# BAM-U019a
# Polychlorinated Biphenyls in Soil
## Certification Report

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## Contents

## List of Abbreviations

| | |
|---|---|
| BAM | Bundesanstalt für Materialforschung und -prüfung |
| BIPM | International Bureau of Weights and Measures |
| CCQM | Consultative Committee for Amount of Substance – Metrology in Chemistry |
| ECD | Electron capture detector |
| FBE | Fluidised bed extraction |
| GC | Gas chromatography |
| MS | Mass spectrometry |
| PCB | Polychlorinated Biphenyls |
| PFE | Pressurised fluid extraction |
| PTFE | Polytetrafluoroethylene |
| ref | Stability study reference conditions (permanent at - 20 °C) |
| SD | Standard deviation |
| RSD | Relative standard deviation |

# 1 Introduction

## 1.1 PCB congeners of interest and available analytical procedures

Polychlorinated Biphenyls (PCB) are persistent in the environment and are listed in ANNEX A of the Stockholm convention [1]. They belong to the organic pollutants regularly monitored on contaminated sites. The quantification of PCBs in soil has been standardised using gas chromatography with electron capture detection (GC-ECD) [2, 3] and gas chromatography-mass spectrometry (GC-MS) [4].
BAM-U019a replaces BAM-U019 that is meanwhile sold out and is certified for the extractable mass fractions of PCB congeners PCB-28, PCB-52, PCB-101, PCB-118, PCB-138, PCB-153, and PCB-180. Among the 209 theoretically possible congeners, these are the most often determined ones and they are listed in the respective international standards [2-4].

## 1.2 Strategy of the certification project

The reference material was obtained from a real-world soil collected on a contaminated site after preliminarily checking at BAM for its appropriateness regarding the contents of the target compounds PCB-28, PCB-52, PCB-101, PCB-118, PCB-138, PCB-153 and PCB-180. A target concentration below 10 mg $kg^{-1}$ for each congener was verified before shipment to BAM. For the certification study, nine external laboratories were chosen on basis of their proficiency in PCB analyses which was proven in the 24th regular PT round "Contaminated sites" (2018) operated by BAM. Additionally, one BAM laboratory took part in the certification project. The calibration and measurement capability (CMC) of BAM with regard to PCB analyses has been recognised internationally 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 relevant analytical methods, GC-MS and GC-ECD along with a variety of different extraction procedures were used for quantification of PCB congeners. Traceability was established using calibration solutions derived from commercially available reference material certified for the contents of these congeners. In case of GC-MS partly $^{13}C$-labelled PCBs were applied as internal standards (see clause 5.2.3). This certification project was conducted in accordance with the principles laid down in ISO 17034 [6] and ISO Guide 35 [7].

# 2 Candidate material

A sandy soil was obtained 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 air-drying to constant weight, the bulk material was classified by means of an automatic sieving station and 4.6 kg of the fraction < 125 µm was collected and mixed with 2.2 kg of an uncontaminated sandy soil (< 125 µm) and with 25.7 kg of a loamy sandy agricultural soil (< 125 µm). 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 with a procedure of partitioning and back-mixing a total of 400 units were bottled in 100 mL amber screw-capped glass bottles containing (81.0 ± 0.6) g each by means of a spinning riffler of Retsch GmbH, Haan, Germany. 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-U019a

| Parameter | | Value | Method |
|---|---|---|---|
| Particle size range | (µm) | < 125 | Sieving |
| Water content | (%) | 1.30 ± 0.03 | Karl Fischer titration |
| Drying loss | (%) | 1.18 ± 0.17 | 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 accredited procedure employed for this study involved pressurised fluid extraction (PFE) with acetone/cyclohexane (1:1) followed by GC-MS. The sample intake for each analysis was 5 g which is recommended as minimum sample intake in the certificate.
Fifteen units were selected equidistantly from the whole batch of the 400 units in the order of bottling. The selected units were analysed in triplicate each. All 15 units were extracted once under repeatability conditions on three consecutive days. All 45 extracts were analysed under repeatability conditions in that the PCB congeners in all 45 extracts were quantified against one calibration. Table 1 contains the synopsis of the 1-way analysis of variance (ANOVA). All measurement results can be found in Annex A.

Table 2: Results of the 1-way ANOVA of the PCB determination in the candidate material

| Congener | *Mean*[a] (mg kg$^{-1}$) | $MS_{between}$[b] (mg$^2$ kg$^{-2}$) | $MS_{within}$[c] (mg$^2$ kg$^{-2}$) | $F_{obs}$[d] | $F_{crit}$[d] | $u_{bb}$[e] (mg kg$^{-1}$) | $u_{bb,r}$[f] |
|---|---|---|---|---|---|---|---|
| PCB 28 | 0.186 | 0.000061 | 0.000060 | 1.02 | 2.04 | 0.000568 | 0.003058 |
| PCB 52 | 2.394 | 0.009178 | 0.007760 | 1.18 | 2.04 | 0.021743 | 0.009081 |
| PCB 101 | 2.709 | 0.010638 | 0.010378 | 1.02 | 2.04 | 0.009292 | 0.003431 |
| PCB 138 | 1.442 | 0.002861 | 0.002885 | 0.99 | 2.04 | 0 | 0 |
| PCB 153 | 1.072 | 0.002944 | 0.002907 | 1.01 | 2.04 | 0.003528 | 0.003292 |
| PCB 180 | 0.292 | 0.000108 | 0.000126 | 0.86 | 2.04 | 0 | 0 |

[a] *Mean of the homogeneity study*
[b] *Mean of squared deviations between bottles (from 1-way ANOVA)*
[c] *Mean of squared deviations within bottles (from 1-way ANOVA)*
[d] *Observed F-values (= $MS_{between}/MS_{within}$) and critical F-values*
[e] *Standard uncertainty between the bottles: Estimate of inhomogeneity contribution to the total uncertainty*
[f] *Relative standard uncertainty between the bottles ($u_{bb}$/mean)*

On basis of these results the candidate material was considered sufficiently homogeneous for the use as reference material. The standard uncertainty between the bottles (units) $u_{bb}$ was determined according to Eq. 1 in cases where $MS_{between} > MS_{within}$ and otherwise set to 0 [7].

$$u_{bb} = \sqrt{\frac{MS_{between} - MS_{within}}{n}} \qquad (1)$$

with n = 3 (number of replicate determinations on each unit).

# 4 Stability study

## 4.1 Initial stability study

From earlier experience with organics in various matrices a temperature-driven deterioration of the PCB content was to be expected also for this material. 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 the respective periods of time individual units were stored at -20 °C. All units were analysed for PCBs under repeatability conditions together with 4 reference samples which had been kept at -20 °C since bottling. For PCB quantification an accredited procedure involving extraction with cyclohexane/acetone followed by GC/MS detection (SOP BAM-1.7-PV028) was employed. All measurement results are collected in Annex B.

Table 3: Accelerated ageing of selected units of BAM-U019a: exposition temperatures, periods and number of exposed units.

| Ageing time [Months] | +4 °C | +20 °C | +40 °C | +60 °C | Remark |
|---|---|---|---|---|---|
| 1 | 2 | 2 | 2 | 2 | *initial study* |
| 3 | 2 | 2 | 2 | 2 | *initial study* |
| 6 | 2 | 2 | 2 | 2 | *initial study* |
| 12 | 2 | 2 | 2 | | *initial study* |
| 24 | x | x | *x* | | *post certification monitoring* |
| 36 | x | x | *x* | | *post certification monitoring* |

Data evaluation and expiry date estimation strictly followed the procedures as comprehensively described in [9]: From semi-logarithmic plots of measured single values over time, effective deterioration rates k_eff were determined and tested against an *Arrhenius* model describing the temperature dependence of the deterioration rates. All of the PCBs matched the model, some of them excellently. Activation energies as determined from the model were in the region between 38 and 88 kJ $mol^{-1}$ depending on the PCB congener considered. Figures 1 and 2 show the dependence of the logarithm of the effective deterioration rate k_eff on the inverse temperature for PCB-101 and PCB-138 as examples. The activation energies $\Delta E$ are 42.5 kJ $mol^{-1}$ and 38.4 kJ $mol^{-1}$, respectively.

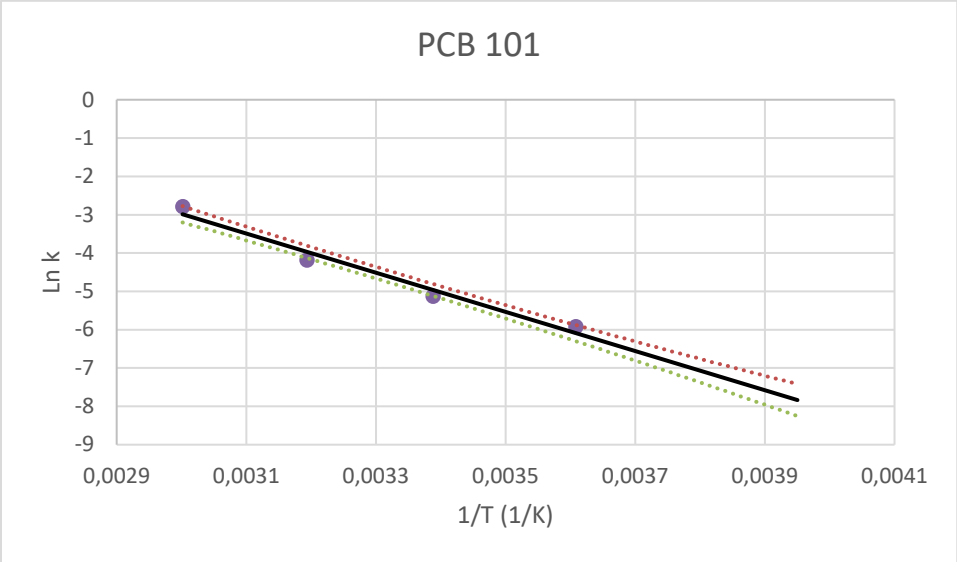


Fig. 1: Effective deterioration rate versus inverse temperature for PCB 101.

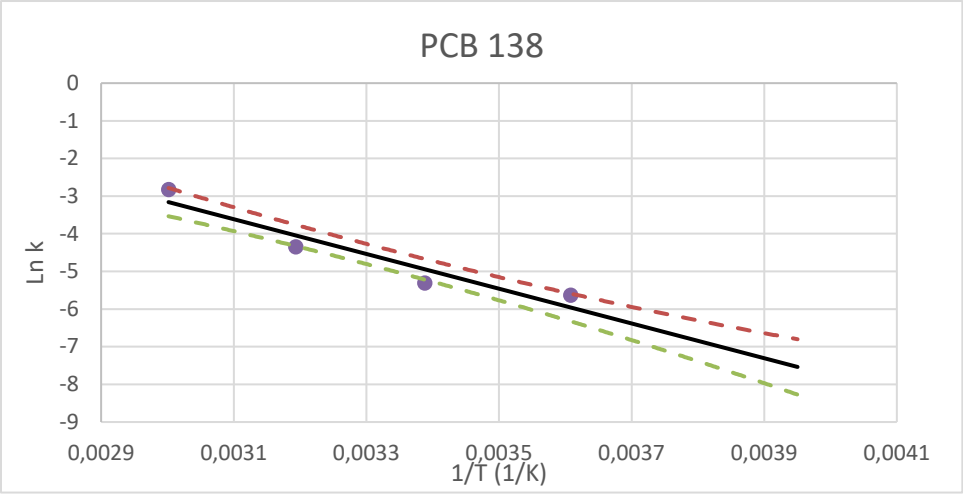


Fig. 2: Effective deterioration rate versus inverse temperature for PCB-138.

Table 4: Estimated period in months until which the certified values will remain within the certified uncertainties *U* at the storage temperature of -20 °C

| Congener | Period (months) |
|---|---|
| PCB-28 | 883 |
| PCB-52 | 6145 |
| PCB-101 | 167 |
| PCB-118 | 16481 |
| PCB-153 | 93 |
| PCB-138 | 91 |
| PCB-180 | 19919 |

Because of the minimum stability estimate of about seven years consider an uncertainty contribution due to long-term (in)stability was not considered. Post-certification monitoring is scheduled for 2024.

### 4.2 Post-certification stability monitoring

The post-certification measurements will be conducted in time and using the estimate given in Table 4. Several units investigated during the initial stability study were stored at +4 °C, +20 °C, and +40 °C, respectively. In addition, sample were stored at -80 °C to serve as reference for the bottles stored at - 20 °C. That way, information on the long-term stability of units of BAM-U019a will be obtained. Earlier experience with similar materials including BAM-U019 does not indicate any enhanced deterioration of bottles stored at -20 °C over several years. or once opened bottles if they were closed directly after subsample withdrawal and stored again at -20 °C. This information is given in the certificate (see also Clause 6.2).

# 5 Certification study

## 5.1 Design of the certification study

The certification project was organised as interlaboratory comparison of selected laboratories (Table 5) with known proficiency for the matrix/analyte combination in question. Their proficiency had been demonstrated in the 24th round of the proficiency testing scheme "Contaminated Sites" run by BAM directly before start of the certification project.

Table 5: Participants in the certification exercise in alphabetical order

| | |
|---|---|
| ALBO-tec | Mülheim an der Ruhr |
| Analysen Service GmbH | Leipzig |
| Bundesanstalt für Materialforschung und -prüfung (BAM) | Berlin |
| CAL GmbH & Co. KG | Darmstadt |
| CLG Chemisches Labor Dr. Graser KG | Schonungen |
| Dr. Ronald Fischer AUb | Bad Berka |
| GBA Gesellschaft für Bioanalytik mbH | Pinneberg |
| GEO-data GmbH | Garbsen |
| Infraserv GmbH & Co. Höchst KG | Frankfurt am Main |
| VKTA - Strahlenschutz, Analytik & Entsorgung Rossendorf e.V. | Dresden |

The applied options for extraction and instrumental quantification (GC-ECD and GC-MS) currently in use were covered by the participants (Table 6). One unit of the candidate material had to be analysed six times by each laboratory. A rough information on the level of content of the PCB congeners to be expected was provided in order to allow a reasonable adjustment of individual calibrations. Commercial calibration standards were used as calibrants (see also Clause 5.2.3). Laboratories using GC-MS applied either labelled PCB congeners or PCB-209 as internal standards as did the laboratories performing GC-ECD.

Table 6: Extraction and determination methods (order not identical to Table 5)

| Laboratory | Extraction | Determination method |
|---|---|---|
| 1 | Sonication; cyclohexane | GC-ECD |
| 2 | Soxhlet; n-hexane | GC-ECD |
| 3 | Microwave assisted extraction; acetone/hexane (1:1) | GC-MS |
| 4 | - no information provided | GC-MS |
| 5 | Sonication; acetone/cyclohexane | GC-MS |
| 6 | Soxhlet | GC-MS |
| 7 | Sonication; n-hexane | GC-MS |
| 8 | Sonication/shaking; acetone/cyclohexane | GC-MS |
| 9 | Shaking; n-hexane | GC-MS |
| 10 | Pressurised fluid extraction; acetone/hexane (1:1) | GC-MS |

## 5.2 Evaluation of results and certified values

The results of the certification study are listed comprehensively in Table 7 (p.11) and were evaluated in accordance with ISO Guide 35 [7]. For all measurement data see ANNEX C. The computer software SoftCRM [10] was used for statistical tests.

### 5.2.1 Statistical evaluation

Since the participants in the interlaboratory comparison used different extraction techniques and solvents according to their specific procedures, and applied both GC-ECD and GC-MS for quantification, 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:

- *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*
- *Tests for skewness and kurtosis as well as variance homogeneity (Scheffé and Bartlett)*

As usually observed in such interlaboratory comparisons the data sets differ significantly (*Scheffé*-Test) and variances are inhomogeneous (*Bartlett*-Test at the significance level of 0.01).

The most important test results based upon the laboratory means and standard deviations are given in Table 7.

A number of outliers were identified by the *Cochran*, *Grubbs*, *Dixon* and *Nalimov* tests. Nevertheless, no measurements data were eliminated on basis of any outlier test.

Table 7: Evaluation of the certification study (testing for outliers)

| | Mean[a] (mg kg$^{-1}$) | SD[b] (mg kg$^{-1}$) | $u_x$[c] (mg kg$^{-1}$) | Data[d] sets | Cochran[e] (0.01) | Grubbs[e] 0.01(0.05) | Dixon[e] 0.01(0.05) | Nalimov[e] 0.01(0.05) | Gauss[f] 0.05;0.01 | Certify | Outliers eliminated |
|---|---|---|---|---|---|---|---|---|---|---|---|
| PCB-28 | 0.156 | 0.032 | 0.0103 | 10 | C01(-) | -(-) | -(-) | -(-) | yes;yes | yes | none |
| PCB-52 | 1.663 | 0.357 | 0.1129 | 10 | C08, C10 | -(-) | -(-) | -(C10) | yes;yes | yes | none |
| PCB-101 | 1.762 | 0.497 | 0.1573 | 10 | C01, C10 | -(-) | -(-) | -(C03,C10) | no;no | yes | none |
| PCB-118 | 1.480 | 0.239 | 0.0755 | 10 | C08, C01 | -(-) | -(-) | -(-) | yes;yes | yes | none |
| PCB-153 | 0.830 | 0.296 | 0.0937 | 10 | -(-) | C10(-) | -(-) | C10(-) | yes;yes | yes | none |
| PCB-138 | 1.014 | 0.193 | 0.0612 | 10 | C01,C10 | -(-) | -(-) | -(C03) | yes;yes | yes | none |
| PCB-180 | 0.213 | 0.046 | 0.0146 | 10 | -(-) | -(C07) | -(-) | L07(-) | no;yes | yes | none |

[a] *Mean of laboratory means (no outliers eliminated)*
[b] *Standard deviation of means of laboratory means*
[c] *Standard uncertainty of the mean of means*
[d] *Number of data sets used for the evaluation of mean, SD, $u_x$ after assessment of outliers (see right column)*
[e] *Laboratories identified as outlier by the respective test on the given level of significance*
[f] *Results of the test for normality*

### 5.2.2 Certified values and combined uncertainties

Table 8 comprises the evaluation of certified values. The means of laboratory means were taken as the best estimates $w_{char}$ for the values to be certified. The standard deviations of the mean of laboratory means were taken as the uncertainty contributions $u_x$ from the interlaboratory exercise.
Besides $u_x$ the contribution from a possibly undetected inhomogeneity $u_{bb}$ (see Clause 3, Table 2) was included in the combined uncertainty $u_{com}$ (Eq. 3) of each congener as recommended in [7, 11].

$$u_{com,r}^2 = u_{x,r}^2 + u_{bb,r}^2 \qquad (3)$$

The index r indicates that the relative values for both uncertainty contributions were related to $w_{char}$ before calculation of $u_{com}$. The expanded uncertainties $U$ were derived according to Eq. 4 with the coverage factor $k$ = 2.

$$U = k \cdot u_{com} \qquad (4)$$

Table 8: Certified PCB mass fractions in BAM-U019a in mg kg$^{-1}$ (before rounding)

| | value[a] | SD[b] | $u_x$[c] | $u_{bb}$[d] | $u_{com}$[e] | $U$ ($k$=2)[f] |
|---|---|---|---|---|---|---|
| PCB-28 | 0.1563 | 0.0324 | 0.0103 | 0.00048 | 0.010269402 | 0.0205 |
| PCB-52 | 1.6629 | 0.3569 | 0.1129 | 0.01510 | 0.11387114 | 0.2277 |
| PCB-101 | 1.7622 | 0.4975 | 0.1573 | 0.00605 | 0.157435543 | 0.3149 |
| PCB-118 | 1.4798 | 0.2389 | 0.0755 | 0.01344 | 0.076717549 | 0.1534 |
| PCB-153 | 1.0138 | 0.1934 | 0.0612 | 0 | 0.06116034 | 0.1223 |
| PCB-138 | 0.8302 | 0.2962 | 0.0937 | 0.00273 | 0.093692453 | 0.1874 |
| PCB-180 | 0.2125 | 0.0463 | 0.0146 | 0 | 0.014647658 | 0.0293 |

[a] *Mean of laboratory means after elimination of outliers*
[b] *Standard deviation of laboratory means*
[c] *Standard deviation of the mean of laboratory means: SD/(n)$^{½}$ with n=number participating laboratories*
[d] *Uncertainty between the bottles (= value * $u_{bb,r}$ with $u_{bb,r}$ taken from Table 2)*
[e] *Combined uncertainty (Eq. 3)*
[f] *Expanded uncertainty (Eq. 4)*

The values and the expanded uncertainties are rounded according to the recommendations of DIN 1333 [12] and are given in Table 9. The water content of BAM-U019a is (1.30 ± 0.03) % and remains stable if the material is handled according to the instructions given in the certificate (see Clause 6).

Table 9: Certified mass fractions of PCB congeners in BAM-U019a in mg kg$^{-1}$ (after rounding)

| | *Value* | *U* |
|---|---|---|
| PCB-28 | 0.157 | 0.021 |
| PCB-52 | 1.67 | 0.23 |
| PCB-101 | 1.8 | 0.4 |
| PCB-118 | 1.48 | 0.16 |
| PCB-153 | 1.02 | 0.13 |
| PCB-138 | 0.84 | 0.19 |
| PCB-180 | 0.213 | 0.030 |

### 5.2.3 Traceability

All certified values refer to the extractable contents of PCB congeners and are conventional to this extent. However, different extraction methods and solvents have been used such that systematic biases will (at least partially) be cancelled out. In order to ensure traceability of the extractable content as defined above, various certified calibration standards commercially available from Dr. Ehrenstorfer (LGC Standards) were employed by the participants. At BAM the certified PCB solution PCB Mix21 (LGC Standards) was employed (accreditation number: D-RM-19883-01. The accreditation on basis of ISO 17034 covers the preparation of calibration standards).

## 6 Information on the proper use of BAM-U019a

### 6.1 Shelf life

From the initial stability study a preliminary shelf life of three years at -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 PCB allows the dispatch of the material at ambient temperature. On receiving, it is to be stored at -20 °C. Before withdrawing a subsample the bottle has to have reached ambient temperature. Thereafter, the bottle must be closed tightly and stored at -20 °C again. The water content remains stable when the material is treated as described.

### 6.3 Safety instructions

The soil was not sterilised, however, it is supposed to not exhibit any biological activity due to having been air-dried to constant mass and displaying a water content of 1.3%. 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 polychlorinated biphenyls. Any unintended contact to this material should be treated as the contact to a dry soil without specifically hazardous properties.

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 Bundesanstalt für Materialforschung und -prüfung (BAM) 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 15308:2016: Characterization of waste - Determination of selected polychlorinated biphenyls (PCB) in solid waste by using capillary gas chromatography with electron capture or mass spectrometric detection.

[4] EN 16167:2012: Soil, treated biowaste and sludge - Determination of polychlorinated biphenyls (PCB) by gas chromatography with mass selective detection (GC-MS) and gas chromatography with electron-capture detection (GC-ECD).

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# ANNEX A

## Results of the homogeneity study

| PCB-28 | | | | PCB-52 | | | |
|---|---|---|---|---|---|---|---|
| Bottle | Replicates | | | Bottle | Replicates | | |
| | 1 (mg kg$^{-1}$) | 2 (mg kg$^{-1}$) | 3 (mg kg$^{-1}$) | | 1 (mg kg$^{-1}$) | 2 (mg kg$^{-1}$) | 3 (mg kg$^{-1}$) |
| 3 | 0.192 | 0.188 | 0.179 | 3 | 2.352 | 2.410 | 2.290 |
| 36 | 0.189 | 0.187 | 0.184 | 36 | 2.430 | 2.412 | 2.383 |
| 62 | 0.200 | 0.188 | 0.187 | 62 | 2.548 | 2.420 | 2.417 |
| 78 | 0.196 | 0.184 | 0.187 | 78 | 2.494 | 2.359 | 2.415 |
| 109 | 0.195 | 0.190 | 0.175 | 109 | 2.461 | 2.438 | 2.291 |
| 144 | 0.192 | 0.181 | 0.187 | 144 | 2.473 | 2.349 | 2.402 |
| 162 | 0.199 | 0.194 | 0.186 | 162 | 2.519 | 2.518 | 2.373 |
| 200 | 0.186 | 0.180 | 0.181 | 200 | 2.392 | 2.354 | 2.367 |
| 225 | 0.155 | 0.182 | 0.181 | 225 | 1.952 | 2.377 | 2.353 |
| 239 | 0.189 | 0.189 | 0.181 | 239 | 2.409 | 2.415 | 2.377 |
| 256 | 0.189 | 0.181 | 0.188 | 256 | 2.417 | 2.364 | 2.428 |
| 295 | 0.194 | 0.185 | 0.179 | 295 | 2.451 | 2.364 | 2.321 |
| 325 | 0.193 | 0.182 | 0.181 | 325 | 2.465 | 2.384 | 2.355 |
| 351 | 0.199 | 0.175 | 0.182 | 351 | 2.546 | 2.329 | 2.372 |
| 397 | 0.187 | 0.188 | 0.179 | 397 | 2.404 | 2.455 | 2.347 |

| PCB-101 | | | | PCB-153 | | | |
|---|---|---|---|---|---|---|---|
| Bottle | Replicates | | | Bottle | Replicates | | |
| | 1 (mg kg$^{-1}$) | 2 (mg kg$^{-1}$) | 3 (mg kg$^{-1}$) | | 1 (mg kg$^{-1}$) | 2 (mg kg$^{-1}$) | 3 (mg kg$^{-1}$) |
| 3 | 2.69 | 2.74 | 2.59 | 3 | 1.43 | 1.45 | 1.38 |
| 36 | 2.75 | 2.72 | 2.69 | 36 | 1.46 | 1.44 | 1.43 |
| 62 | 2.87 | 2.74 | 2.75 | 62 | 1.52 | 1.46 | 1.47 |
| 78 | 2.80 | 2.67 | 2.71 | 78 | 1.49 | 1.41 | 1.44 |
| 109 | 2.79 | 2.77 | 2.57 | 109 | 1.49 | 1.47 | 1.37 |
| 144 | 2.80 | 2.68 | 2.71 | 144 | 1.49 | 1.43 | 1.44 |
| 162 | 2.83 | 2.83 | 2.66 | 162 | 1.50 | 1.51 | 1.42 |
| 200 | 2.72 | 2.67 | 2.66 | 200 | 1.45 | 1.42 | 1.42 |
| 225 | 2.21 | 2.70 | 2.65 | 225 | 1.18 | 1.44 | 1.42 |
| 239 | 2.74 | 2.74 | 2.71 | 239 | 1.46 | 1.47 | 1.43 |
| 256 | 2.74 | 2.67 | 2.77 | 256 | 1.45 | 1.43 | 1.48 |
| 295 | 2.80 | 2.69 | 2.63 | 295 | 1.47 | 1.42 | 1.40 |
| 325 | 2.77 | 2.70 | 2.65 | 325 | 1.49 | 1.43 | 1.42 |
| 351 | 2.88 | 2.63 | 2.68 | 351 | 1.54 | 1.40 | 1.43 |
| 397 | 2.74 | 2.77 | 2.64 | 397 | 1.45 | 1.47 | 1.41 |

## Homogeneity study

| PCB-138 | | | | PCB -180 | | | |
|---|---|---|---|---|---|---|---|
| Bottle | Replicates | | | Bottle | Replicates | | |
| | 1 (mg $kg^{-1}$) | 2 (mg $kg^{-1}$) | 3 (mg $kg^{-1}$) | | 1 (mg $kg^{-1}$) | 2 (mg $kg^{-1}$) | 3 (mg $kg^{-1}$) |
| 3 | 1.09 | 1.17 | 1.02 | 3 | 0.29 | 0.29 | 0.28 |
| 36 | 1.10 | 1.04 | 1.06 | 36 | 0.30 | 0.29 | 0.29 |
| 62 | 1.15 | 1.02 | 1.10 | 62 | 0.31 | 0.29 | 0.29 |
| 78 | 1.13 | 0.96 | 1.11 | 78 | 0.30 | 0.28 | 0.29 |
| 109 | 1.08 | 1.05 | 1.05 | 109 | 0.30 | 0.30 | 0.28 |
| 144 | 1.12 | 1.05 | 1.07 | 144 | 0.30 | 0.29 | 0.29 |
| 162 | 1.11 | 1.09 | 1.08 | 162 | 0.31 | 0.30 | 0.29 |
| 200 | 1.08 | 1.07 | 1.10 | 200 | 0.29 | 0.29 | 0.29 |
| 225 | 0.86 | 1.02 | 1.08 | 225 | 0.24 | 0.29 | 0.29 |
| 239 | 1.09 | 1.02 | 1.10 | 239 | 0.30 | 0.30 | 0.29 |
| 256 | 1.07 | 1.03 | 0.98 | 256 | 0.29 | 0.29 | 0.30 |
| 295 | 1.11 | 1.02 | 1.04 | 295 | 0.30 | 0.29 | 0.28 |
| 325 | 1.10 | 1.08 | 1.08 | 325 | 0.30 | 0.29 | 0.29 |
| 351 | 1.15 | 1.06 | 1.11 | 351 | 0.31 | 0.28 | 0.29 |
| 397 | 1.10 | 1.11 | 1.09 | 397 | 0.30 | 0.30 | 0.29 |

# ANNEX B

## Stability study

| PCB-28 | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| Bottle | t [months] | T [°C] | -1 (mg kg$^{-1}$) | -2 (mg kg$^{-1}$) | -3 (mg kg$^{-1}$) | Mean (mg kg$^{-1}$) | SD (mg kg$^{-1}$) | RSD (%) |
| 110 | ref | -20 | 0.2121 | 0.2119 | 0.2122 | 0.2121 | 0.0002 | 0.08 |
| 111 | ref | -20 | 0.2130 | 0.2116 | 0.2146 | 0.2131 | 0.0015 | 0.71 |
| 110 | ref | -20 | 0.2215 | 0.2281 | 0.2364 | 0.2287 | 0.0074 | 3.25 |
| 111 | ref | -20 | 0.2342 | 0.2232 | 0.2298 | 0.2291 | 0.0055 | 2.42 |
| 114 | 1 | 4 | 0.2284 | 0.2296 | 0.2351 | 0.2310 | 0.0036 | 1.56 |
| 115 | 1 | 4 | 0.2223 | 0.2260 | 0.2146 | 0.2210 | 0.0058 | 2.64 |
| 116 | 3 | 4 | 0.2240 | 0.2220 | 0.2253 | 0.2238 | 0.0017 | 0.74 |
| 117 | 3 | 4 | 0.2258 | 0.2243 | 0.2281 | 0.2261 | 0.0020 | 0.86 |
| 118 | 6 | 4 | 0.2235 | 0.2205 | 0.2211 | 0.2217 | 0.0015 | 0.70 |
| 119 | 6 | 4 | 0.2227 | 0.2229 | 0.2296 | 0.2250 | 0.0039 | 1.74 |
| 120 | 12 | 4 | 0.2284 | 0.2296 | 0.2351 | 0.2310 | 0.0036 | 1.56 |
| 121 | 12 | 4 | 0.2354 | 0.2065 | 0.2275 | 0.2231 | 0.0149 | 6.68 |
| 124 | 1 | 22 | 0.2430 | 0.2356 | 0.2153 | 0.2313 | 0.0144 | 6.21 |
| 125 | 1 | 22 | 0.2231 | 0.2161 | 0.2180 | 0.2191 | 0.0036 | 1.64 |
| 126 | 3 | 22 | 0.2148 | 0.2187 | 0.2181 | 0.2172 | 0.0021 | 0.97 |
| 127 | 3 | 22 | 0.2165 | 0.2182 | 0.2169 | 0.2172 | 0.0009 | 0.40 |
| 128 | 6 | 22 | 0.2171 | 0.2143 | 0.2153 | 0.2156 | 0.0014 | 0.65 |
| 129 | 6 | 22 | 0.2202 | 0.2229 | 0.2189 | 0.2207 | 0.0020 | 0.92 |
| 130 | 12 | 22 | 0.2188 | 0.2216 | 0.2261 | 0.2222 | 0.0037 | 1.65 |
| 131 | 12 | 22 | 0.2259 | 0.2289 | 0.2230 | 0.2259 | 0.0030 | 1.31 |
| 134 | 1 | 40 | 0.2141 | 0.2181 | 0.2148 | 0.2156 | 0.0022 | 1.00 |
| 135 | 1 | 40 | 0.2157 | 0.2114 | 0.2070 | 0.2114 | 0.0043 | 2.05 |
| 136 | 3 | 40 | 0.2086 | 0.2073 | 0.2094 | 0.2085 | 0.0011 | 0.51 |
| 137 | 3 | 40 | 0.2093 | 0.2082 | 0.2125 | 0.2100 | 0.0023 | 1.08 |
| 138 | 6 | 40 | 0.2094 | 0.2045 | 0.2096 | 0.2078 | 0.0029 | 1.40 |
| 139 | 6 | 40 | 0.2113 | 0.2107 | 0.2068 | 0.2096 | 0.0024 | 1.15 |
| 140 | 12 | 40 | 0.2002 | 0.2023 | 0.2089 | 0.2038 | 0.0045 | 2.23 |
| 141 | 12 | 40 | 0.2060 | 0.2048 | 0.2001 | 0.2036 | 0.0031 | 1.53 |
| 142 | 1 | 60 | 0.1811 | 0.1748 | 0.1769 | 0.1776 | 0.0032 | 1.81 |
| 143 | 1 | 60 | 0.2071 | 0.2042 | 0.2011 | 0.2041 | 0.0030 | 1.46 |
| 145 | 3 | 60 | 0.1604 | 0.1585 | 0.1568 | 0.1586 | 0.0018 | 1.13 |
| 146 | 3 | 60 | 0.1450 | 0.1538 | 0.1514 | 0.1500 | 0.0046 | 3.04 |
| 147 | 6 | 60 | 0.1323 | 0.1353 | 0.1298 | 0.1324 | 0.0027 | 2.06 |
| 148 | 6 | 60 | 0.1326 | 0.1335 | 0.1280 | 0.1313 | 0.0029 | 2.22 |

## Stability study

| PCB-52 | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| Bottle | t [months] | T [°C] | -1 (mg kg$^{-1}$) | -2 (mg kg$^{-1}$) | -3 (mg kg$^{-1}$) | Mean (mg kg$^{-1}$) | SD (mg kg$^{-1}$) | RSD (%) |
| 110 | ref | -20 | 2.3146 | 2.3323 | 2.3073 | 2.3181 | 0.0129 | 0.56 |
| 111 | ref | -20 | 2.3187 | 2.3118 | 2.3086 | 2.3130 | 0.0052 | 0.22 |
| 110 | ref | -20 | 2.3033 | 2.3229 | 2.3740 | 2.3334 | 0.0365 | 1.56 |
| 111 | ref | -20 | 2.4085 | 2.2604 | 2.2857 | 2.3182 | 0.0792 | 3.42 |
| 114 | 1 | 4 | 2.3719 | 2.3115 | 2.2030 | 2.2955 | 0.0856 | 3.73 |
| 115 | 1 | 4 | 2.3284 | 2.4086 | 2.3086 | 2.3485 | 0.0529 | 2.25 |
| 116 | 3 | 4 | 2.3719 | 2.3115 | 2.2030 | 2.2955 | 0.0856 | 3.73 |
| 117 | 3 | 4 | 2.3284 | 2.4086 | 2.3086 | 2.3485 | 0.0529 | 2.25 |
| 118 | 6 | 4 | 2.4022 | 2.3645 | 2.3312 | 2.3660 | 0.0355 | 1.50 |
| 119 | 6 | 4 | 2.3910 | 2.4012 | 2.3967 | 2.3963 | 0.0051 | 0.21 |
| 120 | 12 | 4 | 2.3307 | 2.3385 | 2.4095 | 2.3596 | 0.0434 | 1.84 |
| 121 | 12 | 4 | 2.4259 | 2.1308 | 2.3116 | 2.2894 | 0.1487 | 6.50 |
| 124 | 1 | 22 | 2.3207 | 2.3568 | 2.2559 | 2.3111 | 0.0511 | 2.21 |
| 125 | 1 | 22 | 2.4097 | 2.3308 | 2.3441 | 2.3615 | 0.0422 | 1.79 |
| 126 | 3 | 22 | 2.2914 | 2.3710 | 2.3466 | 2.3364 | 0.0408 | 1.74 |
| 127 | 3 | 22 | 2.3372 | 2.3670 | 2.3101 | 2.3381 | 0.0285 | 1.22 |
| 128 | 6 | 22 | 2.2622 | 2.2783 | 2.2817 | 2.2740 | 0.0104 | 0.46 |
| 129 | 6 | 22 | 2.3387 | 2.3635 | 2.2571 | 2.3198 | 0.0557 | 2.40 |
| 130 | 12 | 22 | 2.2347 | 2.2304 | 2.2866 | 2.2506 | 0.0313 | 1.39 |
| 131 | 12 | 22 | 2.3009 | 2.3308 | 2.2434 | 2.2917 | 0.0444 | 1.94 |
| 134 | 1 | 40 | 2.2849 | 2.3127 | 2.2492 | 2.2823 | 0.0318 | 1.39 |
| 135 | 1 | 40 | 2.3034 | 2.2506 | 2.1989 | 2.2510 | 0.0523 | 2.32 |
| 136 | 3 | 40 | 2.1635 | 2.1802 | 2.2034 | 2.1824 | 0.0201 | 0.92 |
| 137 | 3 | 40 | 2.1986 | 2.1885 | 2.2465 | 2.2112 | 0.0310 | 1.40 |
| 138 | 6 | 40 | 2.0998 | 2.1376 | 2.1747 | 2.1374 | 0.0374 | 1.75 |
| 139 | 6 | 40 | 2.1994 | 2.2250 | 2.1783 | 2.2009 | 0.0234 | 1.06 |
| 140 | 12 | 40 | 1.9338 | 1.9400 | 1.9844 | 1.9527 | 0.0276 | 1.42 |
| 141 | 12 | 40 | 2.0056 | 1.9820 | 1.9299 | 1.9725 | 0.0387 | 1.96 |
| 142 | 1 | 60 | 1.8211 | 1.7211 | 1.7536 | 1.7653 | 0.0510 | 2.89 |
| 143 | 1 | 60 | 2.1302 | 2.1315 | 2.0659 | 2.1092 | 0.0375 | 1.78 |
| 145 | 3 | 60 | 1.5929 | 1.5900 | 1.5487 | 1.5772 | 0.0248 | 1.57 |
| 146 | 3 | 60 | 1.4661 | 1.5333 | 1.5263 | 1.5086 | 0.0369 | 2.45 |
| 147 | 6 | 60 | 1.3778 | 1.3858 | 1.3788 | 1.3808 | 0.0044 | 0.32 |
| 148 | 6 | 60 | 1.4004 | 1.4004 | 1.3586 | 1.3865 | 0.0241 | 1.74 |

## Stability study

| PCB-101 | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| Bottle | t [months] | T [°C] | -1 (mg kg$^{-1}$) | -2 (mg kg$^{-1}$) | -3 (mg kg$^{-1}$) | Mean (mg kg$^{-1}$) | SD (mg kg$^{-1}$) | RSD (%) |
| 110 | ref | -20 | 2.1078 | 2.1296 | 2.1301 | 2.1225 | 0.0127 | 0.60 |
| 111 | ref | -20 | 2.2041 | 2.1458 | 2.1389 | 2.1630 | 0.0358 | 1.66 |
| 110 | ref | -20 | 2.1513 | 2.1944 | 2.2432 | 2.1963 | 0.0460 | 2.09 |
| 111 | ref | -20 | 2.1167 | 2.1101 | 2.1751 | 2.1340 | 0.0358 | 1.68 |
| 114 | 1 | 4 | 2.2049 | 2.1698 | 2.1429 | 2.1726 | 0.0311 | 1.43 |
| 115 | 1 | 4 | 2.2131 | 2.2264 | 2.1389 | 2.1928 | 0.0471 | 2.15 |
| 116 | 3 | 4 | 2.2546 | 2.2266 | 2.2505 | 2.2439 | 0.0151 | 0.67 |
| 117 | 3 | 4 | 2.2902 | 2.2463 | 2.3035 | 2.2800 | 0.0299 | 1.31 |
| 118 | 6 | 4 | 2.2625 | 2.2223 | 2.2864 | 2.2571 | 0.0324 | 1.44 |
| 119 | 6 | 4 | 2.2567 | 2.2602 | 2.3435 | 2.2868 | 0.0491 | 2.15 |
| 120 | 12 | 4 | 2.1825 | 2.1078 | 2.1610 | 2.1504 | 0.0385 | 1.79 |
| 121 | 12 | 4 | 2.2513 | 1.8916 | 2.1823 | 2.1084 | 0.1909 | 9.06 |
| 124 | 1 | 22 | 2.2234 | 2.2315 | 2.2105 | 2.2218 | 0.0106 | 0.48 |
| 125 | 1 | 22 | 2.2887 | 2.1823 | 2.1843 | 2.2184 | 0.0608 | 2.74 |
| 126 | 3 | 22 | 2.1381 | 2.2080 | 2.2057 | 2.1839 | 0.0397 | 1.82 |
| 127 | 3 | 22 | 2.1858 | 2.2306 | 2.1137 | 2.1767 | 0.0590 | 2.71 |
| 128 | 6 | 22 | 2.1680 | 2.1390 | 2.1535 | 2.1535 | 0.0145 | 0.67 |
| 129 | 6 | 22 | 2.1963 | 2.2028 | 2.1763 | 2.1918 | 0.0138 | 0.63 |
| 130 | 12 | 22 | 2.0947 | 2.0017 | 2.0538 | 2.0501 | 0.0466 | 2.27 |
| 131 | 12 | 22 | 2.0677 | 2.0920 | 2.0427 | 2.0675 | 0.0247 | 1.19 |
| 134 | 1 | 40 | 2.1369 | 2.1646 | 2.1097 | 2.1370 | 0.0274 | 1.28 |
| 135 | 1 | 40 | 2.1526 | 2.1121 | 2.0987 | 2.1211 | 0.0281 | 1.32 |
| 136 | 3 | 40 | 2.0347 | 2.0854 | 1.9931 | 2.0377 | 0.0462 | 2.27 |
| 137 | 3 | 40 | 2.0003 | 1.9950 | 2.0299 | 2.0084 | 0.0188 | 0.94 |
| 138 | 6 | 40 | 1.9758 | 1.9416 | 1.9554 | 1.9576 | 0.0172 | 0.88 |
| 139 | 6 | 40 | 2.0025 | 2.0164 | 1.9902 | 2.0030 | 0.0131 | 0.66 |
| 140 | 12 | 40 | 1.7976 | 1.7451 | 1.8181 | 1.7869 | 0.0376 | 2.11 |
| 141 | 12 | 40 | 1.7818 | 1.7719 | 1.7441 | 1.7660 | 0.0196 | 1.11 |
| 142 | 1 | 60 | 1.6850 | 1.6108 | 1.6240 | 1.6399 | 0.0396 | 2.41 |
| 143 | 1 | 60 | 1.9498 | 1.9459 | 1.9083 | 1.9347 | 0.0229 | 1.19 |
| 145 | 3 | 60 | 1.4791 | 1.4842 | 1.4660 | 1.4764 | 0.0094 | 0.63 |
| 146 | 3 | 60 | 1.3935 | 1.4304 | 1.4333 | 1.4191 | 0.0222 | 1.56 |
| 147 | 6 | 60 | 1.3139 | 1.3321 | 1.3192 | 1.3217 | 0.0094 | 0.71 |
| 148 | 6 | 60 | 1.3473 | 1.3372 | 1.3166 | 1.3337 | 0.0156 | 1.17 |

## Stability study

| PCB-118 | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| Bottle | t [months] | T [°C] | -1 (mg kg$^{-1}$) | -2 (mg kg$^{-1}$) | -3 (mg kg$^{-1}$) | Mean (mg kg$^{-1}$) | SD (mg kg$^{-1}$) | RSD (%) |
| 110 | ref | -20 | 1.9848 | 1.9485 | 1.9469 | 1.9600 | 0.0214 | 1.09 |
| 111 | ref | -20 | 2.0204 | 1.9404 | 1.9396 | 1.9668 | 0.0464 | 2.36 |
| 110 | ref | -20 | 1.9805 | 1.9921 | 2.0217 | 1.9981 | 0.0213 | 1.06 |
| 111 | ref | -20 | 2.0540 | 1.9675 | 1.9824 | 2.0013 | 0.0462 | 2.31 |
| 114 | 1 | 4 | 1.9755 | 1.9306 | 1.9345 | 1.9469 | 0.0249 | 1.28 |
| 115 | 1 | 4 | 1.9915 | 1.9867 | 1.9396 | 1.9726 | 0.0287 | 1.45 |
| 116 | 3 | 4 | 2.0076 | 2.0432 | 2.0602 | 2.0370 | 0.0268 | 1.32 |
| 117 | 3 | 4 | 2.0687 | 2.0792 | 2.0334 | 2.0604 | 0.0240 | 1.16 |
| 118 | 6 | 4 | 2.0594 | 2.0397 | 2.0890 | 2.0627 | 0.0249 | 1.20 |
| 119 | 6 | 4 | 2.0651 | 2.0505 | 2.1057 | 2.0738 | 0.0286 | 1.38 |
| 120 | 12 | 4 | 2.0164 | 2.0359 | 2.0437 | 2.0320 | 0.0141 | 0.69 |
| 121 | 12 | 4 | 2.0692 | 1.8516 | 1.9861 | 1.9690 | 0.1098 | 5.57 |
| 124 | 1 | 22 | 2.0480 | 2.0298 | 1.9964 | 2.0247 | 0.0261 | 1.29 |
| 125 | 1 | 22 | 2.0478 | 2.0004 | 1.9994 | 2.0158 | 0.0277 | 1.37 |
| 126 | 3 | 22 | 1.9623 | 1.9768 | 1.9825 | 1.9739 | 0.0104 | 0.53 |
| 127 | 3 | 22 | 2.0091 | 2.0490 | 1.9854 | 2.0145 | 0.0321 | 1.59 |
| 128 | 6 | 22 | 1.9810 | 1.9510 | 1.9157 | 1.9492 | 0.0327 | 1.68 |
| 129 | 6 | 22 | 2.0111 | 2.0427 | 2.0015 | 2.0184 | 0.0215 | 1.07 |
| 130 | 12 | 22 | 1.9584 | 1.9517 | 1.9710 | 1.9604 | 0.0098 | 0.50 |
| 131 | 12 | 22 | 2.0089 | 2.0077 | 1.9474 | 1.9880 | 0.0352 | 1.77 |
| 134 | 1 | 40 | 1.9567 | 2.0008 | 1.9579 | 1.9718 | 0.0251 | 1.27 |
| 135 | 1 | 40 | 1.9872 | 1.9120 | 1.9095 | 1.9362 | 0.0442 | 2.28 |
| 136 | 3 | 40 | 1.8872 | 1.8339 | 1.9324 | 1.8845 | 0.0493 | 2.62 |
| 137 | 3 | 40 | 1.9172 | 1.9067 | 1.9441 | 1.9226 | 0.0193 | 1.00 |
| 138 | 6 | 40 | 1.8620 | 1.8901 | 1.8532 | 1.8684 | 0.0193 | 1.03 |
| 139 | 6 | 40 | 1.9142 | 1.9406 | 1.9148 | 1.9232 | 0.0150 | 0.78 |
| 140 | 12 | 40 | 1.7416 | 1.7555 | 1.7804 | 1.7592 | 0.0197 | 1.12 |
| 141 | 12 | 40 | 1.7888 | 1.7901 | 1.7606 | 1.7798 | 0.0167 | 0.94 |
| 142 | 1 | 60 | 1.6519 | 1.5693 | 1.5479 | 1.5897 | 0.0549 | 3.45 |
| 143 | 1 | 60 | 1.8931 | 1.8754 | 1.7919 | 1.8535 | 0.0541 | 2.92 |
| 145 | 3 | 60 | 1.4148 | 1.4039 | 1.4324 | 1.4170 | 0.0144 | 1.01 |
| 146 | 3 | 60 | 1.3512 | 1.3938 | 1.3966 | 1.3805 | 0.0255 | 1.85 |
| 147 | 6 | 60 | 1.1849 | 1.2558 | 1.1824 | 1.2077 | 0.0417 | 3.45 |
| 148 | 6 | 60 | 1.2067 | 1.2375 | 1.1748 | 1.2063 | 0.0314 | 2.60 |

## Stability study

| PCB-153 | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| Bottle | t [months] | T [°C] | -1 (mg kg$^{-1}$) | -2 (mg kg$^{-1}$) | -3 (mg kg$^{-1}$) | Mean (mg kg$^{-1}$) | SD (mg kg$^{-1}$) | RSD (%) |
| 110 | ref | -20 | 1.0767 | 1.0233 | 1.0339 | 1.0446 | 0.0283 | 2.71 |
| 111 | ref | -20 | 1.0417 | 1.0341 | 1.0362 | 1.0373 | 0.0039 | 0.38 |
| 110 | ref | -20 | 1.0025 | 1.0097 | 1.0326 | 1.0149 | 0.0157 | 1.55 |
| 111 | ref | -20 | 1.0428 | 0.9905 | 1.0128 | 1.0154 | 0.0263 | 2.59 |
| 114 | 1 | 4 | 1.0543 | 1.0449 | 1.0365 | 1.0452 | 0.0089 | 0.86 |
| 115 | 1 | 4 | 1.0785 | 1.0682 | 1.0362 | 1.0610 | 0.0220 | 2.08 |
| 116 | 3 | 4 | 1.0589 | 1.0434 | 1.0661 | 1.0561 | 0.0116 | 1.10 |
| 117 | 3 | 4 | 1.0727 | 1.0615 | 1.0735 | 1.0692 | 0.0067 | 0.63 |
| 118 | 6 | 4 | 1.0557 | 1.0395 | 1.0008 | 1.0320 | 0.0282 | 2.73 |
| 119 | 6 | 4 | 1.0569 | 1.0451 | 1.0820 | 1.0613 | 0.0189 | 1.78 |
| 120 | 12 | 4 | 1.0204 | 1.0267 | 1.0534 | 1.0335 | 0.0175 | 1.70 |
| 121 | 12 | 4 | 1.0455 | 0.9238 | 1.0072 | 0.9922 | 0.0622 | 6.27 |
| 124 | 1 | 22 | 1.0656 | 1.0373 | 1.0291 | 1.0440 | 0.0192 | 1.84 |
| 125 | 1 | 22 | 1.0667 | 1.0280 | 1.0330 | 1.0426 | 0.0211 | 2.02 |
| 126 | 3 | 22 | 1.0035 | 1.0360 | 1.0405 | 1.0267 | 0.0202 | 1.97 |
| 127 | 3 | 22 | 1.0271 | 1.0535 | 1.0197 | 1.0334 | 0.0177 | 1.72 |
| 128 | 6 | 22 | 1.0181 | 1.0062 | 1.0120 | 1.0121 | 0.0059 | 0.59 |
| 129 | 6 | 22 | 1.0306 | 1.0376 | 1.0275 | 1.0319 | 0.0052 | 0.50 |
| 130 | 12 | 22 | 0.9803 | 0.9715 | 1.0009 | 0.9842 | 0.0151 | 1.54 |
| 131 | 12 | 22 | 1.0138 | 1.0112 | 0.9790 | 1.0013 | 0.0194 | 1.94 |
| 134 | 1 | 40 | 0.9888 | 0.9976 | 0.9815 | 0.9893 | 0.0081 | 0.82 |
| 135 | 1 | 40 | 1.0165 | 0.9922 | 0.9695 | 0.9927 | 0.0235 | 2.36 |
| 136 | 3 | 40 | 0.9598 | 0.9707 | 0.9755 | 0.9687 | 0.0080 | 0.83 |
| 137 | 3 | 40 | 0.9789 | 0.9735 | 0.9842 | 0.9789 | 0.0053 | 0.54 |
| 138 | 6 | 40 | 0.9697 | 0.9415 | 0.9677 | 0.9596 | 0.0158 | 1.64 |
| 139 | 6 | 40 | 0.9717 | 0.9691 | 0.9585 | 0.9664 | 0.0070 | 0.73 |
| 140 | 12 | 40 | 0.8567 | 0.8591 | 0.8787 | 0.8648 | 0.0121 | 1.40 |
| 141 | 12 | 40 | 0.8817 | 0.8749 | 0.8545 | 0.8704 | 0.0141 | 1.62 |
| 142 | 1 | 60 | 0.8264 | 0.7936 | 0.8035 | 0.8079 | 0.0168 | 2.08 |
| 143 | 1 | 60 | 0.9372 | 0.9360 | 0.9091 | 0.9274 | 0.0159 | 1.71 |
| 145 | 3 | 60 | 0.7404 | 0.7398 | 0.7212 | 0.7338 | 0.0109 | 1.49 |
| 146 | 3 | 60 | 0.6797 | 0.7061 | 0.7136 | 0.6998 | 0.0178 | 2.54 |
| 147 | 6 | 60 | 0.6592 | 0.6560 | 0.6560 | 0.6571 | 0.0019 | 0.28 |
| 148 | 6 | 60 | 0.6753 | 0.6665 | 0.6607 | 0.6675 | 0.0074 | 1.10 |

## Stability study

| PCB-138 | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| Bottle | t [months] | T [°C] | -1 (mg kg$^{-1}$) | -2 (mg kg$^{-1}$) | -3 (mg kg$^{-1}$) | Mean (mg kg$^{-1}$) | SD (mg kg$^{-1}$) | RSD (%) |
| 110 | ref | -20 | 1.6096 | 1.6180 | 1.6572 | 1.6283 | 0.0254 | 1.56 |
| 111 | ref | -20 | 1.6641 | 1.6519 | 1.6440 | 1.6533 | 0.0101 | 0.61 |
| 110 | ref | -20 | 1.5779 | 1.6070 | 1.6497 | 1.6115 | 0.0361 | 0.02 |
| 111 | ref | -20 | 1.6397 | 1.5525 | 1.5825 | 1.5916 | 0.0443 | 0.03 |
| 114 | 1 | 4 | 1.6863 | 1.6422 | 1.6417 | 1.6567 | 0.0256 | 1.55 |
| 115 | 1 | 4 | 1.6911 | 1.6826 | 1.6440 | 1.6726 | 0.0251 | 1.50 |
| 116 | 3 | 4 | 1.6861 | 1.6642 | 1.6919 | 1.6807 | 0.0146 | 0.87 |
| 117 | 3 | 4 | 1.7039 | 1.6932 | 1.6922 | 1.6964 | 0.0065 | 0.38 |
| 118 | 6 | 4 | 1.6899 | 1.6646 | 1.6962 | 1.6835 | 0.0167 | 0.99 |
| 119 | 6 | 4 | 1.6748 | 1.6978 | 1.7341 | 1.7023 | 0.0299 | 1.76 |
| 120 | 12 | 4 | 1.5986 | 1.6147 | 1.6518 | 1.6217 | 0.0273 | 1.68 |
| 121 | 12 | 4 | 1.6501 | 1.4811 | 1.5881 | 1.5731 | 0.0855 | 5.43 |
| 124 | 1 | 22 | 1.6558 | 1.6559 | 1.6317 | 1.6478 | 0.0140 | 0.85 |
| 125 | 1 | 22 | 1.7030 | 1.6283 | 1.6399 | 1.6571 | 0.0402 | 2.43 |
| 126 | 3 | 22 | 1.5982 | 1.6306 | 1.6471 | 1.6253 | 0.0249 | 1.53 |
| 127 | 3 | 22 | 1.6348 | 1.6753 | 1.6096 | 1.6399 | 0.0332 | 2.02 |
| 128 | 6 | 22 | 1.6237 | 1.5856 | 1.6122 | 1.6072 | 0.0195 | 1.21 |
| 129 | 6 | 22 | 1.6405 | 1.6535 | 1.6289 | 1.6410 | 0.0123 | 0.75 |
| 130 | 12 | 22 | 1.5455 | 1.5248 | 1.5704 | 1.5469 | 0.0228 | 1.47 |
| 131 | 12 | 22 | 1.5844 | 1.5730 | 1.5279 | 1.5618 | 0.0299 | 1.91 |
| 134 | 1 | 40 | 1.5872 | 1.5971 | 1.5650 | 1.5831 | 0.0165 | 1.04 |
| 135 | 1 | 40 | 1.6036 | 1.5720 | 1.5561 | 1.5772 | 0.0242 | 1.53 |
| 136 | 3 | 40 | 1.5172 | 1.5402 | 1.5627 | 1.5400 | 0.0228 | 1.48 |
| 137 | 3 | 40 | 1.5534 | 1.5520 | 1.5791 | 1.5615 | 0.0153 | 0.98 |
| 138 | 6 | 40 | 1.5424 | 1.5045 | 1.5158 | 1.5209 | 0.0195 | 1.28 |
| 139 | 6 | 40 | 1.5461 | 1.5472 | 1.5348 | 1.5427 | 0.0069 | 0.45 |
| 140 | 12 | 40 | 1.3445 | 1.3634 | 1.3750 | 1.3610 | 0.0154 | 1.13 |
| 141 | 12 | 40 | 1.3884 | 1.3779 | 1.3475 | 1.3713 | 0.0213 | 1.55 |
| 142 | 1 | 60 | 1.3209 | 1.2476 | 1.2710 | 1.2799 | 0.0374 | 2.93 |
| 143 | 1 | 60 | 1.5020 | 1.4875 | 1.4646 | 1.4847 | 0.0188 | 1.27 |
| 145 | 3 | 60 | 1.1555 | 1.1665 | 1.1294 | 1.1505 | 0.0191 | 1.66 |
| 146 | 3 | 60 | 1.0770 | 1.1104 | 1.1173 | 1.1016 | 0.0215 | 1.96 |
| 147 | 6 | 60 | 1.0270 | 1.0229 | 1.0328 | 1.0276 | 0.0050 | 0.49 |
| 148 | 6 | 60 | 1.0551 | 1.0484 | 1.0233 | 1.0423 | 0.0168 | 1.61 |

## Stability study

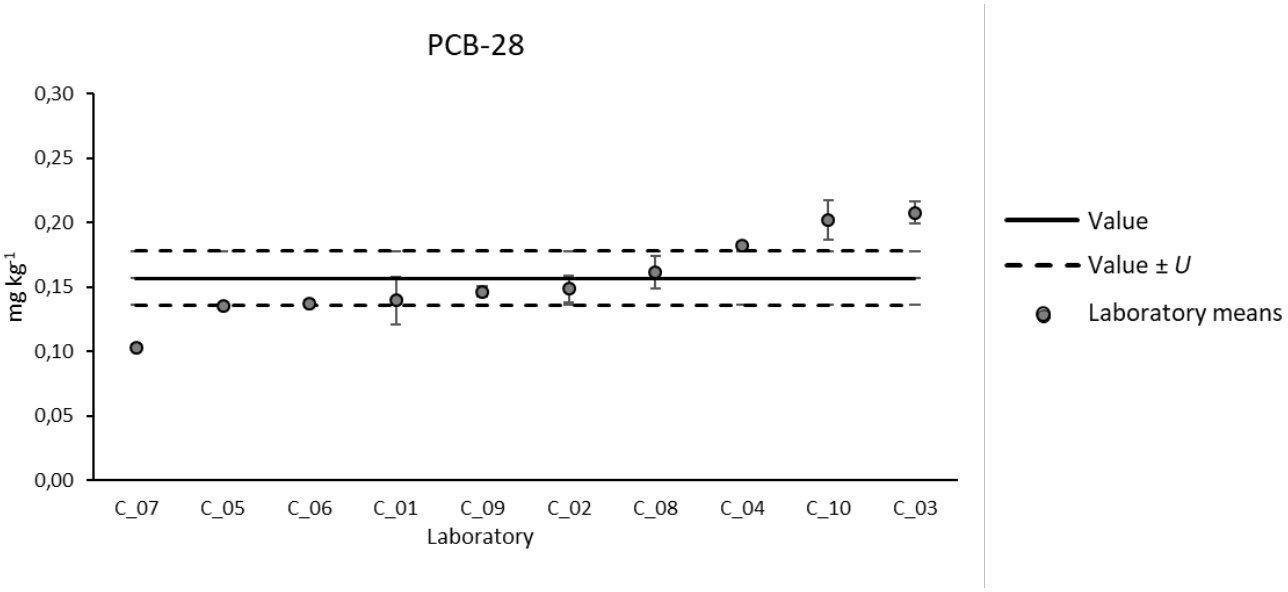
| PCB-180 | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| Bottle | t [months] | T [°C] | -1 (mg kg$^{-1}$) | -2 (mg kg$^{-1}$) | -3 (mg kg$^{-1}$) | Mean (mg kg$^{-1}$) | SD (mg kg$^{-1}$) | RSD (%) |
| 110 | ref | -20 | 0.2872 | 0.2912 | 0.2978 | 0.2920 | 0.0054 | 1.84 |
| 111 | ref | -20 | 0.2998 | 0.2987 | 0.2994 | 0.2993 | 0.0006 | 0.18 |
| 110 | ref | -20 | 0.3006 | 0.2981 | 0.3029 | 0.3006 | 0.0024 | 0.79 |
| 111 | ref | -20 | 0.3191 | 0.2928 | 0.2947 | 0.3022 | 0.0146 | 4.85 |
| 114 | 1 | 4 | 0.3012 | 0.3002 | 0.2990 | 0.3001 | 0.0011 | 0.37 |
| 115 | 1 | 4 | 0.3068 | 0.3092 | 0.2994 | 0.3051 | 0.0051 | 1.67 |
| 116 | 3 | 4 | 0.3066 | 0.3029 | 0.3083 | 0.3059 | 0.0027 | 0.90 |
| 117 | 3 | 4 | 0.3078 | 0.3068 | 0.3111 | 0.3086 | 0.0022 | 0.73 |
| 118 | 6 | 4 | 0.3070 | 0.3022 | 0.3079 | 0.3057 | 0.0031 | 1.01 |
| 119 | 6 | 4 | 0.3018 | 0.3034 | 0.3160 | 0.3070 | 0.0078 | 2.53 |
| 120 | 12 | 4 | 0.3044 | 0.3023 | 0.3117 | 0.3061 | 0.0049 | 1.61 |
| 121 | 12 | 4 | 0.3127 | 0.2755 | 0.3035 | 0.2973 | 0.0194 | 6.51 |
| 124 | 1 | 22 | 0.2955 | 0.2977 | 0.2991 | 0.2974 | 0.0018 | 0.61 |
| 125 | 1 | 22 | 0.3057 | 0.2937 | 0.2969 | 0.2988 | 0.0062 | 2.08 |
| 126 | 3 | 22 | 0.2928 | 0.2955 | 0.2980 | 0.2954 | 0.0026 | 0.87 |
| 127 | 3 | 22 | 0.2950 | 0.3022 | 0.2920 | 0.2964 | 0.0053 | 1.77 |
| 128 | 6 | 22 | 0.2967 | 0.2892 | 0.3086 | 0.2982 | 0.0098 | 3.28 |
| 129 | 6 | 22 | 0.2985 | 0.3023 | 0.2977 | 0.2995 | 0.0024 | 0.81 |
| 130 | 12 | 22 | 0.2873 | 0.2881 | 0.2957 | 0.2904 | 0.0047 | 1.60 |
| 131 | 12 | 22 | 0.2996 | 0.2946 | 0.2890 | 0.2944 | 0.0053 | 1.80 |
| 134 | 1 | 40 | 0.2862 | 0.2939 | 0.2821 | 0.2874 | 0.0060 | 2.08 |
| 135 | 1 | 40 | 0.2962 | 0.2844 | 0.2833 | 0.2880 | 0.0071 | 2.47 |
| 136 | 3 | 40 | 0.2764 | 0.2814 | 0.2824 | 0.2801 | 0.0032 | 1.15 |
| 137 | 3 | 40 | 0.2827 | 0.2822 | 0.2854 | 0.2834 | 0.0017 | 0.61 |
| 138 | 6 | 40 | 0.2766 | 0.2734 | 0.2751 | 0.2751 | 0.0016 | 0.58 |
| 139 | 6 | 40 | 0.2806 | 0.2803 | 0.2756 | 0.2788 | 0.0028 | 1.01 |
| 140 | 12 | 40 | 0.2568 | 0.2546 | 0.2647 | 0.2587 | 0.0053 | 2.05 |
| 141 | 12 | 40 | 0.2590 | 0.2625 | 0.2554 | 0.2590 | 0.0036 | 1.37 |
| 142 | 1 | 60 | 0.2435 | 0.2359 | 0.2390 | 0.2394 | 0.0038 | 1.60 |
| 143 | 1 | 60 | 0.2659 | 0.2680 | 0.2628 | 0.2656 | 0.0026 | 0.99 |
| 145 | 3 | 60 | 0.2201 | 0.2222 | 0.2139 | 0.2187 | 0.0043 | 1.99 |
| 146 | 3 | 60 | 0.2033 | 0.2115 | 0.2105 | 0.2084 | 0.0045 | 2.14 |
| 147 | 6 | 60 | 0.2054 | 0.1991 | 0.1980 | 0.2008 | 0.0040 | 1.97 |
| 148 | 6 | 60 | 0.2071 | 0.2060 | 0.2020 | 0.2050 | 0.0027 | 1.31 |

# ANNEX C

## Results of the interlaboratory certification study

PCB-28

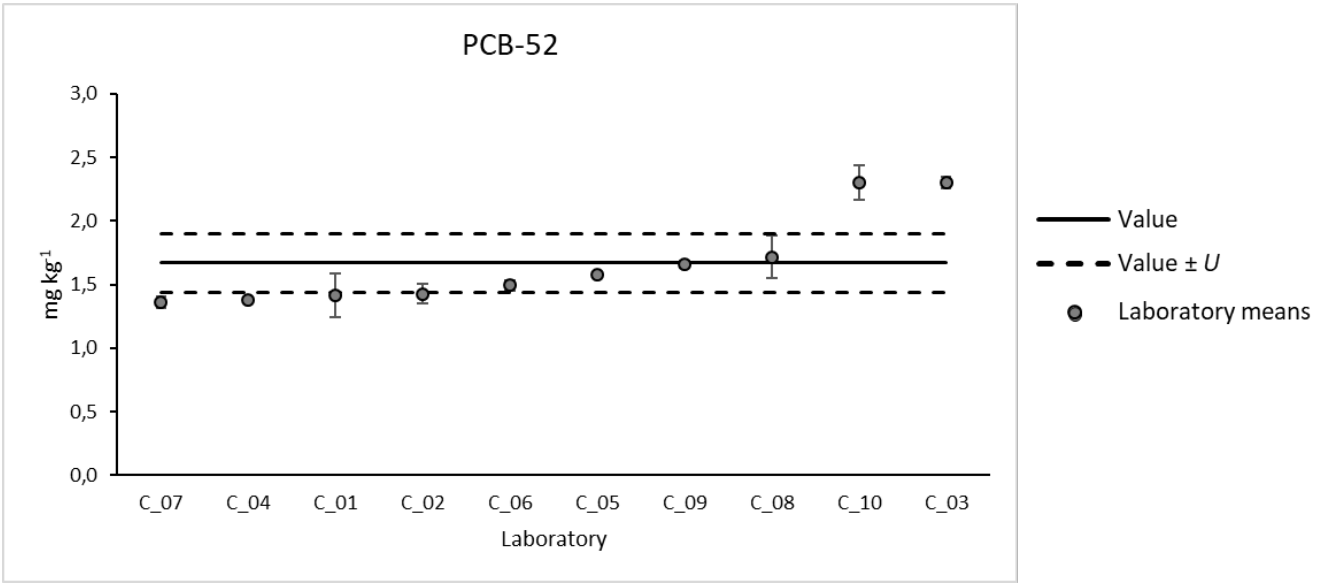
| Laboratory | Replicate determinations (mg $kg^{-1}$) | | | | | | Mean (mg $kg^{-1}$) | Standard deviation (mg$kg^{-1}$) |
|---|---|---|---|---|---|---|---|---|
| C01 | 0.13 | 0.17 | 0.12 | 0.13 | 0.14 | 0.15 | 0.14 | 0.018 |
| C02 | 0.16 | 0.16 | 0.14 | 0.16 | 0.14 | 0.14 | 0.15 | 0.011 |
| C03 | 0.21 | 0.20 | 0.20 | 0.22 | 0.21 | 0.21 | 0.21 | 0.008 |
| C04 | 0.18 | 0.18 | 0.18 | 0.18 | 0.19 | 0.18 | 0.18 | 0.003 |
| C05 | 0.14 | 0.14 | 0.13 | 0.13 | 0.14 | 0.14 | 0.14 | 0.002 |
| C06 | 0.14 | 0.14 | 0.14 | 0.14 | 0.14 | 0.13 | 0.14 | 0.003 |
| C07 | 0.10 | 0.10 | 0.11 | 0.10 | 0.10 | 0.10 | 0.10 | 0.002 |
| C08 | 0.17 | 0.18 | 0.15 | 0.15 | 0.17 | 0.15 | 0.16 | 0.013 |
| C09 | 0.15 | 0.15 | 0.15 | 0.14 | 0.15 | 0.15 | 0.15 | 0.004 |
| C10 | 0.21 | 0.19 | 0.21 | 0.21 | | | 0.20 | 0.015 |



## Certification study

PCB-52

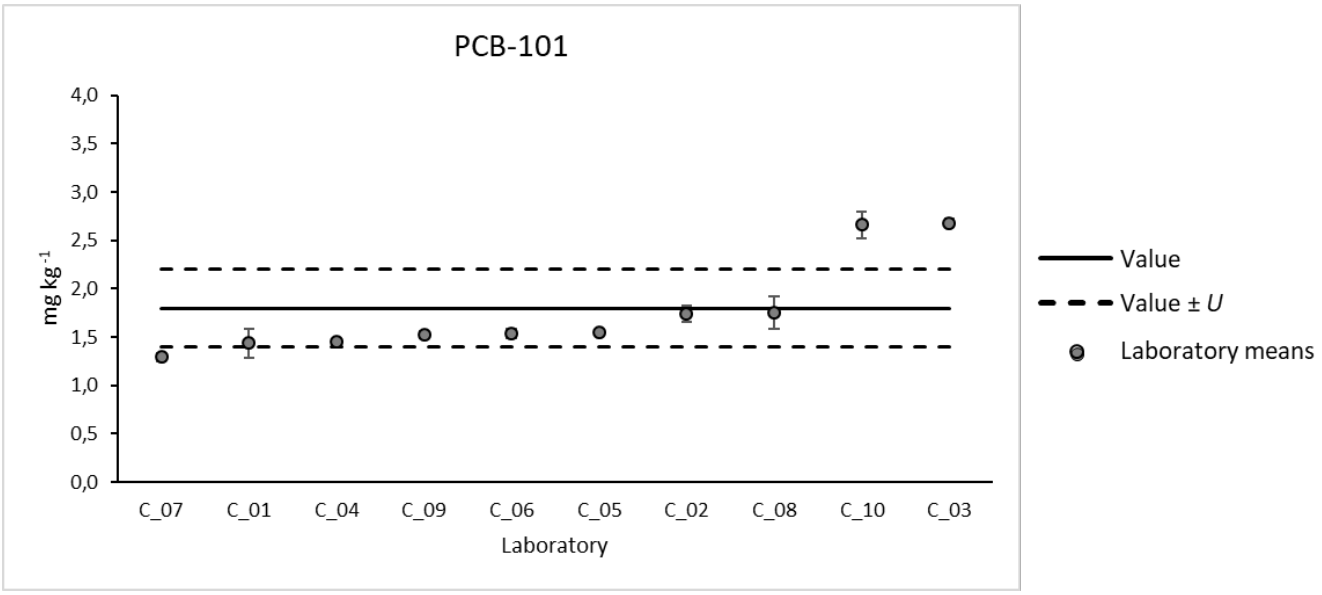
| Laboratory | Replicate determinations (mg kg$^{-1}$) | | | | | | Mean (mg kg$^{-1}$) | Standard deviation (mg kg$^{-1}$) |
|---|---|---|---|---|---|---|---|---|
| C01 | 1.26 | 1.72 | 1.35 | 1.27 | 1.43 | 1.48 | 1.42 | 0.172 |
| C02 | 1.42 | 1.44 | 1.31 | 1.38 | 1.46 | 1.54 | 1.43 | 0.077 |
| C03 | 2.30 | 2.35 | 2.23 | 2.30 | 2.32 | 2.34 | 2.30 | 0.044 |
| C04 | 1.35 | 1.39 | 1.39 | 1.39 | 1.36 | 1.37 | 1.37 | 0.017 |
| C05 | 1.59 | 1.56 | 1.57 | 1.58 | 1.57 | 1.60 | 1.58 | 0.015 |
| C06 | 1.56 | 1.50 | 1.50 | 1.50 | 1.47 | 1.43 | 1.49 | 0.043 |
| C07 | 1.31 | 1.34 | 1.33 | 1.43 | 1.35 | 1.41 | 1.36 | 0.048 |
| C08 | 1.73 | 1.75 | 1.53 | 1.53 | 1.97 | 1.80 | 1.72 | 0.169 |
| C09 | 1.69 | 1.66 | 1.64 | 1.62 | 1.65 | 1.68 | 1.66 | 0.028 |
| C10 | 2.38 | 2.14 | 2.38 | 2.37 | | | 2.30 | 0.135 |



## Certification study

PCB-101

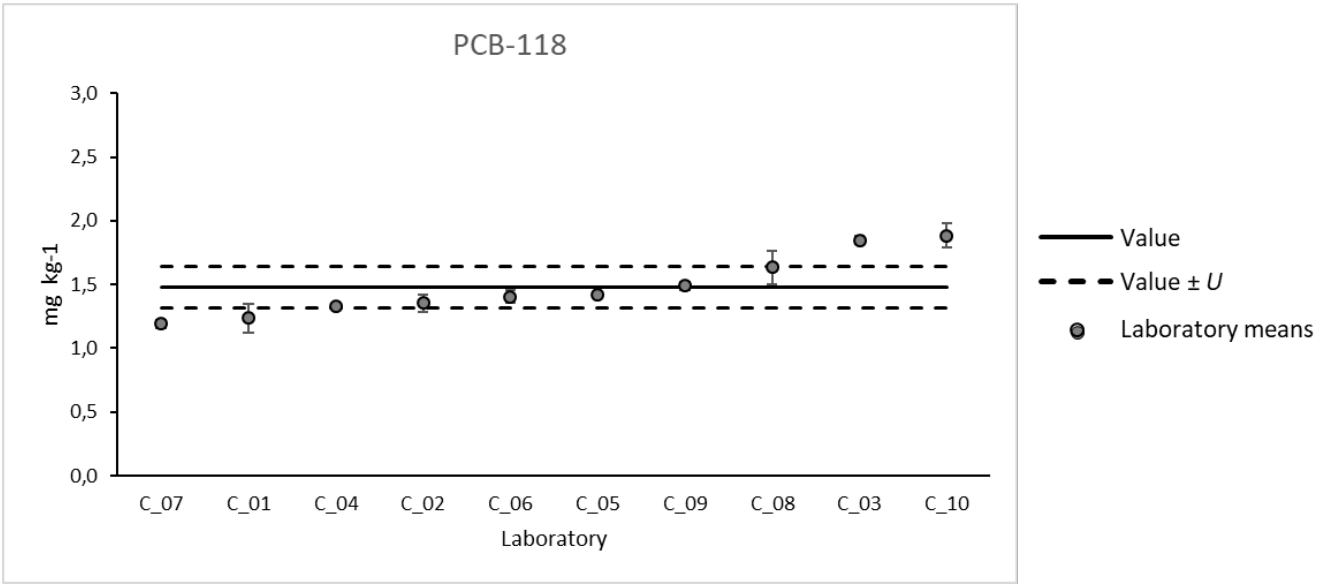
| Laboratory | Replicate determinations (mg kg$^{-1}$) | | | | | | Mean (mg kg$^{-1}$) | Standard deviation (mg kg$^{-1}$) |
|---|---|---|---|---|---|---|---|---|
| C01 | 1.28 | 1.69 | 1.39 | 1.31 | 1.50 | 1.46 | 1.44 | 0.15 |
| C02 | 1.75 | 1.75 | 1.60 | 1.71 | 1.77 | 1.85 | 1.74 | 0.08 |
| C03 | 2.67 | 2.73 | 2.61 | 2.68 | 2.70 | 2.69 | 2.68 | 0.04 |
| C04 | 1.47 | 1.44 | 1.43 | 1.45 | 1.47 | 1.45 | 1.45 | 0.01 |
| C05 | 1.53 | 1.51 | 1.55 | 1.59 | 1.55 | 1.56 | 1.55 | 0.03 |
| C06 | 1.61 | 1.54 | 1.55 | 1.55 | 1.52 | 1.46 | 1.54 | 0.05 |
| C07 | 1.27 | 1.31 | 1.24 | 1.37 | 1.30 | 1.27 | 1.29 | 0.05 |
| C08 | 1.69 | 1.72 | 1.58 | 1.60 | 1.98 | 1.93 | 1.75 | 0.17 |
| C09 | 1.57 | 1.53 | 1.51 | 1.47 | 1.50 | 1.54 | 1.52 | 0.03 |
| C10 | 2.76 | 2.51 | 2.72 | 2.77 | | | 2.66 | 0.14 |



## Certification study

PCB-118

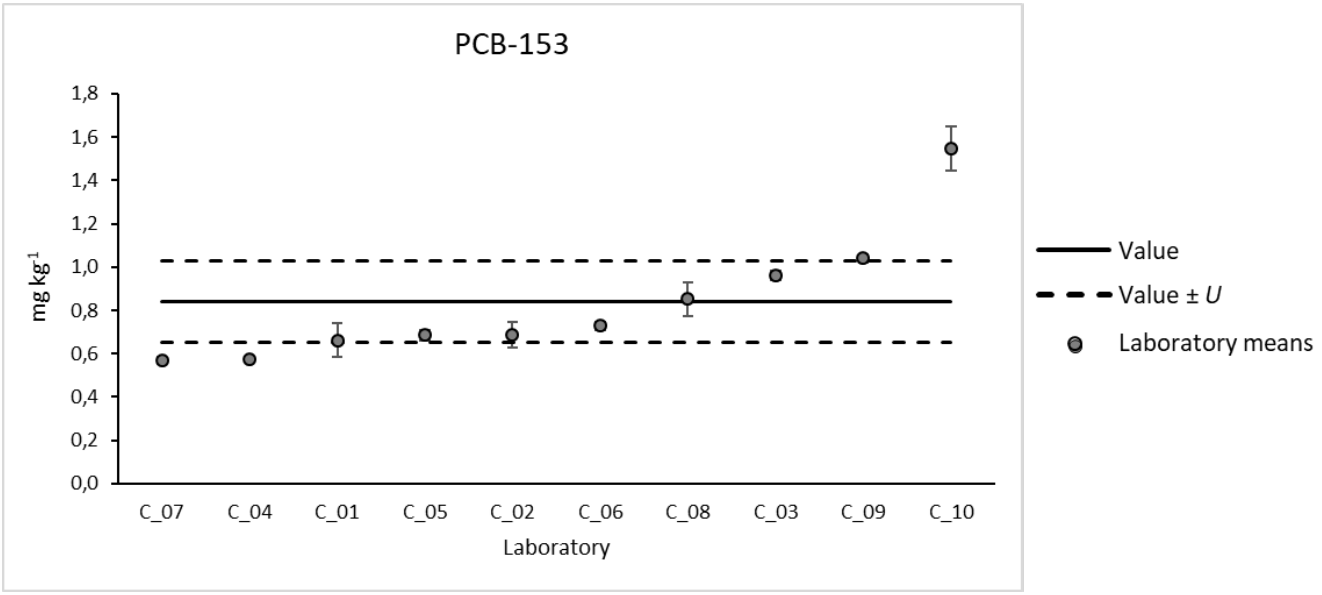
| Laboratory | Replicate determinations (mg kg$^{-1}$) | | | | | | Mean (mg kg$^{-1}$) | Standard deviation (mg kg$^{-1}$) |
|---|---|---|---|---|---|---|---|---|
| C01 | 1.13 | 1.42 | 1.19 | 1.13 | 1.28 | 1.29 | 1.24 | 0.113 |
| C02 | 1.35 | 1.34 | 1.25 | 1.33 | 1.39 | 1.46 | 1.35 | 0.069 |
| C03 | 1.83 | 1.89 | 1.81 | 1.84 | 1.88 | 1.85 | 1.85 | 0.031 |
| C04 | 1.35 | 1.35 | 1.34 | 1.31 | 1.34 | 1.31 | 1.33 | 0.019 |
| C05 | 1.42 | 1.40 | 1.41 | 1.43 | 1.41 | 1.43 | 1.42 | 0.010 |
| C06 | 1.48 | 1.42 | 1.42 | 1.40 | 1.37 | 1.34 | 1.41 | 0.048 |
| C07 | 1.15 | 1.23 | 1.16 | 1.23 | 1.18 | 1.19 | 1.19 | 0.034 |
| C08 | 1.57 | 1.59 | 1.53 | 1.53 | 1.86 | 1.73 | 1.63 | 0.132 |
| C09 | 1.53 | 1.49 | 1.48 | 1.44 | 1.48 | 1.52 | 1.49 | 0.031 |
| C10 | 1.97 | 1.78 | 1.91 | 1.85 | | | 1.89 | 0.096 |



## Certification study

PCB-153

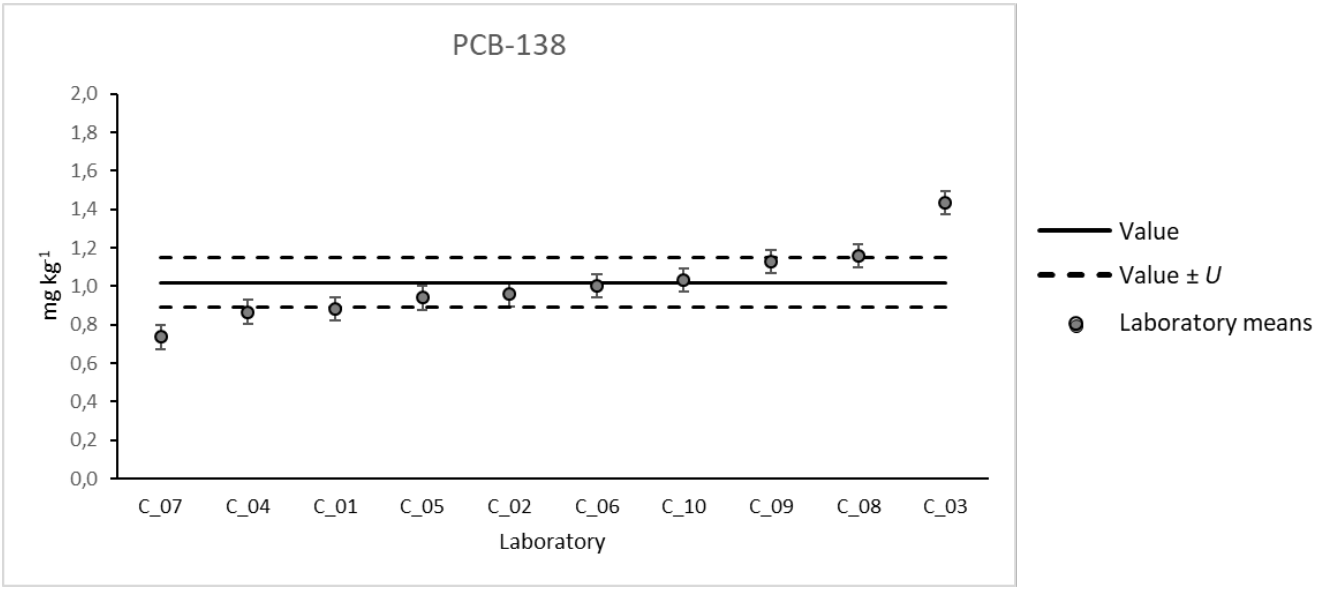
| Laboratory | Replicate determinations (mg kg$^{-1}$) | | | | | | Mean (mg kg$^{-1}$) | Standard deviation (mg kg$^{-1}$) |
|---|---|---|---|---|---|---|---|---|
| C01 | 0.57 | 0.79 | 0.65 | 0.60 | 0.69 | 0.67 | 0.66 | 0.077 |
| C02 | 0.72 | 0.72 | 0.61 | 0.67 | 0.63 | 0.77 | 0.69 | 0.062 |
| C03 | 0.96 | 0.99 | 0.93 | 0.95 | 0.96 | 0.97 | 0.96 | 0.020 |
| C04 | 0.57 | 0.56 | 0.59 | 0.57 | 0.58 | 0.57 | 0.57 | 0.009 |
| C05 | 0.73 | 0.66 | 0.67 | 0.69 | 0.67 | 0.68 | 0.69 | 0.025 |
| C06 | 0.75 | 0.71 | 0.75 | 0.72 | 0.73 | 0.71 | 0.73 | 0.020 |
| C07 | 0.56 | 0.55 | 0.59 | 0.56 | 0.56 | 0.59 | 0.57 | 0.016 |
| C08 | 0.80 | 0.82 | 0.77 | 0.83 | 0.97 | 0.93 | 0.85 | 0.077 |
| C09 | 1.06 | 1.03 | 1.02 | 1.02 | 1.05 | 1.06 | 1.04 | 0.019 |
| C10 | 1.61 | 1.43 | 1.59 | 1.61 | 0.00 | 0.00 | 1.55 | 0.100 |
| C11 | 0.57 | 0.79 | 0.65 | 0.60 | 0.69 | 0.67 | 0.66 | 0.077 |
| C12 | 0.72 | 0.72 | 0.61 | 0.67 | 0.63 | 0.77 | 0.69 | 0.062 |
| C13 | 0.96 | 0.99 | 0.93 | 0.95 | 0.96 | 0.97 | 0.96 | 0.020 |
| C14 | 0.57 | 0.56 | 0.59 | 0.57 | 0.58 | 0.57 | 0.57 | 0.009 |
| C15 | 0.73 | 0.66 | 0.67 | 0.69 | 0.67 | 0.68 | 0.69 | 0.025 |
| C16 | 0.75 | 0.71 | 0.75 | 0.72 | 0.73 | 0.71 | 0.73 | 0.020 |



## Certification study

PCB-138

| Laboratory | Replicate determinations (mg kg$^{-1}$) | | | | | | Mean (mg kg$^{-1}$) | Standard deviation (mg kg$^{-1}$) |
|---|---|---|---|---|---|---|---|---|
| C01 | 0.78 | 1.04 | 0.84 | 0.77 | 0.93 | 0.93 | 0.88 | 0.104 |
| C02 | 0.98 | 0.98 | 0.96 | 0.92 | 0.95 | 0.98 | 0.96 | 0.023 |
| C03 | 1.44 | 1.45 | 1.41 | 1.44 | 1.45 | 1.42 | 1.43 | 0.017 |
| C04 | 0.88 | 0.86 | 0.87 | 0.87 | 0.85 | 0.86 | 0.87 | 0.012 |
| C05 | 0.93 | 0.93 | 0.94 | 0.94 | 0.94 | 0.96 | 0.94 | 0.012 |
| C06 | 1.05 | 1.01 | 1.01 | 1.01 | 0.98 | 0.96 | 1.00 | 0.031 |
| C07 | 0.73 | 0.74 | 0.74 | 0.74 | 0.73 | 0.74 | 0.74 | 0.005 |
| C08 | 1.15 | 1.15 | 1.11 | 1.14 | 1.24 | 1.17 | 1.16 | 0.042 |
| C09 | 1.16 | 1.11 | 1.11 | 1.11 | 1.13 | 1.15 | 1.13 | 0.023 |
| C10 | 1.06 | 0.95 | 1.09 | 1.07 | 0.00 | 0.00 | 1.03 | 0.074 |



## Certification study

PCB-180

| Laboratory | Replicate determinations (mg kg$^{-1}$) | | | | | | Mean (mg kg$^{-1}$) | Standard deviation (mg kg$^{-1}$) |
|---|---|---|---|---|---|---|---|---|
| C01 | 0.19 | 0.24 | 0.21 | 0.19 | 0.23 | 0.21 | 0.21 | 0.022 |
| C02 | 0.20 | 0.21 | 0.19 | 0.21 | 0.22 | 0.21 | 0.21 | 0.010 |
| C03 | 0.29 | 0.26 | 0.29 | 0.29 | 0.27 | 0.26 | 0.28 | 0.014 |
| C04 | 0.20 | 0.20 | 0.20 | 0.20 | 0.19 | 0.19 | 0.19 | 0.004 |
| C05 | 0.22 | 0.20 | 0.21 | 0.21 | 0.21 | 0.21 | 0.21 | 0.006 |
| C06 | 0.21 | 0.20 | 0.20 | 0.20 | 0.19 | 0.19 | 0.20 | 0.008 |
| C07 | 0.10 | 0.12 | 0.10 | 0.11 | 0.12 | 0.11 | 0.11 | 0.006 |
| C08 | 0.21 | 0.21 | 0.21 | 0.20 | 0.25 | 0.23 | 0.22 | 0.017 |
| C09 | 0.22 | 0.22 | 0.21 | 0.22 | 0.22 | 0.23 | 0.22 | 0.004 |