990	1.740	1.85	0.095
3	2.209	2.327	2.205	2.278	2.253	2.352	2.27	0.060
4	1.830	1.870	1.910	1.940	1.930	1.880	1.89	0.041
5								
6								
7	1.990	2.190	2.060	2.040	2.230	2.100	2.10	0.092
8								
9	1.921	1.602	1.844	1.681	1.582	1.763	1.73	0.135
10	2.156	2.349	2.134	1.874	1.976	2.012	2.08	0.167
11	2.250	2.550	2.680	1.870	2.140	2.390	2.31	0.292

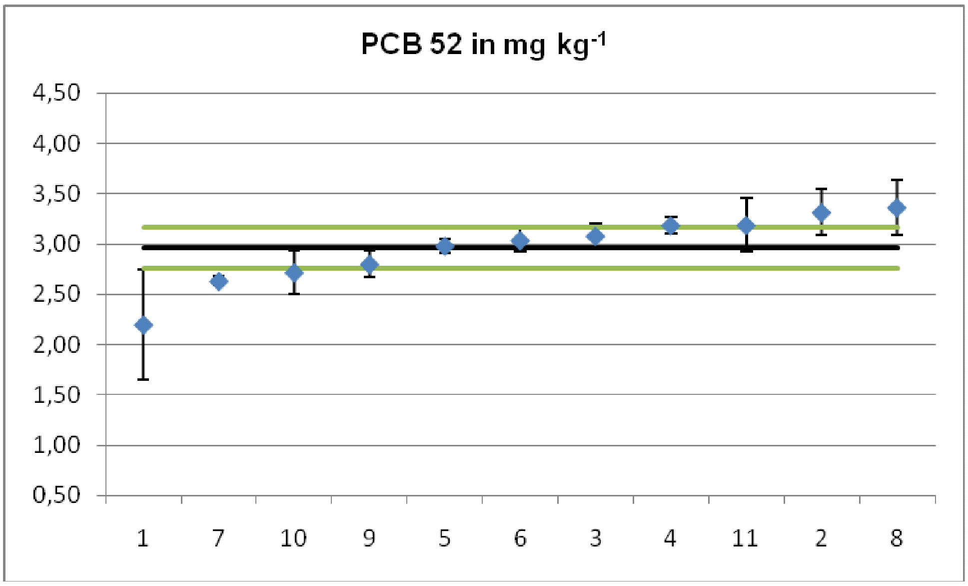


mean of laboratory means (m)

m ± 2 u_{char}

Characterisation study: **PCB 52** in mg kg^{-1}

Lab	1	2	3	4	5	6	Mean	s
1	1.817	2.883	1.961	2.749	2.315	1.481	2.20	0.548
2	3.330	3.480	3.570	2.910	3.260	3.350	3.32	0.228
3	3.150	3.140	2.970	3.160	3.190	2.880	3.08	0.126
4	3.100	3.263	3.144	3.165	3.146	3.307	3.19	0.080
5	2.870	2.960	3.020	3.050	3.040	2.960	2.98	0.068
6	3.110	2.950	3.180	3.070	2.860	3.060	3.04	0.115
7	2.630	2.590	2.700	2.620	2.590	2.670	2.63	0.044
8	3.290	3.680	3.060	3.310	3.720	3.130	3.37	0.276
9	2.822	2.633	3.013	2.683	2.885	2.778	2.80	0.138
10	3.044	2.519	2.892	2.665	2.474	2.723	2.72	0.218
11	3.342	3.618	3.243	2.855	3.002	3.078	3.19	0.272

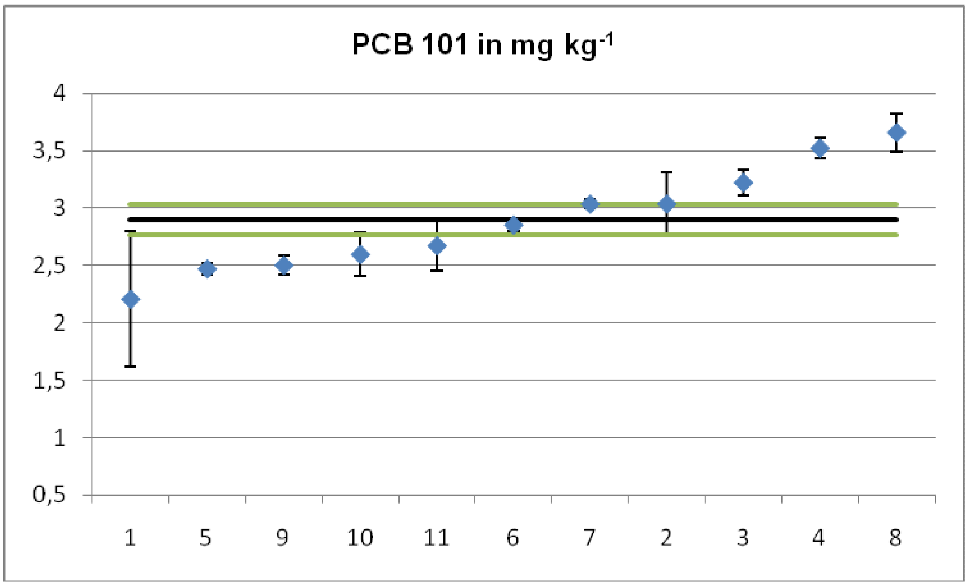


mean of laboratory means (m)

m ± 2 u_{char}

Characterisation study: **PCB 101** in mg kg^{-1}

Lab	1	2	3	4	5	6	Mean	s
1	1.781	2.999	1.896	2.752	2.355	1.478	2.21	0.593
2	2.910	3.210	3.350	2.570	3.050	3.140	3.04	0.273
3	3.280	3.280	3.130	3.250	3.350	3.050	3.22	0.111
4	3.431	3.621	3.459	3.495	3.480	3.650	3.52	0.090
5	2.380	2.450	2.500	2.530	2.500	2.480	2.47	0.053
6	2.910	2.780	2.900	2.890	2.810	2.840	2.86	0.053
7	3.050	3.080	3.070	2.990	3.000	3.030	3.04	0.037
8	3.540	3.852	3.470	3.640	3.880	3.570	3.66	0.170
9	2.426	2.560	2.575	2.505	2.580	2.384	2.51	0.083
10	2.838	2.381	2.755	2.574	2.401	2.647	2.60	0.185
11	2.749	3.022	2.750	2.386	2.543	2.596	2.67	0.218

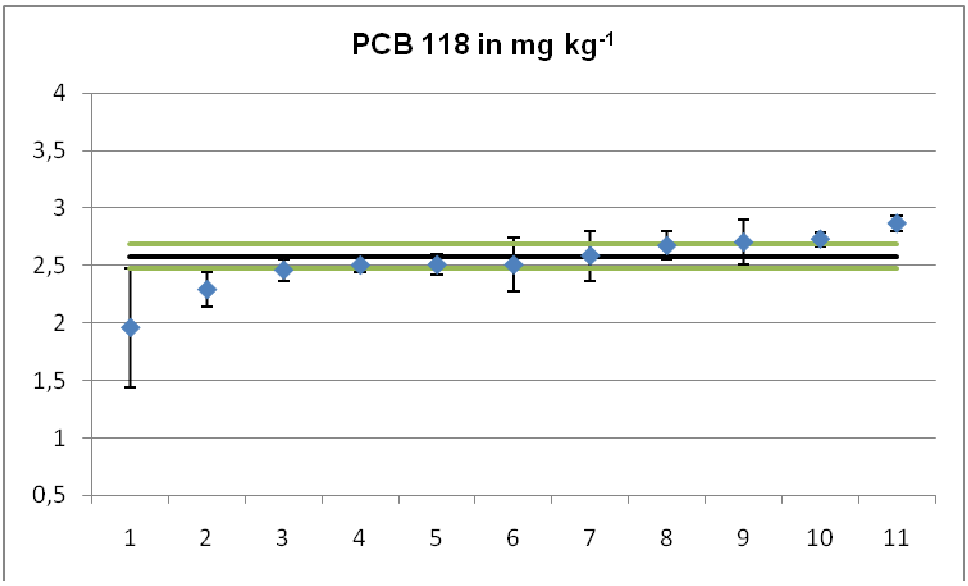


mean of laboratory means (m)

m ± 2 u_{char}

Characterisation study: **PCB 118** in mg kg^{-1}

Lab	1	2	3	4	5	6	Mean	s
1	1.661	2.611	1.635	2.489	2.070	1.292	1.96	0.521
2	2.530	2.880	2.970	2.460	2.640	2.740	2.70	0.198
3	2.560	2.520	2.420	2.610	2.550	2.380	2.51	0.088
4	2.798	2.949	2.839	2.856	2.811	2.948	2.87	0.067
5	2.410	2.470	2.530	2.560	2.300	2.490	2.46	0.094
6	2.600	2.490	2.500	2.530	2.400	2.500	2.50	0.065
7	2.740	2.760	2.740	2.760	2.610	2.770	2.73	0.060
8	2.630	2.230	2.610	2.720	2.190	2.660	2.51	0.233
9	2.641	2.556	2.810	2.525	2.815	2.693	2.67	0.123
10	2.439	2.082	2.437	2.260	2.160	2.369	2.29	0.149
11	2.500	2.950	2.684	2.295	2.505	2.577	2.59	0.219

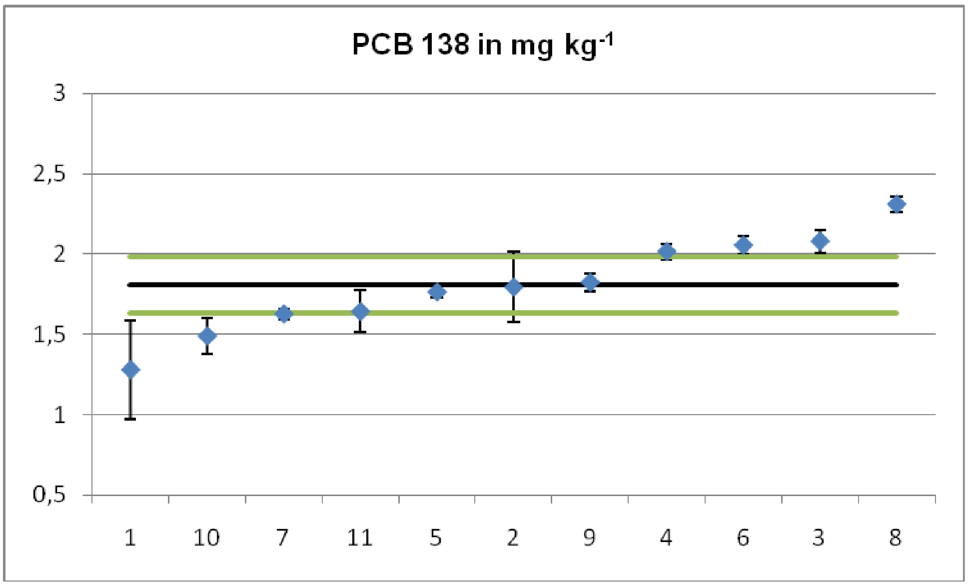


mean of laboratory means (m)

m ± 2 u_{char}

Characterisation study: **PCB 138** in mg kg^{-1}

Lab	1	2	3	4	5	6	Mean	s
1	1.096	1.656	1.082	1.618	1.315	0.911	1.28	0.305
2	1.700	1.890	2.040	1.410	1.800	1.920	1.79	0.220
3	2.090	2.140	2.040	2.040	2.180	1.990	2.08	0.071
4	1.940	2.054	1.990	1.998	2.033	2.081	2.02	0.050
5	1.710	1.760	1.770	1.820	1.780	1.740	1.76	0.037
6	2.130	1.990	2.070	2.080	1.990	2.070	2.06	0.055
7	1.640	1.600	1.670	1.580	1.620	1.650	1.63	0.033
8	2.290	2.260	2.310	2.380	2.260	2.360	2.31	0.051
9	1.866	1.749	1.893	1.771	1.834	1.829	1.82	0.055
10	1.571	1.358	1.641	1.501	1.364	1.502	1.49	0.112
11	1.640	1.848	1.717	1.456	1.579	1.614	1.64	0.132

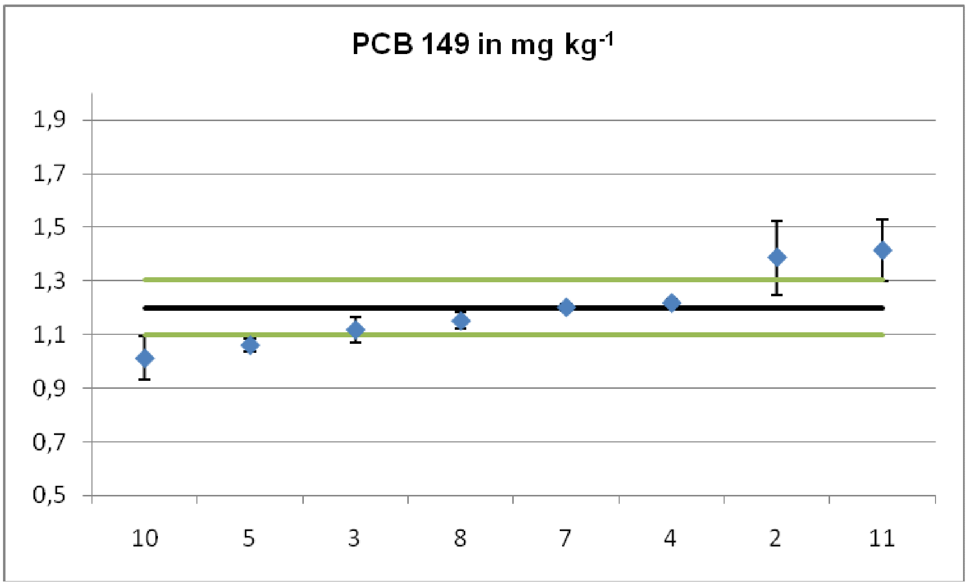


mean of laboratory means (m)

m ± 2 u_{char}

Characterisation study: **PCB 149** in mg kg^{-1}

Lab	1	2	3	4	5	6	Mean	s
1								
2	1.310	1.470	1.550	1.160	1.380	1.450	1.39	0.138
3	1.160	1.110	1.060	1.160	1.160	1.060	1.12	0.049
4	1.196	1.236	1.224	1.217	1.212	1.224	1.22	0.013
5	1.020	1.050	1.070	1.080	1.090	1.060	1.06	0.025
6								
7	1.220	1.190	1.200	1.200	1.210	1.190	1.20	0.012
8	1.120	1.200	1.150	1.150	1.120	1.170	1.15	0.031
9								
10	1.121	0.957	1.097	0.999	0.907	1.000	1.01	0.082
11	1.425	1.585	1.473	1.248	1.360	1.386	1.41	0.113

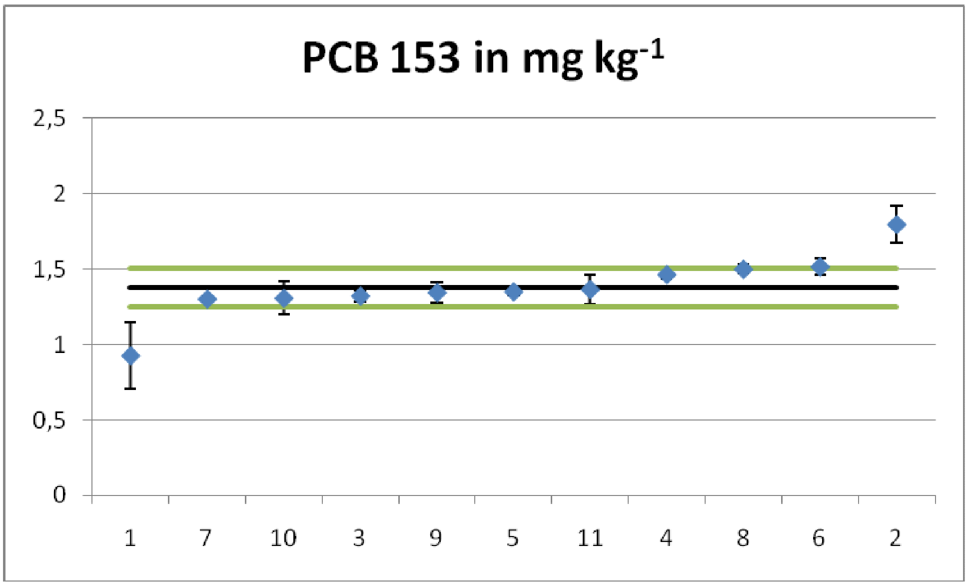


mean of laboratory means (m)

m ± 2 u_{char}

Characterisation study: **PCB 153** in mg kg^{-1}

Lab	1	2	3	4	5	6	Mean	s
1	0.767	1.211	0.798	1.162	0.949	0.665	0.93	0.222
2	1.740	1.890	1.990	1.640	1.730	1.790	1.80	0.125
3	1.330	1.350	1.300	1.310	1.370	1.270	1.32	0.036
4	1.444	1.518	1.450	1.448	1.439	1.489	1.46	0.032
5	1.310	1.330	1.360	1.380	1.370	1.350	1.35	0.026
6	1.610	1.440	1.500	1.540	1.510	1.500	1.52	0.056
7	1.300	1.280	1.290	1.330	1.320	1.290	1.30	0.019
8	1.450	1.520	1.490	1.510	1.510	1.520	1.50	0.027
9	1.315	1.283	1.417	1.299	1.438	1.318	1.35	0.065
10	1.423	1.193	1.438	1.266	1.193	1.332	1.31	0.109
11	1.400	1.524	1.411	1.242	1.304	1.310	1.37	0.101

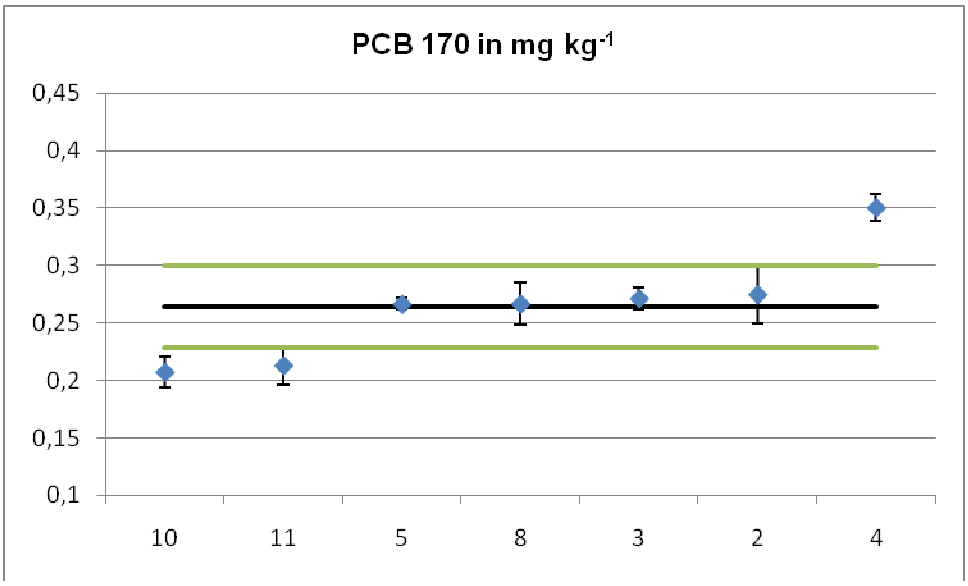


mean of laboratory means (m)

m ± 2 u_{char}

Characterisation study: **PCB 170** in mg kg^{-1}

Lab	1	2	3	4	5	6	Mean	s
1								
2	0.245	0.278	0.283	0.246	0.289	0.309	0.275	0.025
3	0.276	0.279	0.264	0.270	0.283	0.257	0.272	0.010
4	0.352	0.366	0.360	0.352	0.333	0.342	0.351	0.012
5	0.260	0.260	0.270	0.270	0.270	0.270	0.267	0.005
6								
7								
8	0.270	0.232	0.264	0.285	0.277	0.273	0.267	0.018
9								
10	0.222	0.206	0.225	0.198	0.194	0.199	0.207	0.013
11	0.207	0.237	0.228	0.190	0.207	0.210	0.213	0.017

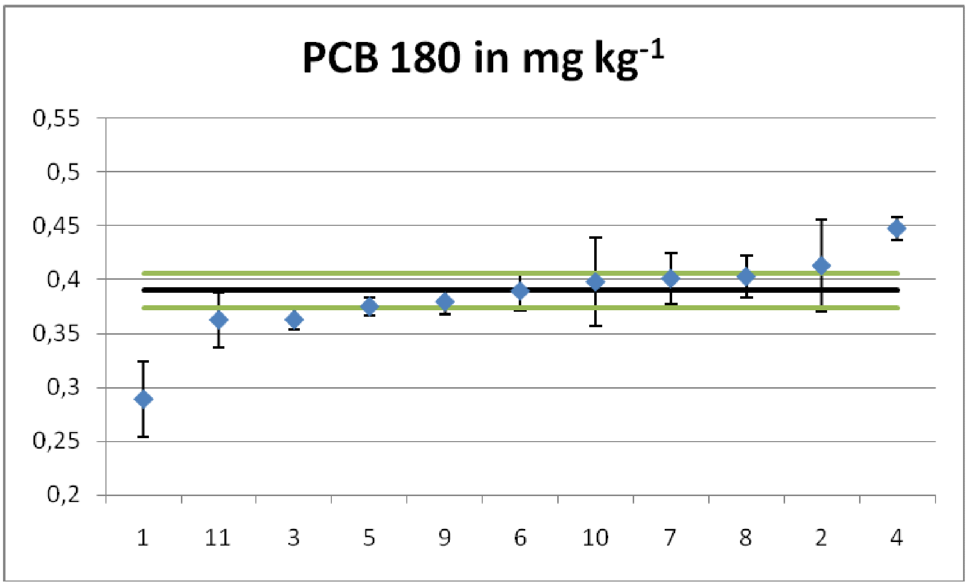


mean of laboratory means (m)

m ± 2 u_{char}

Characterisation study: **PCB 180** in mg kg^{-1}

Lab	1	2	3	4	5	6	Mean	s
1	0.283	0.301	0.275	0.307	0.233	0.336	0.289	0.035
2	0.392	0.442	0.474	0.356	0.388	0.425	0.413	0.042
3	0.356	0.379	0.363	0.358	0.368	0.353	0.363	0.010
4	0.447	0.466	0.450	0.445	0.432	0.446	0.447	0.011
5	0.360	0.370	0.380	0.380	0.380	0.380	0.375	0.008
6	0.419	0.375	0.384	0.403	0.376	0.379	0.389	0.018
7	0.427	0.434	0.381	0.397	0.386	0.381	0.401	0.024
8	0.414	0.381	0.405	0.428	0.377	0.411	0.403	0.020
9	0.379	0.401	0.367	0.373	0.383	0.374	0.380	0.012
10	0.456	0.347	0.409	0.396	0.357	0.422	0.398	0.041
11	0.370	0.402	0.375	0.330	0.348	0.350	0.363	0.025



mean of laboratory means (m)

m ± 2 u_{char}