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Certification of Mass Fractions of Polycyclic Aromatic Hydrocarbons, Organochlorines and Polybrominated Diphenyl Ethers in IAEA-459 Marine Sediment Sample

IAEA
International Atomic Energy Agency

CERTIFICATION OF MASS FRACTIONS OF POLYCYCLIC AROMATIC HYDROCARBONS, ORGANOCHLORINES AND POLYBROMINATED DIPHENYL ETHERS IN IAEA-459 MARINE SEDIMENT SAMPLE

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INTERNATIONAL ATOMIC ENERGY AGENCY
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FOREWORD

One of the major programmes of the IAEA Environment Laboratories is to assist Member States in the understanding, monitoring and protection of both terrestrial and marine environments. To assess the impact of land and sea based pollution sources on marine coastal environments, it is imperative to ensure the quality and comparability of the analytical data generated by national and regional pollution monitoring programmes. Since the early 1970s, the IAEA has assisted national laboratories and regional laboratory networks through the production of certified reference materials, training in quality assurance and evaluation of measurement performances by organizing worldwide and regional interlaboratory comparison exercises and proficiency tests.

This publication describes the production of certified reference material IAEA-459, which is produced following ISO Guides 34:2009 and 35:2006. This certified reference material is a sediment sample with certified mass fractions of polycyclic aromatic hydrocarbons, organochlorines and polybrominated diphenyl ethers. The assigned final values and their associated uncertainties were derived from robust statistics on the results provided by selected laboratories with demonstrated technical and quality competence, following the guidance given in the ISO Guides. The material is used for quality control and assessment of method performance for a number of organic analytes listed in the Stockholm Convention on Persistent Organic Pollutants as well as other pollutants listed as priority substances included in many environment monitoring programmes.

The IAEA is grateful to the Government of Monaco for its support and wishes to thank all laboratories and participants who took part in the characterization study of this reference material, in particular the Korea Institute of Ocean Science and Technology for the donation of the raw material. The IAEA officers responsible for this publication were I. Tolosa, R. Cassi and D. Huertas of the IAEA Environment Laboratories.

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1. INTRODUCTION

The Marine Environmental Studies Laboratory (MESL) of IAEA-EL provides assistance to Member States' laboratories to enhance the quality of the analytical measurement results, in trace elements and organic contaminants in the marine environmental samples. This is achieved through the production of certified reference materials, organization of interlaboratory comparisons and proficiency tests, and by conducting training courses on the analysis of contaminants in marine samples. This activity is undertaken in the framework of the EL's subprogramme 2.4.1 "Reference Products for Science and Trade" and the Project 2.4.1.1 "Provision of Reference Products and Assurance of Laboratory Performance".

Certified Reference Materials (CRMs) are valuable tools for analytical method development and validation to improve the measurements and quality control in analytical laboratories. More specifically, marine matrix CRMs are needed to ensure the reliability of the analytical measurements and ensure the use of high quality data as the basis for decision making in national or regional marine pollution monitoring programmes. Furthermore, since the Stockholm Convention (SC) on persistent organic pollutants (POPs) was adopted 15 years ago, a great number of different analytical methodologies have been extensively developed and there is great need of CRMs for these contaminants. While there are several CRMs certified for organic contaminants, there is still a noticeable lack of matrix CRMs, in particular for POPs in marine sediments, where the concentrations levels are in the low range of $\mu g\ kg^{-1}$. To meet this need, MESL has developed a sediment CRM for the determination of a great number of organic analytes listed as Persistent Organic Pollutants (POPs) by the Stockholm Convention as well as other POPs listed as priority substances (PSs), such as polycyclic aromatic hydrocarbons included in many environment monitoring programmes.

This report describes and provides information on the sample preparation methodology followed and on the assignment of property values with their associated uncertainties for a number of persistent organic contaminants in a marine sediment sample. Certification of the mass fractions was made for major POPs, including polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), chlorinated pesticides and polybrominated diphenyl ethers (PBDEs). Results on aliphatic hydrocarbons and other minor POPs are also provided as information values.

The CRM IAEA-459 was produced to satisfy the needs of laboratories to strengthen data quality assurance in the analysis of POPs and other priority substances in marine samples

2. METHODOLOGY

2.1. COLLECTION AND PREPARATION OF THE MATERIAL

A sample of marine sediment was collected in Han River estuary, South Korea. This sample was dried, ground and sieved at 125 μm. The powder obtained, about 26 kg, was homogenized by mixing it in a stainless steel rotating drum for three weeks. Then, aliquots of about 50 grams were packaged into cleaned amber glass bottles with aluminium screw caps, labeled IAEA-459 and sealed with Teflon tape. This material was previously used in a worldwide interlaboratory comparison (ILC) exercise performed in 2012, where 82 laboratories from 43 countries reported data on organochlorine compounds, polybrominated diphenyl ethers and petroleum hydrocarbons [1].

2.2. SELECTION OF LABORATORIES FOR THE CHARACTERIZATION STUDY

The selection of participating laboratories was based on the results they have provided during a previous ILC for the same compounds in sediment. Only laboratories that provided reliable and satisfactory data by applying valid quality control and quality assurance procedures were invited to participate in this characterization study.

One bottle of sediment sample was sent to each participating laboratory, accompanied by an information sheet and a reporting form to include the data results for chlorinated pesticides, polychlorinated biphenyls (PCBs), polybrominated diphenyl ethers (PBDEs), polycyclic aromatic hydrocarbons (PAHs) and aliphatic hydrocarbons. The systematic numbering of PCB and PBDE congeners is listed respectively in Appendixes X and XI. Six replicate aliquots were requested to be analysed using their usual technique, and the reported results had to be accompanied with the description of the method used together with the applied quality control procedures, including results for the organic contaminants in a CRM with a matrix similar to the candidate reference material.

The laboratories participating in the characterization study are listed on page 129.

2.3. HOMOGENEITY ASSESSMENT

In order to establish the degree of homogeneity of the reference material with respect to the properties of interest, both within- and between-unit homogeneity have to be evaluated to ensure that all distributed units are the same within the stated uncertainty [2].

Homogeneity test was performed by MESL after the bottling of the sample material. The between bottle homogeneity of the material was assessed by determining the concentration of selected organochlorine pesticides, polychlorinated biphenyls, polybrominated diphenyl

ethers and petroleum hydrocarbons in sample aliquots of 6 g taken from 10 bottles (about 3% of the total batch) randomly selected during the whole bottling process of the bulk dry powder. Each bottle unit was extracted and analysed without subsampling. Homogeneity assessment based on the analysis of variance (ANOVA) to calculate between-unit variation (s_{bb}) and within bottle heterogeneity (s_{wb}) could therefore not be applied [2], but the ANOVA-like approach [3, 4] was followed. The within-bottle homogeneity of the material was assessed by determinations of the concentration of organochlorine (OC) pesticides, polychlorinated biphenyls, polybrominated diphenyl ethers and petroleum hydrocarbons in six sample aliquots of 6 g taken from 1 bottle. The same method of analysis was used to test the between-bottle and within-bottle homogeneity. Samples were run in a random order to avoid the instrumental analysis sequence following the same order of preparation.

2.4. STABILITY STUDY

Stability information is important to determine the presence of any potential degradation of the analytes during sample transport to the customers (short-term stability) as well as conditions for storage (long-term stability) of the material. Time, temperature, moisture and radiation are usually the more pertinent parameters affecting stability of the samples. The influence of light was minimized by using amber bottles, which were stored in the dark and moisture was minimized by the drying preparation of the material. Consequently, only the influence of time and temperature were investigated by using an isochronous stability design.

To test stability, selected units are exposed to different storage conditions during different periods of time. Then, the units are transferred to conditions where additional potential degradation is considered extremely unlikely (reference conditions). At the end of the isochronous storage, the units are measured under repeatability conditions in a single run, providing the best available precision for the stability study.

2.4.1. Short-term stability

The approach used to assess the transportation stability was based on an isochronous design over 4 weeks. For that, a set of 4 units were stored in the dark at +20°C immediately after the bottling and another set of 4 bottles at +40°C for 1, 2, 3 and 4 weeks respectively. After the planned exposure time for each unit, were moved to reference conditions (-20°C temperature). One independent measurement per bottle was performed under repeatability conditions. The obtained results were compared with the results from samples kept at -20°C from the time zero of this study. The tests were performed by measuring phenanthrene, fluoranthene, pyrene by GC-MS and PCB 138, PCB 153, pp'-DDE and BDE-99 by GC-ECD. All measurements were performed under repeatability conditions, with the purpose to improve the measurement precision and consequently the power of the isochronous stability study.

2.5. CHARACTERIZATION

Characterization refers to the process of assigning a property value that can be reliably assessed when its value is confirmed by several laboratories working independently and using different methods, for each of which the accuracy has been well established [5]. The material was initially analysed in the IAEA/NAEL in Monaco. The final characterization was based on the results provided by selected laboratories with demonstrated technical and quality competence

The characterization of the PAHs was performed by using three different analytical techniques: gas chromatography/mass spectrometry (GC-MS), gas chromatography/high resolution mass spectrometry (GC-HRMS) and high performance liquid chromatography/fluorescence detector (HPLC–FLD) as summarized in Figure 1.

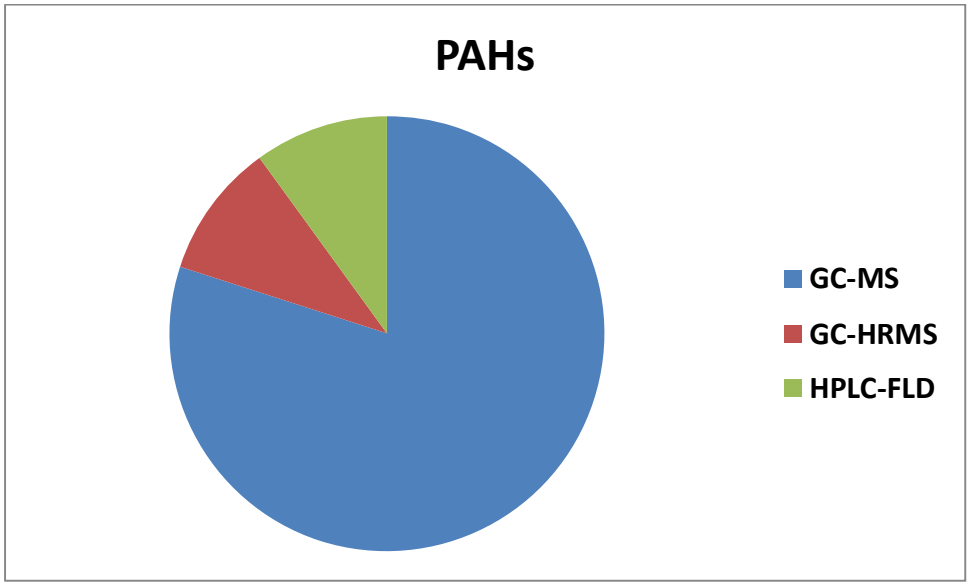


Fig. 1. Analytical methods used for the characterization of PAHs in the IAEA-459 sediment sample. Abbreviations used to describe the instrumental techniques are given in Table 1.

The characterization of the PCBs was based on the application of five different analytical techniques: two-dimensional gas chromatography/electron capture detector (GCxGC-ECD), gas chromatography coupled to tandem mass spectrometry (GC-MS/MS), GC-MS, GC-HRMS and gas chromatography/electron capture detector (GC-ECD) as summarized in Figure 2.

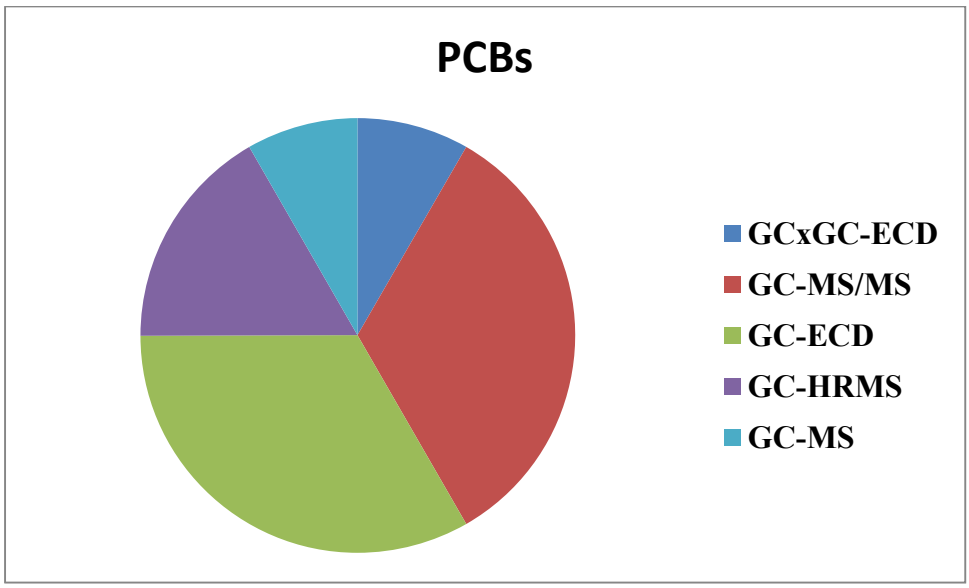


Fig. 2. Analytical methods used for the characterization of PCBs in the IAEA-459 sediment sample. Abbreviations used to describe the instrumental techniques are given in Table 1.

For organochlorine (OC) pesticides, the material was characterized by using four different analytical techniques: (GCxGC-ECD), GC-MS/MS, GC-HRMS and GC-ECD as illustrated in Figure 3.

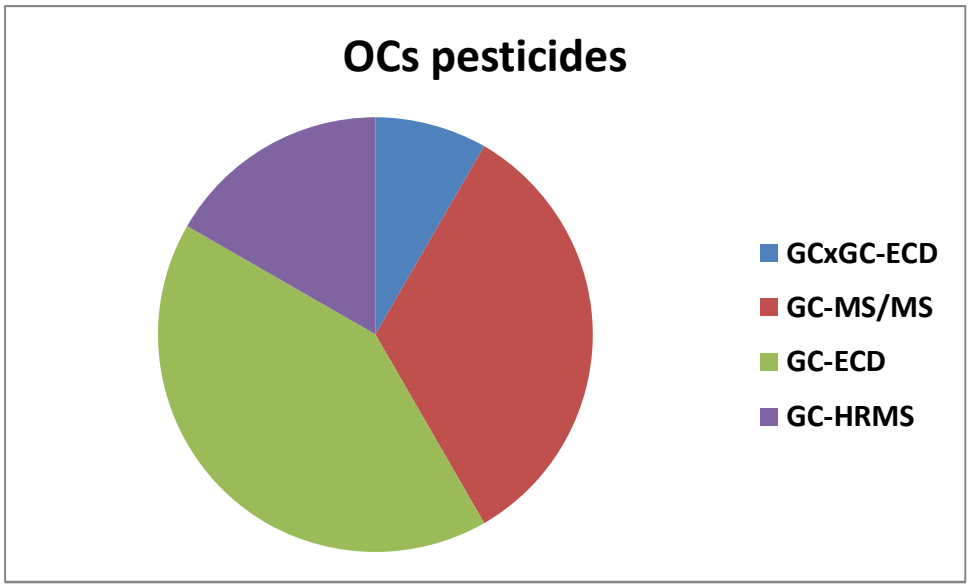


Fig. 3. Analytical methods used for the characterization of OC pesticides in the IAEA-459 sediment sample. Abbreviations used to describe the instrumental techniques are given in Table 1.

The characterization of the PBDEs was based on the application of four different analytical techniques: gas chromatography/mass spectrometry by electron impact (GC-MS-EI), gas chromatography/mass spectrometry by negative ion chemical ionization (GC-MS-NICI), GC-HRMS and GC-ECD as summarized in Figure 4.

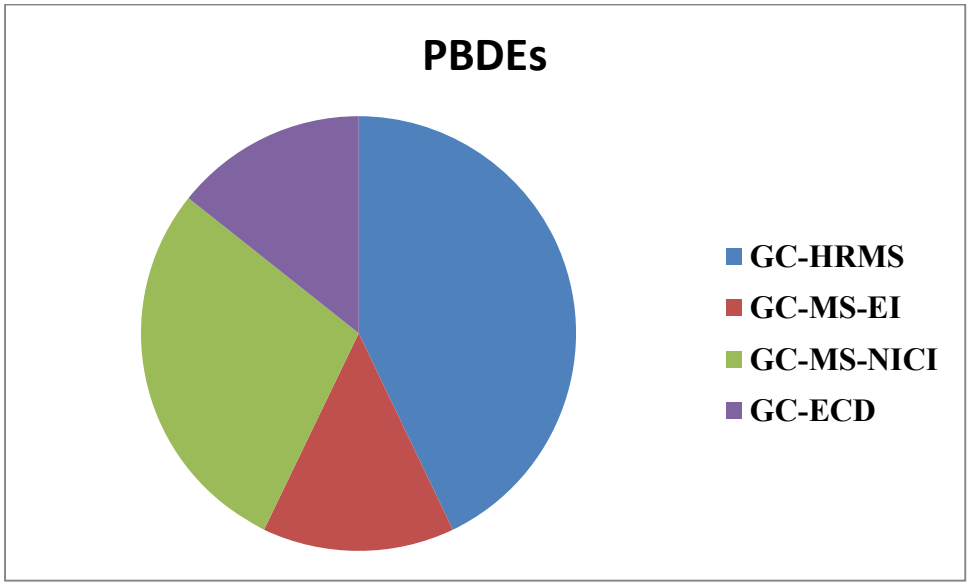


Fig. 4. Analytical methods used for the characterization of PBDEs in the IAEA-459 sediment sample. Abbreviations used to describe the instrumental techniques are given in Table 1.

TABLE 1. INSTRUMENTAL TECHNIQUES

Method code	**Instrumental technique**
GC-MS	Gas chromatography/mass spectrometry
GC-HRMS	Gas chromatography/high resolution mass spectrometry
HPLC–FLD	High performance liquid chromatography/fluorescence detector
GCxGC-ECD	Two-dimensional gas chromatography/electron capture detector
GC-MS/MS	Gas chromatography coupled to tandem mass spectrometry
GC-ECD	Gas chromatography/electron capture detector
GC-MS-EI	Gas chromatography/mass spectrometry by electron impact
GC-MS-NICI	Gas chromatography/mass spectrometry by negative ion chemical ionization

Laboratories participating in the characterization campaign provided their results with their method validation data in accordance with the guidelines of ISO/IEC 17025. The number of independent datasets obtained for PAHs, organochlorinated compounds and PBDEs was

respectively 10, 12 and 7. The basic principles for evaluation of measurement uncertainty were followed according to the ISO Guide 35 [2] and the Guide to the Expression of Uncertainty in Measurement (GUM) [6], which combines the different uncertainties of characterization, inhomogeneity and instability.

2.6. MOISTURE CONTENT

The moisture content of the lyophilized material, determined by drying an aliquot to a constant mass at 105°C, was found to be 1.7±0.1 % at the time of the preparation of this sample and 2.8 ±0.1% at the time of this characterization exercise.

2.7. ELEMENTAL COMPOSITION AND ISOTOPES VALUES

Total carbon and nitrogen percent composition as well as carbon and nitrogen isotopic composition ($\delta^{13}C$ and $\delta^{15}N$) were measured on a Thermo Fisher Flash 2000 elemental analyzer coupled to a Delta V Advantage isotope ratio mass spectrometer. The values are shown in Table 2.

TABLE 2. ELEMENTAL COMPOSITION AND STABLE ISOTOPE COMPOSITION OF THE IAEA-459

	% Amount (by dry weight)	Delta Value (‰)
Total Carbon	2.62 ± 0.01	-20.77 ± 0.07
Nitrogen	0.21 ± 0.01	5.25 ± 0.04

3.RESULTS AND DISCUSSION

3.1. RESULTS OF THE HOMOGENEITY STUDY

3.1.1. Between-unit homogeneity

To assess the uncertainty related to the inhomogeneity, 10 bottle units (about 3% of the total batch) of sediment samples were selected by using a random stratified sample picking scheme and analysed for their organic contaminants under repeatability conditions. Each bottle unit was extracted and analysed without subsampling. Homogeneity assessment based on the analysis of variance (ANOVA) to calculate between-unit variation (s_{bb}) and within-bottle heterogeneity (s_{wb}) could therefore not be applied [2], but the ANOVA-like approach was applied, according to Linsinger et al , 2001 [3] and Van der Veen et al., 2001 [4], as shown in Eq. 1:

$$u_{c,bb}^2 = s_{bb}^2 + s_{meas}^2 \quad (1)$$

which implies that

$$s_{bb}^2 = u_{c,bb}^2 - s_{meas}^2 \quad (2)$$

where $u_{c,bb}$ is the combined uncertainty of the between-unit experiment, expressed as the uncertainty on a single unit; s_{bb} is the variation between units and s_{meas} is the intrinsic variability of the method (s_{method}) divided by the square root of n, the number of replicates per unit; s_{bb} is the estimate of between-bottle variation in the material, named u_{hom}.

Tables 3, 4 and 5 summarize the estimates of inhomogeneity contributions to the total uncertainty and the number of outliers at 95% and 99% confidence levels by using Grubbs tests. Only outliers at 99% confidence level were removed for the statistical analysis.

TABLE 3. THE ESTIMATE OF INHOMOGENEITY CONTRIBUTIONS TO THE TOTAL UNCERTAINTY FOR THE SELECTED PARENT PAHs COMPOUNDS

Compounds	outliers 95%	outliers 99%	$u_{c,bb}$ %	s_{meas} %	u_{hom} %
Naphthalene	2	0	10.6	7.4	7.5
Biphenyl	0	0	10.9	3.6	10.3
Acenaphthylene	3	3	4.2	5.2	5.2*
Fluorene	0	0	9.7	2.6	9.4
Acenaphthene	1	1	4.8	2.3	4.3
Dibenzothiophene	2	0	3.9	2.0	3.4
Phenanthrene	0	0	5.5	1.3	5.3
Fluoranthene	1	0	2.6	1.2	2.3
Pyrene	1	1	2.0	1.1	1.6
Chrysene+triphenylene	0	0	4.6	2.2	4.0
Benzo(b+j)fluoranthene	2	1	5.6	2.5	5.0
Benzo(a)pyrene	2	1	4.3	1.3	4.1
Indeno[1,2,3-c,d]pyrene	1	1	6.4	3.3	5.5
Benzo(g,h,i)perylene	2	1	4.3	2.2	3.6
Perylene	0	0	4.7	4.0	2.5

* taken as s_{meas} because $s_{bb}<s_{meas}$

TABLE 4. THE ESTIMATE OF INHOMOGENEITY CONTRIBUTIONS TO THE TOTAL UNCERTAINTY FOR THE SELECTED PCBs AND PBDEs

Compounds	outliers 95%	outliers 99%	$u_{c,bb}$ %	s_{meas} %	u_{hom} %
PCB 8	1	1	18.8	2.6	18.7
PCB 18	0	0	36.2*	2.9	36.1*
PCB 31	2	2	11.4	2.1	11.2
PCB 28	2	1	9.1	2.0	8.9
PCB 52	0	0	11.7	1.4	11.7
PCB 49	1	0	7.5	1.2	7.4
PCB 44	0	0	17.5	1.2	17.5
PCB 66	0	0	3.5	1.5	3.1
PCB 101	0	0	4.1	0.9	4.0
PCB 99	0	0	4.0	0.5	3.9
PCB 97	1	0	4.4	2.0	3.9
PCB 87	0	0	4.4	1.1	4.3
PCB110	0	0	4.1	0.7	4.1
PCB 151	0	0	6.3	2.1	5.9
PCB 149	0	0	3.2	0.7	3.2
PCB 118	0	0	3.9	0.7	3.9
PCB 153	0	0	3.8	1.2	3.6
PCB 105	0	0	6.1	1.9	5.8
PCB 138	0	0	3.6	0.9	3.5
PCB 187	2	0	4.7	1.0	4.6
PCB 183	0	0	5.6	1.8	5.3
PCB 128	0	0	6.1	1.8	5.9
PCB 174	0	0	4.7	1.3	4.5
PCB 177	0	0	8.6	2.2	8.3
PCB 156	0	0	6.4	5.4	3.5
PCB 201	0	0	10.0	3.8	9.2
PCB 180	0	0	3.7	1.4	3.5
PCB 170	0	0	4.9	1.3	4.8
PCB 195	0	0	9.6	1.5	9.5
PCB 194	0	0	6.0	1.4	5.8
PCB 206	0	0	15.1	1.5	15.1
PCB 209	0	0	15.0	3.1	14.7
BDE 47	0	0	10.8	4.1	10.0
BDE 99	0	0	10.0	3.1	9.5

*the high $u_{c,bb}$ for PCB 18 is probably due to some interference; therefore its $u_{homogeneity}$ wil be set at 18.7% as PCB 8.

TABLE 5. THE ESTIMATE OF INHOMOGENEITY CONTRIBUTIONS TO THE TOTAL UNCERTAINTY FOR THE SELECTED ORGANOCHLORINE PESTICIDES.

Compounds	OUTLIERS 95%	outliers 99%	$u_{c,bb}$ %	s_{meas} %	u_{hom} %
α HCH	3	0	15.9	5.3	15.0
HCB	1	1	18.0	2.3	17.8
β HCH	0	0	10.4	3.5	9.8
γ HCH (Lindane)	0	0	7.0	5.4	4.5
op DDE	0	0	4.1	2.3	3.4
pp'DDE	0	0	3.3	1.3	3.0
op DDD	2	0	8.4	1.5	8.3
pp'DDD	0	0	7.0	2.1	6.7
op DDT	0	0	13.3	7.1	11.3
pp'DDT	0	0	12.9	5.9	11.5

The coefficient of variation for the content of the major analytes between the 10 different sample bottles was below 10%. Thus the material was considered sufficiently homogeneous for the parent PAHs, the organochlorinated and PBDEs compounds at 6 g sample size. The uncertainty of inhomogeneity for alkylated PAHs not included in the homogeneity study was set at the same percentage as their respective parent PAH compounds; for the PBDEs not included in the homogeneity study was set at the same percentage as the selected PBDEs (BDE 47 and BDE 99).

3.1.2. Within-unit homogeneity

For within-bottle homogeneity studies, a similar approach as between-unit homogeneity might be developed, but the intrinsic variability of the method (s_{method}) can not be determined independently because a similar material with perfect within-unit homogeneity does not exist [4]. In this respect, we can assume that the within-bottle homogeneity can be set to the intrinsic variability of the method (s_{meas}) which is shown in Tables 3, 4 and 5. This variability was measured by using a sample size of 6 g. However, to assess the minimum sample intake that is representative for the entire unit and can be used in the analysis of the target compounds, we used the method information provided by the laboratories contributing to this characterization study. We set the minimum sample intake as the lowest sample intake that provided data results within the accepted assigned values and stated uncertainty. In this study, they were set at 3 g, 1 g and 2 g, respectively for PAHs, OCs and PBDEs. However, taking in account the low concentration levels of the target analytes, we recommend a minimum sample size of 3 g for all families of POPs.

3.2. RESULTS FOR STABILITY STUDY

3.2.1. Short-term stability study

The samples selected for short-term stability study were analysed and selected organic contaminants (phenanthrene, fluoranthene, pyrene, PCB 138, PCB 153, pp'-DDE and BDE-99) were evaluated individually for each temperature (20°C and 40°C). Measurements were performed under repeatability conditions and linear regression lines were calculated for each selected analyte and tested for significance on a 95% confidence level using a Student t-test for slope significantly different from zero. No slope associated with any of the linearity plots generated for each selected analyte was detected that differed significantly from zero. As results did not show any significant trend of degradation over the timeframe at different temperatures +20°C and +40°C, no special precautions regarding temperature control during transport are needed. These results are supported by the chemical nature of the persistent organic contaminants which owe a high chemical stability and persistence. Therefore, no additional uncertainty with respect to instability due to transport needs to be taken into account and the uncertainty associated with short-term stability under transport conditions is taken as zero.

3.2.2. Long-term stability study

Long-term stability evaluation aims to determine if the certified values of the analyte(s) remain valid during the 5 to 10 year lifetime of the certified reference material. Based on experience with similar materials issued by the IAEA and statements of expiry dates on certificates of other CRM providers such as NIST, the material is expected to be stable for 5-10 years, provided that it will be stored in the dark at temperatures below 30°C [7, 8].

As no measurements on instability was determined over long term storage conditions, the uncertainty for long-term stability was set as the intrinsic variability of the method $s_{meas,}$ which is shown in Tables 3 for PAHs and Tables 4 and 5 for PCBs, OCs and PBDEs. The long-term stability test will be monitored at regular intervals and changes, which may occur, will be reflected in an updated certificate of analysis provided to the users.

3.3. DETERMINATION OF ASSIGNED VALUES AND UNCERTAINTIES

The determination of the assigned values and its standard uncertainty for organic contaminants in the IAEA-459 sample were derived using the robust statistics approach, which provide high resistance to the influence of extreme outlying values [2]. The robust values of the average and standard deviation of the data was calculated using the Algorithm A provided in the Annex C.1 from the ISO standard 13528 [9].

First, the set of *p* individual results were ranked in increasing order by (x_1, x_2, x_i,…, x_p). Then, the median x* of the data was calculated as:

x^* = median of x_i (i=1,2,3….p) (3)

The robust standard deviation of the data set was calculated:

$s^* = 1.483 \times \text{median } |x_i - x^*|$ (i=1,2,3…..p) (4)

The final "cut-off" values used in the robust algorithm was estimated as:

$$\delta = 1.5 \times s^* \quad (5)$$

For each x_i, it was calculated:

$$x_i^* = \begin{cases} x_i^* = x^* - \delta, & \text{if } x_i < x^* - \delta \\ x_i^* = x^* + \delta, & \text{if } x_i > x^* + \delta \\ x_i^* = x^*, & \text{otherwise} \end{cases} \quad (6)$$

New values for the robust mean x* and s^* were updated as follows:

$$x^* = \frac{\sum_1^p x_i^*}{p} \quad (7)$$

$$s^* = 1.134 \sqrt{\frac{\Sigma\,(x_i{*} - x^*)^2}{(p-1)}} \quad (8)$$

where the summation is over *i*

The robust estimates x^* and s^* were derived by an iterative calculation by updating the values of x* and s* several times using the modified data, until the process converged to the third significant figure of the robust standard deviation and robust average.

These estimate values were similar to other robust estimates of the mean calculated with the algorithms offered by the Analytical Methods Committee of the Royal Society of Chemistry (AMC) [10, 11].

The uncertainties associated with the assigned property values were conducted according to ISO Guide 35 [2]. The relative combined uncertainty of the assigned property value of the CRM involved combining the standard uncertainties associated with the characterization (u_{char}), homogeneity (u_{hom}), short term stability (u_{short}) and long-term stability (u_{stab}). Because the uncertainty component derived from the short term stability was insignificant and assumed to be zero, the final expanded uncertainty was a combination of the other three different contributions using the law of propagation of uncertainty as shown in Eq. 9:

$$U = k \times \sqrt{u_{char}^2 + u_{stab}^2 + u_{hom}^2} \quad (9)$$

Where k is the coverage factor of 2, representing a confidence level of 95%

u_{hom} was calculated as described in section 3.1.1.

u_{stab} was calculated as described in section 3.2.2.

u_{char} was calculated as described in ISO 13528 [8] using Eq. 10:

$$u_{char} = 1.25 \times \frac{s^*}{\sqrt{p}} \qquad (10)$$

Where: s^* is the robust standard deviation calculated from Eq. 8 and p is the number of participating laboratories.

The final assigned values derived by robust mean are shown in Tables 6, 7, 8 and 9 together with their individual and final uncertainty budget.

TABLE 6. ROBUST MEANS AND RELATIVE UNCERTAINTIES FOR PAHs (µg kg^{-1} dry mass)

	No. Results	x* µg kg^{-1}	u_{char} (%)	u_{hom} (%)	u_{stab} (%)	U_{rel} (k=2) (%)
Naphthalene	10	20.9	19.1	7.5	7.4	44
2-Methylnaphthalene	8	15.5	14.0	7.5	1.9	32
1-Methylnaphthalene	8	9.2	17.9	7.5	1.5	39
C2-Naph	5	55.4	27.2	7.5	2.1	57
C3-Naph	5	66.1	19.5	7.5	2.4	42
Biphenyl	3	10.5	6.2	10.3	3.6	25
Acenaphthylene	9	3.2	19.4	5.2	5.2	41
Fluorene	10	4.7	18.1	9.4	2.6	41
Acenaphthene	10	1.78	20.0	4.3	2.3	41
C1-Fluorenes	2	11.1	--	9.4	2.5	--
C2-Fluorenes	2	21.9	--	9.4	2.3	--
C3-Fluorenes	2	30.1	--	9.4	2.3	--
Dibenzothiophene	6	9.4	8.5	3.4	2.0	19
C1-Dibenzothiophenes	4	35.0	13.6	3.4	2.1	28
C2-Dibenzothiophene	4	62.8	18.3	3.4	0.9	37
C3-Dibenzothiophene	4	98.8	20.3	3.4	1.5	41
Phenanthrene	10	33.9	6.9	5.3	1.3	18
Anthracene	10	6.0	5.2	5.3	2.6	16
1methylphenanthrene	6	7.7	26.0	5.3	1.8	53
2methylphenanthrene	5	20.3	27.4	5.3	1.6	56
C1- Phen/Anth	5	45.2	22.4	5.3	1.1	46
C2- Phen/Anth	5	47.0	13.2	5.3	1.1	29
C3- Phen/Anth	4	39.1	8.9	5.3	0.9	21
C4- Phen/Anth	3	34.0	15.5	5.3	1.7	33
Fluoranthene	10	37.3	3.1	2.3	1.2	8
Pyrene	10	46.3	8.7	1.6	1.1	18
1methyl Pyr	3	8.8	4.1	2.3	3.2	11
C1-Fluor/Pyr	4	43.6	9.6	2.3	2.4	20
C2-Fluor/Pyr	4	49.1	6.0	2.3	2.4	14
C3-Fluoranthenes/pyrenes	2	36.0		2.3	2.4	
Benz(a)anthracene	10	19.3	10.2	4.0	1.8	22
Chrysene	4	18.4	6.8	4.0	2.2	16
Triphenylene	1	8.0		4.0		
C1-Chrysenes	4	34.9	3.4	4.0	4.3	14
C2-Chrysenes	3	50.0	8.2	4.0	2.2	19
C3-Chrysenes	3	39.7	4.7	4.0	1.8	13
Benzo(b)fluoranthene	5	44.1	8.9	5.0	2.5	21
Benzo(j)fluoranthene	3	20.4	25.1	5.0	2.5	51
Benzo(k)fluoranthene	9	19.0	12.8	5.0	3.0	28
Benzo(a)fluoranthene	3	7.0	35.2	5.0	2.6	71
Benzo(e)pyrene	6	35.9	14.9	4.1	5.8	33
Benzo(a)pyrene	10	22.7	8.4	4.1	1.3	19
Indeno[1,2,3-c,d]pyrene	9	35.6	13.9	5.5	3.3	31

TABLE 6. ROBUST MEANS AND RELATIVE UNCERTAINTIES FOR PAHs (µg kg^{-1} dry mass) (cont.)

	No. Results	x* µg kg^{-1}	u_{char} (%)	u_{hom} (%)	u_{stab} (%)	U_{rel} (k=2) (%)
Dibenz(a,h)anthracene	10	6.6	20.5	5.5	0.9	42
Benzo(g,h,i)perylene	10	35.5	15.4	3.6	2.2	32
Perylene	6	31.6	27.2	2.5	4.0	55
Chrysene+triphenylene	7	27.5	14.9	4.0	2.2	31
Benzo(b+j)Fl	8	58.8	11.3	5.0	2.5	25

TABLE 7. ROBUST MEANS AND RELATIVE UNCERTAINTIES FOR PCBs (µg kg^{-1} dry mass)

	No. Results	x* µg kg^{-1}	u_{char} (%)	u_{hom} (%)	u_{stab} (%)	U_{rel} (k=2) (%)
PCB 8	3	0.46	23.88	18.66	2.62	61
PCB 18	7	1.11	15.9	36.1	2.9	79
PCB 28	11	2.27	8.4	8.9	2.0	25
PCB 31	7	2.41	4.8	11.2	2.1	25
PCB 44	7	1.72	5.9	17.5	1.2	37
PCB 49	5	2.64	1.2	7.4	1.2	15
PCB 52	12	2.38	7.8	11.7	1.4	28
PCB 66	5	3.10	12.6	3.1	1.5	26
PCB 95	2	2.42	0.0	0.0	4.0	8.0
PCB 87	5	1.24	5.6	4.0	1.1	14
PCB 97	3	1.42	14.0	4.0	2.0	29
PCB 99	3	2.54	5.3	3.9	0.5	13
PCB 101	12	3.78	3.8	4.2	0.9	11
PCB 105	9	1.29	10.0	6.1	1.9	24
PCB 110	6	3.70	8.2	4.2	0.7	18
PCB 118	11	2.98	5.2	4.1	0.7	13
PCB 128	5	0.62	6.0	6.2	1.8	18
PCB 138	12	3.25	13.2	3.8	0.9	27
PCB 149	6	2.88	8.2	3.2	0.7	18
PCB 151	5	0.66	11.9	6.3	2.1	27
PCB 153	12	3.75	7.9	3.8	1.2	18
PCB 156	7	0.336	7.0	3.3	5.4	19
PCB 170	7	1.02	9.8	4.7	1.3	22
PCB 174	4	0.90	3.8	4.3	1.3	12
PCB 177	2	0.50	0.0	8.8	2.2	18
PCB 180	12	2.22	6.9	3.0	1.4	15
PCB 183	5	0.72	18.1	5.3	1.8	38
PCB 187	6	1.39	5.5	4.7	1.0	14
PCB 194	4	0.47	31.2	5.8	1.4	63
PCB 195	3	0.10	59.4	7.7	1.5	120
PCB 201	3	0.18	0.8	9.5	3.8	20
PCB 206	3	0.22	0.7	15.4	1.5	31
PCB 209	5	0.199	5.5	15.5	3.1	34

TABLE 8. ROBUST MEANS AND RELATIVE UNCERTAINTIES FOR ORGANOCHLORINATED PESTICIDES (μg kg^{-1} dry mass)

	No. Results	x* μg kg^{-1}	u_{char} (%)	u_{hom} (%)	u_{stab} (%)	U_{rel} (k=2) (%)
HCB	10	0.153	6.8	17.8	2.3	38
α HCH	7	0.145	16.8	15.0	5.3	46
β HCH	7	0.136	28.8	9.8	3.5	61
γ HCH- Lindane	7	0.182	16.0	4.5	5.4	35
pp' DDE	12	3.60	5.8	3.0	1.3	13
pp' DDD	12	3.00	13.8	6.8	2.1	31
pp' DDT	12	1.32	14.7	11.6	5.9	39
δ HCH	1	0.03	-	-	-	-
op DDE	7	0.47	11.0	3.6	2.3	24
op DDD	8	0.75	15.7	8.8	1.5	36
op DDT	7	0.35	11.4	12.1	7.1	36
Heptachlor	2	0.15	-	-	-	-
Aldrin	3	<0.10	-	-	-	-
Dieldrin	3	0.10	-	-	-	-
Endrin	3	<0.03	-	-	-	-
cis-Chlordane	4	0.05	-	-	-	-
trans-Chlordane	4	0.07	-	-	-	-
cis-Nonachlor	2	0.06	-	-	-	-
trans-Nonachlor	3	0.01	-	-	-	-
α Endosulfan	4	0.06	-	-	-	-
β Endosulfan	4	0.05	-	-	-	-
Endosulfan sulfate	4	0.05	-	-	-	-

TABLE 9. ROBUST MEANS AND RELATIVE UNCERTAINTIES FOR PBDEs (μg kg^{-1}dry mass)

	No. Results	x* μg kg^{-1}	u_{char} (%)	u_{hom} (%)	u_{stab} (%)	U_{rel} (k=2) (%)
BDE 28	7	0.021	18.6	10	4	43
BDE 47	7	0.177	13.1	10	4	34
BDE 66	5	0.010	21.5	10	4	48
BDE 85	5	0.009	29.9	10	4	64
BDE 99	7	0.240	9.0	10	4	28
BDE 100	7	0.029	9.2	10	4	28
BDE 153	7	0.097	2.9	10	4	22
BDE 154	7	0.025	22.2	10	4	49
BDE 183	7	0.282	4.1	10	4	23
BDE 209	7	10.8	7.9	10	4	27

The robust mean of the laboratory means were assigned as certified values, for those compounds where the assigned value was derived from at least five datasets and its relative expanded uncertainty was less than 40 % of the assigned value. The certified values for 18 PAHs, 22 PCBs, 6 organochlorinated pesticides and 5 PBDEs are summarized in Tables 10, 11, 12 and 13 together with their expanded uncertainty. For all these compounds, the values were derived from at least 3 different analytical techniques excepting, dibenzothiophene, benzo(e)pyrene, Chrysene+triphenylene, and Benzo(b+j)fluoranthene which were derived from two analytical techniques. Mass fractions of compounds that did not fulfill the criteria of certification are considered information values. Tables 14, 15, 16 and 17 shows the information values for 29 PAHs, 11 PCBs, 16 organochlorinated pesticides and 5 PBDEs together with the expanded uncertainty for the compounds that could be calculated. Appendix IX also shows some few datasets of aliphatic hydrocarbons for information values.

TABLE 10. CERTIFIED VALUES FOR PAHs MASS FRACTIONS AND THEIR EXPANDED UNCERTAINTY (*k=2*) IN THE IAEA-459 SEDIMENT SAMPLE

Compound	Unit	Certified value[1]	Expanded uncertainty (*k*=2) [2]
2-Methylnaphthalene	µg kg^{-1}	15.5	5.0
1-Methylnaphthalene	µg kg^{-1}	9.2	3.6
Acenaphthylene	µg kg^{-1}	3.2	1.3
Fluorene	µg kg^{-1}	4.7	1.9
Acenaphthene	µg kg^{-1}	1.78	0.73
Dibenzothiophene	µg kg^{-1}	9.4	1.8
Phenanthrene	µg kg^{-1}	33.9	6.0
Anthracene	µg kg^{-1}	6.0	1.0
Fluoranthene	µg kg^{-1}	37.3	3.0
Pyrene	µg kg^{-1}	46.3	8.3
Benz(a)anthracene	µg kg^{-1}	19.3	4.3
Chrysene+triphenylene	µg kg^{-1}	27.5	8.5
Benzo(b)fluoranthene	µg kg^{-1}	44.1	9.3
Benzo(b+j)fluoranthene	µg kg^{-1}	59	15
Benzo(k)fluoranthene	µg kg^{-1}	19.0	5.3
Benzo(e)pyrene	µg kg^{-1}	36	12
Benzo(a)pyrene	µg kg^{-1}	22.7	4.3
Indeno[1,2,3-c,d]pyrene	µg kg^{-1}	36	11
Benzo(g,h,i)perylene	µg kg^{-1}	36	11

[1] The value is the robust mean of accepted sets of data, each set being obtained by different laboratory. The certified values are reported on dry mass basis and are traceable to the SI.
[2] Expanded uncertainty with a coverage factor *k*=2 estimated in accordance with the JCGM 100:2008 Evaluation of measurement data – Guide to the expression of uncertainty in measurement [6], corresponding to the level of confidence of about 95%.

TABLE 11. CERTIFIED VALUES FOR PCBs MASS FRACTIONS AND THEIR EXPANDED UNCERTAINTY (*k=2*) IN THE IAEA-459 SEDIMENT SAMPLE

Compound	Unit	Certified value[1]	Expanded uncertainty (*k*=2) [2]
PCB 28	µg kg^{-1}	2.27	0.56
PCB 31	µg kg^{-1}	2.41	0.60
PCB 44	µg kg^{-1}	1.72	0.64
PCB 49	µg kg^{-1}	2.64	0.40
PCB 52	µg kg^{-1}	2.38	0.67
PCB 66	µg kg^{-1}	3.10	0.81
PCB 87	µg kg^{-1}	1.24	0.17
PCB 101	µg kg^{-1}	3.78	0.43
PCB 105	µg kg^{-1}	1.29	0.31
PCB 110	µg kg^{-1}	3.70	0.68
PCB 118	µg kg^{-1}	2.98	0.39
PCB 128	µg kg^{-1}	0.62	0.11
PCB 138	µg kg^{-1}	3.25	0.89
PCB 149	µg kg^{-1}	2.88	0.51
PCB 151	µg kg^{-1}	0.66	0.18
PCB 153	µg kg^{-1}	3.75	0.66
PCB 156	µg kg^{-1}	0.336	0.063
PCB 170	µg kg^{-1}	1.02	0.22
PCB 180	µg kg^{-1}	2.22	0.34
PCB 183	µg kg^{-1}	0.72	0.27
PCB 187	µg kg^{-1}	1.39	0.20
PCB 209	µg kg^{-1}	0.199	0.067

[1] The value is the robust mean of accepted sets of data, each set being obtained by different laboratory. The certified values are reported on dry mass basis and are traceable to the SI.

[2] Expanded uncertainty with a coverage factor *k*=2 estimated in accordance with the JCGM 100:2008 Evaluation of measurement data – Guide to the expression of uncertainty in measurement [6], corresponding to the level of confidence of about 95%.

TABLE 12. CERTIFIED VALUES FOR ORGANOCHLORINATED PESTICIDES MASS FRACTIONS AND THEIR EXPANDED UNCERTAINTY (*k=2*) IN THE IAEA-459 SEDIMENT SAMPLE

Compound	Unit	Certified value[1]	Expanded uncertainty (*k*=2) [2]
pp' DDE	µg kg^{-1}	3.60	0.48
pp' DDD	µg kg^{-1}	3.00	0.93
pp' DDT	µg kg^{-1}	1.32	0.52
op DDE	µg kg^{-1}	0.47	0.11
op DDD	µg kg^{-1}	0.75	0.27
op DDT	µg kg^{-1}	0.35	0.13

[1] The value is the robust mean of accepted sets of data, each set being obtained by different laboratory. The certified values are reported on dry mass basis and are traceable to the SI.
[2] Expanded uncertainty with a coverage factor *k*=2 estimated in accordance with the JCGM 100:2008 Evaluation of measurement data – Guide to the expression of uncertainty in measurement [6], corresponding to the level of confidence of about 95%.

TABLE 13. CERTIFIED VALUES FOR PBDEs MASS FRACTIONS AND THEIR EXPANDED UNCERTAINTY (*k=2*) IN THE IAEA-459 SEDIMENT SAMPLE

Compound	Unit	Certified value[1]	Expanded uncertainty (k=2) [2]
BDE 47	µg kg^{-1}	0.177	0.060
BDE 99	µg kg^{-1}	0.240	0.067
BDE 153	µg kg^{-1}	0.097	0.022
BDE 183	µg kg^{-1}	0.282	0.065
BDE 209	µg kg^{-1}	10.8	2.9

[1] The value is the robust mean of accepted sets of data, each set being obtained by different laboratory. The certified values are reported on dry mass basis and are traceable to the SI.
[2] Expanded uncertainty with a coverage factor *k*=2 estimated in accordance with the JCGM 100:2008 Evaluation of measurement data – Guide to the expression of uncertainty in measurement [6], corresponding to the level of confidence of about 95%.

TABLE 14. INFORMATION VALUES FOR PAHs MASS FRACTIONS AND THEIR EXPANDED UNCERTAINTY (*k=2*) IN THE IAEA-459 SEDIMENT SAMPLE

Compound	Unit	Information value[1]	Expanded uncertainty (k=2)[2]
Naphthalene	µg kg^{-1}	20.9	9.1
C2-Naphthalene	µg kg^{-1}	55	31
C3-Naphthalene	µg kg^{-1}	66	28
Biphenyl	µg kg^{-1}	10.5	2.6
C1-Fluorenes	µg kg^{-1}	11.1	--
C2-Fluorenes	µg kg^{-1}	21.9	--
C3-Fluorenes	µg kg^{-1}	30.1	--
C1-Dibenzothiophene	µg kg^{-1}	35.0	9.9
C2-Dibenzothiophene	µg kg^{-1}	63	23
C3-Dibenzothiophene	µg kg^{-1}	99	41
1methylphenanthrene	µg kg^{-1}	7.7	4.1
2methylphenanthrene	µg kg^{-1}	20	11
C1- Phen/Anth	µg kg^{-1}	45	21
C2- Phen/Anth	µg kg^{-1}	47	13
C3- Phen/Anth	µg kg^{-1}	39.1	8.1
C4- Phen/Anth	µg kg^{-1}	34	11
1methyl Pyrene	µg kg^{-1}	8.8	1.0
C1-Fluor/Pyrenes	µg kg^{-1}	43.6	8.9
C2-Fluor/Pyrenes	µg kg^{-1}	49.1	6.7
C3-Fluoranthenes/pyrenes	µg kg^{-1}	36.0	--
Chrysene	µg kg^{-1}	18.4	3.0
Triphenylene	µg kg^{-1}	8.0	--
C1-Chrysenes	µg kg^{-1}	34.9	4.7
C2-Chrysenes	µg kg^{-1}	50.0	9.4
C3-Chrysenes	µg kg^{-1}	39.7	5.1
Benzo(j)fluoranthene	µg kg^{-1}	20	11
Benzo(a)fluoranthene	µg kg^{-1}	7.0	5.0
Dibenz(a,h)anthracene	µg kg^{-1}	6.6	2.8
Perylene	µg kg^{-1}	32	18

[1] The value is the robust mean of accepted sets of data, each set being obtained by different laboratory.
[2] Expanded uncertainty with a coverage factor *k=2* estimated in accordance with the JCGM 100:2008 Evaluation of measurement data – Guide to the expression of uncertainty in measurement [6], corresponding to the level of confidence of about 95%.

TABLE 15. INFORMATION VALUES FOR PCBs MASS FRACTIONS AND THEIR EXPANDED UNCERTAINTY (*k=2*) IN THE IAEA-459 SEDIMENT SAMPLE

Compound	Unit	Information value[1]	Expanded uncertainty (*k*=2)[2]
PCB 8	µg kg^{-1}	0.46	0.28
PCB 18	µg kg^{-1}	1.11	0.53
PCB 95	µg kg^{-1}	2.42[3]	--
PCB 97	µg kg^{-1}	1.42	0.42
PCB 99	µg kg^{-1}	2.54	0.33
PCB 174	µg kg^{-1}	0.90	0.10
PCB 177	µg kg^{-1}	0.50[3]	--
PCB 194	µg kg^{-1}	0.47	0.30
PCB 195	µg kg^{-1}	0.10	0.12
PCB 201	µg kg^{-1}	0.184	0.038
PCB 206	µg kg^{-1}	0.204	0.062

[1] The value is the robust mean of accepted sets of data, each set being obtained by different laboratory.
[2] Expanded uncertainty with a coverage factor *k=2* estimated in accordance with the JCGM 100:2008 Evaluation of measurement data – Guide to the expression of uncertainty in measurement [6], corresponding to the level of confidence of about 95%.
[3] The value is the mean of the two values for different laboratories.

TABLE 16. INFORMATION VALUES FOR ORGANOCHLORINATED PESTICIDES MASS FRACTIONS AND THEIR EXPANDED UNCERTAINTY (*k=2*) IN THE IAEA-459 SEDIMENT SAMPLE

Compound	Unit	Information value[1]	Expanded uncertainty (*k*=2)[2]
HCB	µg kg^{-1}	0.153	0.058
α HCH	µg kg^{-1}	0.145	0.067
β HCH	µg kg^{-1}	0.136	0.083
γ HCH- Lindane	µg kg^{-1}	0.182	0.064
cis-Chlordane	µg kg^{-1}	0.05	--
trans-Chlordane	µg kg^{-1}	0.07	--
δ HCH	µg kg^{-1}	0.03	--
Heptachlor	µg kg^{-1}	0.15	--
Aldrin	µg kg^{-1}	<0.10	--
Dieldrin	µg kg^{-1}	0.10	--
Endrin	µg kg^{-1}	<0.03	--
cis-Nonachlor	µg kg^{-1}	0.06	--
trans-Nonachlor	µg kg^{-1}	0.01	--
α Endosulfan	µg kg^{-1}	0.06	--
β Endosulfan	µg kg^{-1}	0.05	--
Endosulfan sulfate	µg kg^{-1}	0.05	--

[1] The value is the robust mean of accepted sets of data, each set being obtained by different laboratory.
[2] Expanded uncertainty with a coverage factor *k=2* estimated in accordance with the JCGM 100:2008 Evaluation of measurement data – Guide to the expression of uncertainty in measurement [6], corresponding to the level of confidence of about 95%.

TABLE 17. INFORMATION VALUES FOR PBDEs MASS FRACTIONS AND THEIR EXPANDED UNCERTAINTY (*k=2*) IN THE IAEA-459 SEDIMENT SAMPLE

Compound	Unit	Information value[1]	Expanded uncertainty (k=2)[2]
BDE 28	μg kg^{-1}	0.0213	0.0092
BDE 66	μg kg^{-1}	0.0100	0.0048
BDE 85	μg kg^{-1}	0.0092	0.0058
BDE 100	μg kg^{-1}	0.0293	0.0083
BDE 154	μg kg^{-1}	0.0252	0.0124

[1] The value is the robust mean of accepted sets of data, each set being obtained by different laboratory.
[2] Expanded uncertainty with a coverage factor *k=2* estimated in accordance with the JCGM 100:2008 Evaluation of measurement data – Guide to the expression of uncertainty in measurement [6], corresponding to the level of confidence of about 95%.

4. METROLOGICAL TRACEABILITY AND COMMUTABILITY

Metrological traceability is defined as the property of a measurement result where the result is related to a reference through a documented unbroken chain of calibrations, each contributing to the measurement uncertainty [10]. The methods used by all participating laboratories were validated by using matrix standard reference materials (CRMs) from NIST (SRM1941b, SRM 1944), IAEA (IAEA-408, IAEA-159) and materials characterized by QUASIMEME proficiency tests (MS3 polycyclic aromatic hydrocarbons in sediment, MS2 chlorinated organics in sediment). The fact, that values reported by participants are based on calibration standard solutions of known purity, issued by accredited commercial companies with documented unbroken chain of calibrations, demonstrates that the assigned values derived from combining the individual results are traceable to International System of Unis (SI). Furthermore, the agreement between the results generated by different analytical methodologies ensures the comparability of the measurement results and validates the identity of the measurand.

Commutability is a property of an RM, demonstrated by the closeness of agreement between the relation among the measurement results for a stated quantity in this material, obtained according to two given measurement procedures, and the relation obtained among the measurement results for other specified materials [5].

A material is said to be commutable when the measurand in the routine test samples behaves similar as in the CRM with respect to the different measurement procedures. This implies that the application of the procedures to the CRM would produce the same quantitative value as normal routine samples containing the same concentration of the measurand. In this respect, IAEA-459 is a natural marine sediment sample and their analytical behaviour should be the same as for a routine sample of dried sediment samples. The agreement between the data acquired with different analytical procedures for the IAEA-459 characterization study endorses the absence of any significant method bias and shows commutability of the material for all certified organic compounds.

5. CONCLUSIONS

Combination of different data sets from at least two different analytical techniques has allowed the assignment of certified concentrations for 22 PCBs, 6 OC pesticides, 5 PBDEs

and 18 PAHs following the recommendation of ISO Guide 35. The extensive characterization at very low concentration levels and associated uncertainties will make CRM 459 a valuable sediment reference material for use in the validation of analytical methods for the determination of a great number of persistent organic contaminants listed at the Stockholm Convention as well as other persistent and priority substances (PSs), such as polycyclic aromatic hydrocarbons included within the environmental monitoring programs.

All available IAEA reference materials may be found in the Reference Material Online Catalogue, http://nucleus.iaea.org/rpst/ReferenceProducts/ReferenceMaterials.

APPENDIX I

RESULTS FOR THE CERTIFIED MEASUREMENTS OF PAHS

TABLE 18. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF 2-METHYLNAPHTHALENE REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM used for QC
4	9.25	0.48	1.00	GC-MS	SRM1941b
6	11.36	1.11	0.17	GC-MS/MS	IAEA-408
8	16.00	0.20	1.12	GC-MS	NIST 1944
9	13.73	0.33	<1	GC-MS	RM IAEA-159
10	15.06		0.50	GC-MS	IMR LRM
11	24.64	1.75	0.60	GC-HRMS	
13	15.56	0.51	0.10	GC-MS	NIST 1944
16	24.74	1.48	0.02	GC-MS	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported by participants.

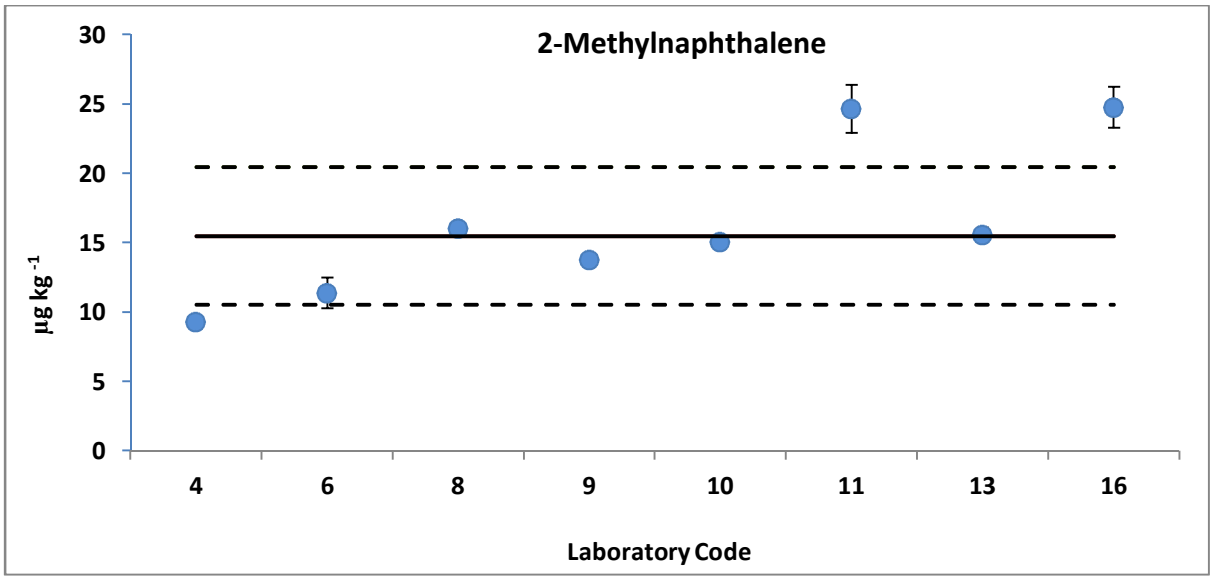


*FIG. 5. Laboratory results for 2-Methylnaphthalene in IAEA-459 (*µg kg^{-1}*).*

TABLE 19. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF 1-METHYLNAPHTHALENE REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
4	5.09	0.29	1.00	GC-MS	SRM1941b
6	4.72	0.55	0.10	GC-MS/MS	IAEA-408
8	9.83	0.07	0.89	GC-MS	NIST 1944
9	7.42	0.17	<1	GC-MS	RM IAEA-159
10	10.08		0.50	GC-MS	IMR LRM
11	13.98	1.12	0.60	GC-HRMS	
13	9.95	0.38	0.10	GC-MS	NIST 1944
16	12.44	0.70	0.02	GC-MS	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported by participants.

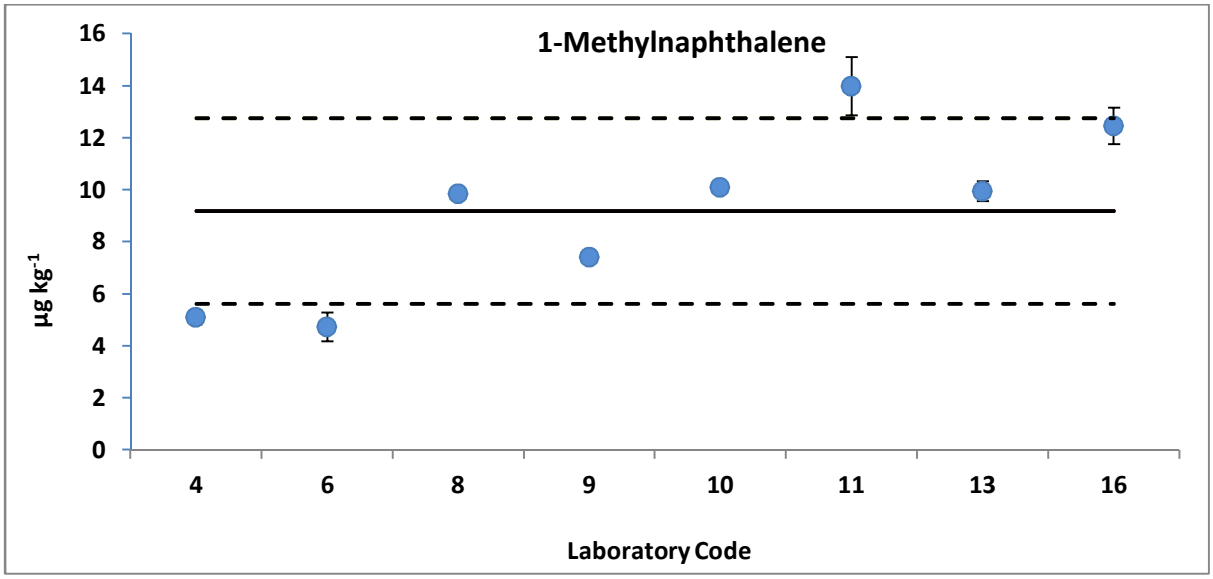


*FIG. 6. Laboratory results for 1-Methylnaphthalene in IAEA-459 (*µg kg^{-1}*).*

TABLE 20. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF ACENAPHTHYLENE REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
3	2.52		0.20	GC-MS	
4	1.75	0.11	2.00	GC-MS	SRM1941b
6	1.22	0.20	0.17	GC-MS/MS	IAEA-408
8	6.80	2.31	0.63	GC-MS	NIST 1944
9	3.01	0.17	<1	GC-MS	RM IAEA-159
10	2.66		0.50	GC-MS	IMR LRM
11	3.65	0.38	0.40	GC-HRMS	
13	3.88	0.66	0.10	GC-MS	NIST 1944
16	6.29	0.58	0.02	GC-MS	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported by participants.

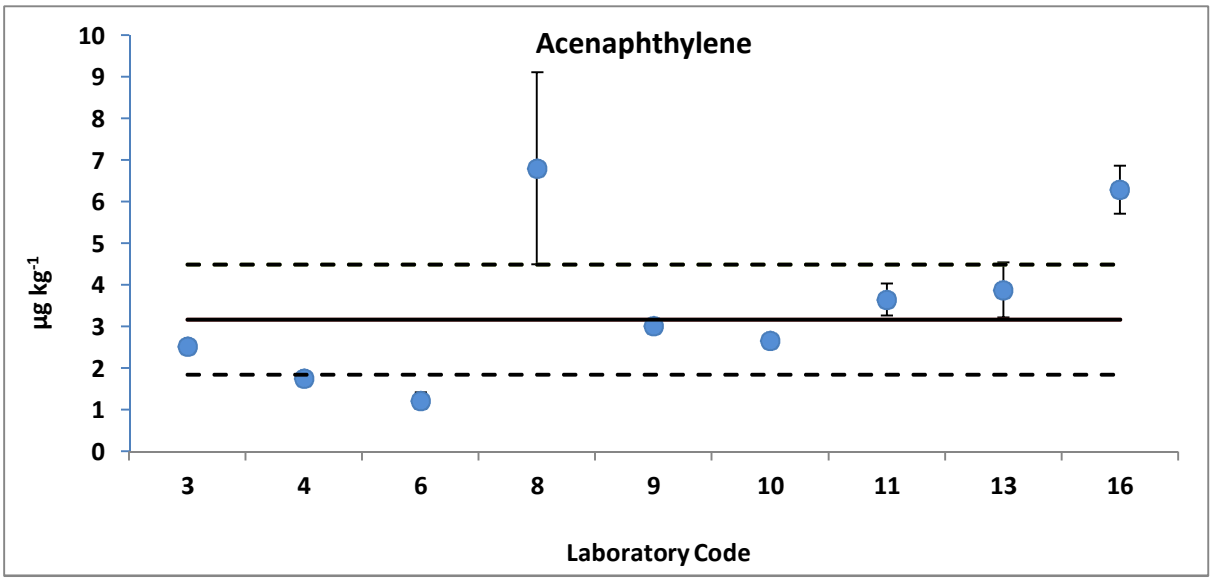


*FIG. 7. Laboratory results for Acenaphthylene in IAEA-459 (*µg kg^{-1}*).*

TABLE 21. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF FLUORENE REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
3	8.91		0.20	GC-MS	
4	2.42	0.12	2.00	GC-MS	SRM1941b
6	4.32	0.22	0.18	GC-MS/MS	IAEA-408
7	2.77	0.41	0.50	HPLC-FLD	Quasimeme QPH087
8	7.77	0.52	0.71	GC-MS	NIST 1944
9	3.81	0.28	<1	GC-MS	RM IAEA-159
10	4.95		0.50	GC-MS	IMR LRM
11	3.00	0.35	0.50	GC-HRMS	
13	4.59	0.35	0.10	GC-MS	NIST 1944
16	5.95	0.41	0.02	GC-MS	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

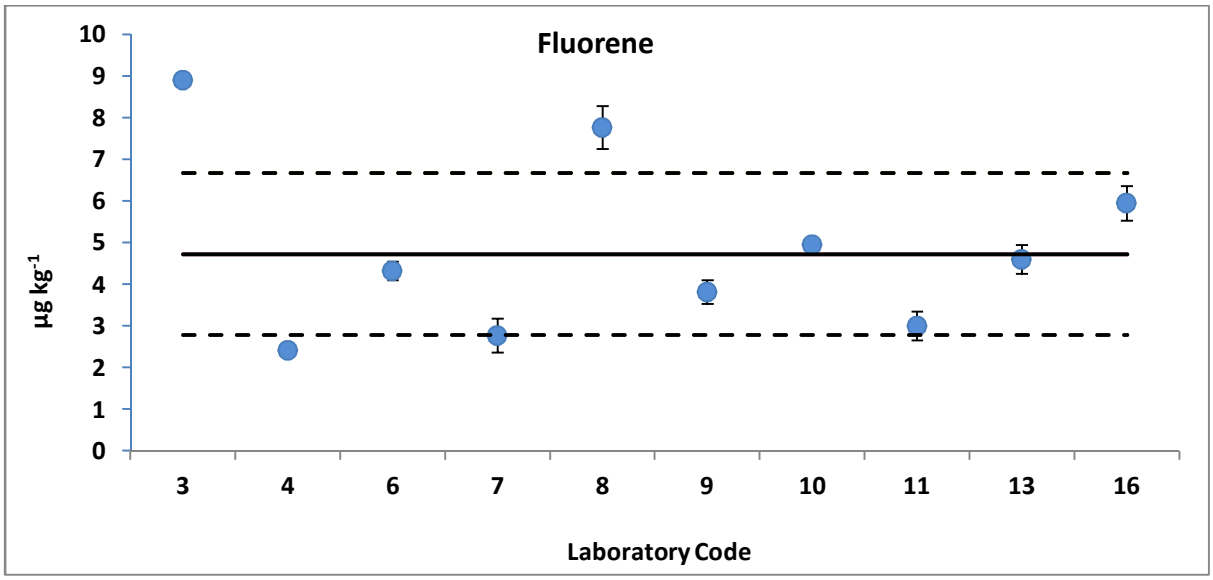


*FIG. 8. Laboratory results for Fluorene in IAEA-459 (*µg kg^{-1}*).*

TABLE 22. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF ACENAPHTHENE REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
3	2.72		0.20	GC-MS	
4	0.99	0.06	2.00	GC-MS	SRM1941b
6	0.59	0.15	0.21	GC-MS/MS	IAEA-408
7	0.98	0.14	0.50	HPLC-FLD	Quasimeme QPH087
8	2.43	0.18	0.54	GC-MS	NIST 1944
9	1.55	0.07	<1	GC-MS	RM IAEA-159
10	1.42		0.50	GC-MS	IMR LRM
11	2.81	0.30	0.40	GC-HRMS	
13	1.92	0.18	0.10	GC-MS	NIST 1944
16	2.44	0.11	0.02	GC-MS	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

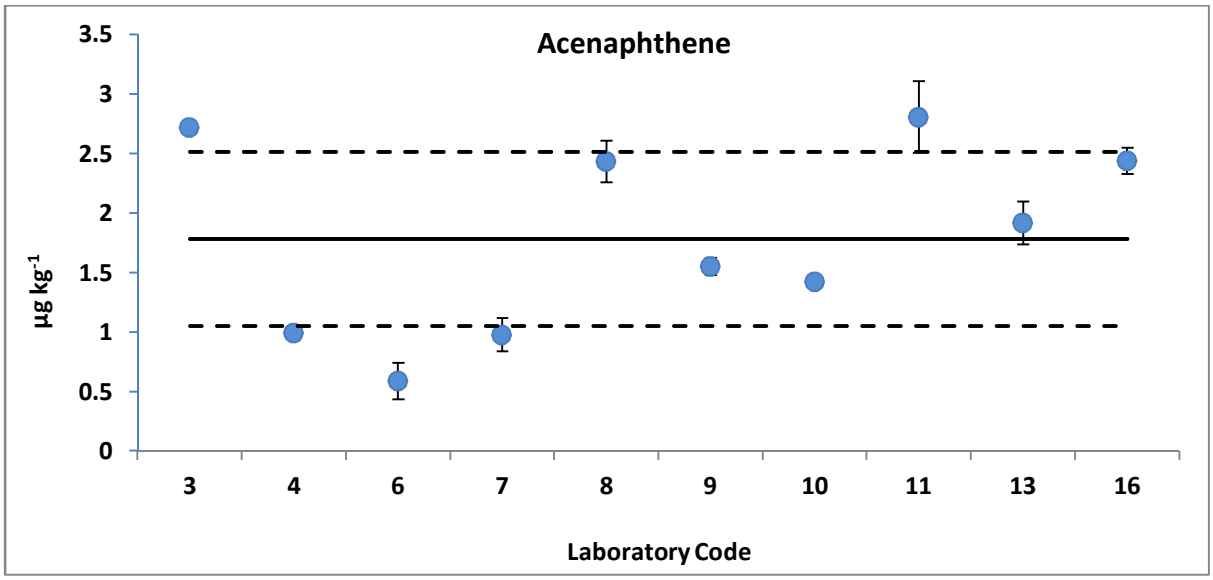


*FIG. 9. Laboratory results for Acenaphthene in IAEA-459 (*µg kg^{-1}*).*

TABLE 23. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF DIBENZOTHIOPHENE REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
4	4.73	0.16	2.00	GC-MS	SRM1941b
8	9.43	0.37		GC-MS	NIST 1944
10	10.28		0.50	GC-MS	IMR LRM
11	9.37	0.88	0.30	GC-HRMS	
13	8.49	0.26	0.10	GC-MS	NIST 1944
16	16.14	0.50	0.01	GC-MS	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

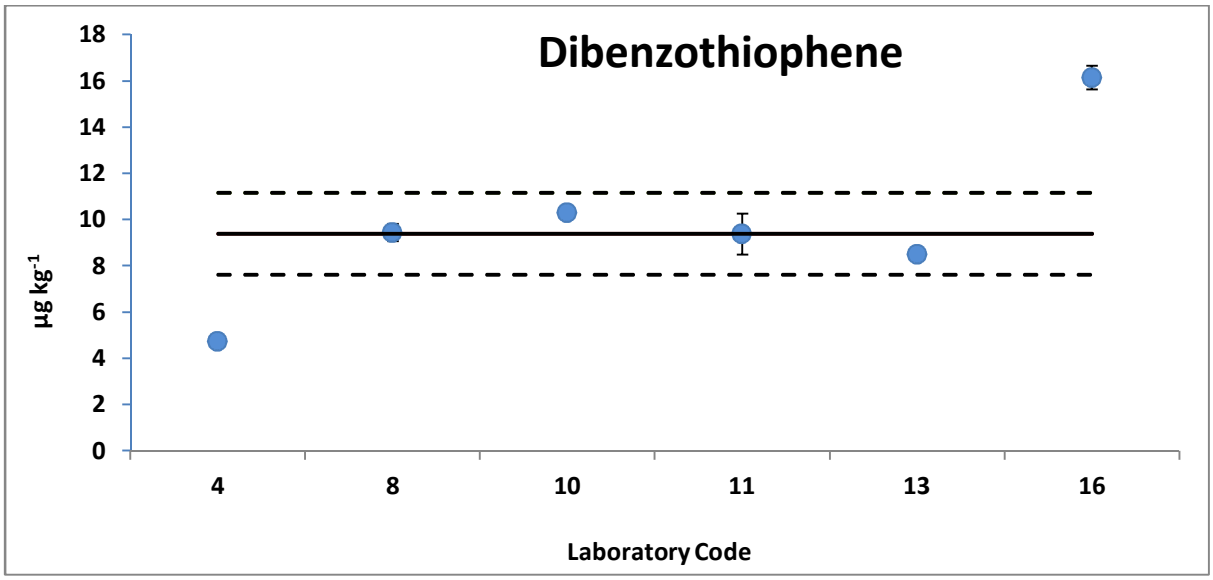


*FIG. 10. Laboratory results for Dibenzothiophene in IAEA-459 (*µg kg^{-1}*).*

TABLE 24. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF PHENANTHRENE REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
3	49.30		0.10	GC-MS	
4	22.93	0.46	2.00	GC-MS	SRM1941b
6	31.48	2.38	0.10	GC-MS/MS	IAEA-408
7	37.18	2.46	0.50	HPLC-FLD	Quasimeme QPH087
8	31.60	0.12	0.76	GC-MS	NIST 1944
9	29.60	0.68	<1	GC-MS	RM IAEA-159
10	34.39		0.50	GC-MS	IMR LRM
11	37.25	3.90	0.30	GC-HRMS	
13	30.43	0.65	0.10	GC-MS	NIST 1944
16	40.75	0.90	0.01	GC-MS	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

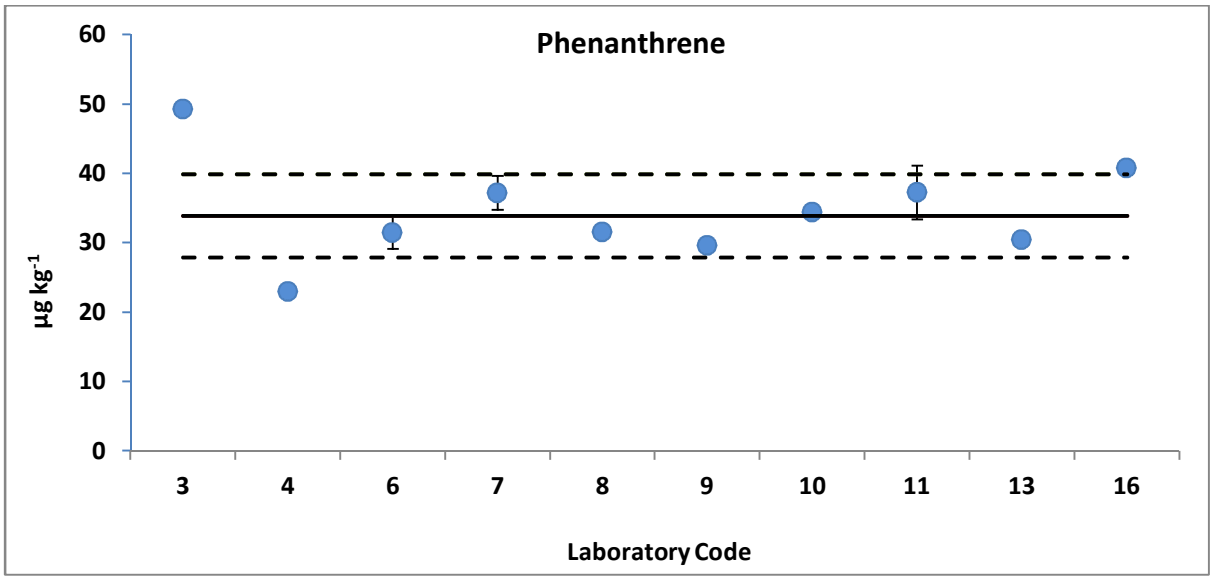


*FIG. 11. Laboratory results for Phenanthrene in IAEA-459 (*µg kg^{-1}*).*

TABLE 25. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF ANTHRACENE REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
3	5.84		0.20	GC-MS	
4	3.42	0.07	2.00	GC-MS	SRM1941b
6	6.57	0.38	0.12	GC-MS/MS	IAEA-408
7	5.79	0.31	0.50	HPLC-FLD	Quasimeme QPH087
8	6.20	0.40	0.82	GC-MS	NIST 1944
9	6.00	0.30	<1	GC-MS	RM IAEA-159
10	7.51		0.50	GC-MS	IMR LRM
11	5.06	0.52	0.30	GC-HRMS	
13	5.77	0.42	0.10	GC-MS	NIST 1944
16	9.87	0.53	0.02	GC-MS	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported by participants.

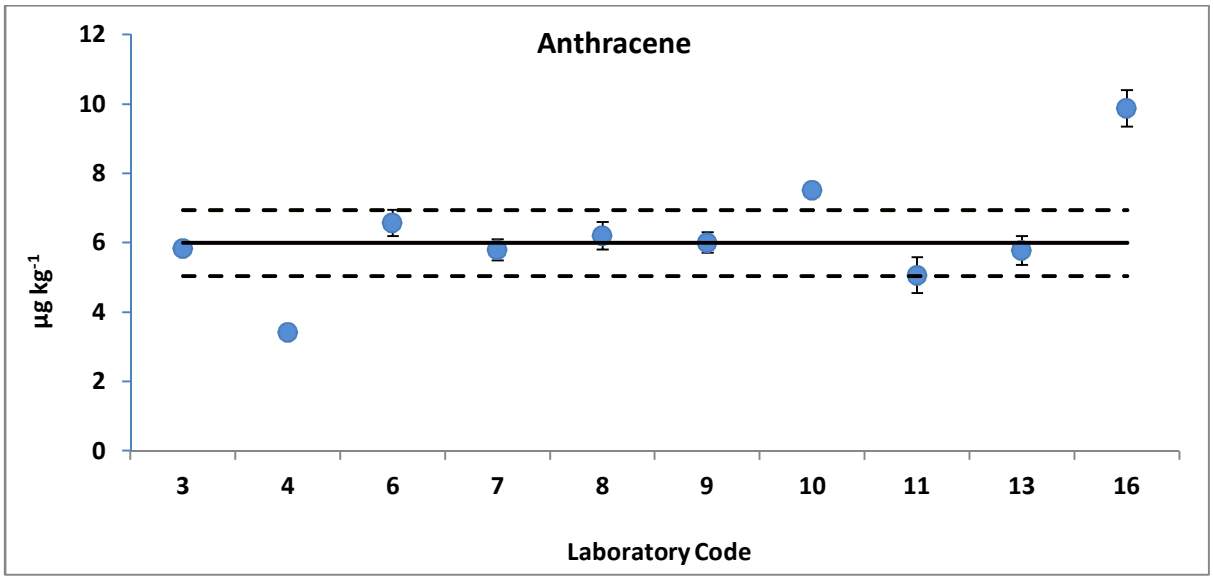


*FIG. 12. Laboratory results for Anthracene in IAEA-459 (*µg kg^{-1}*).*

TABLE 26. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF FLUORANTHENE REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
3	35.50		0.20	GC-MS	
4	30.98	0.69	1.00	GC-MS	SRM1941b
6	38.26	0.98	0.11	GC-MS/MS	IAEA-408
7	37.34	1.90	0.50	HPLC-FLD	Quasimeme QPH087
8	40.93	1.70	0.46	GC-MS	NIST 1944
9	36.13	0.87	<1	GC-MS	RM IAEA-159
10	36.13		0.50	GC-MS	IMR LRM
11	34.99	3.39	0.40	GC-HRMS	
13	46.65	1.56	0.10	GC-MS	NIST 1944
16	60.94	1.28	0.01	GC-MS	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported by participants.

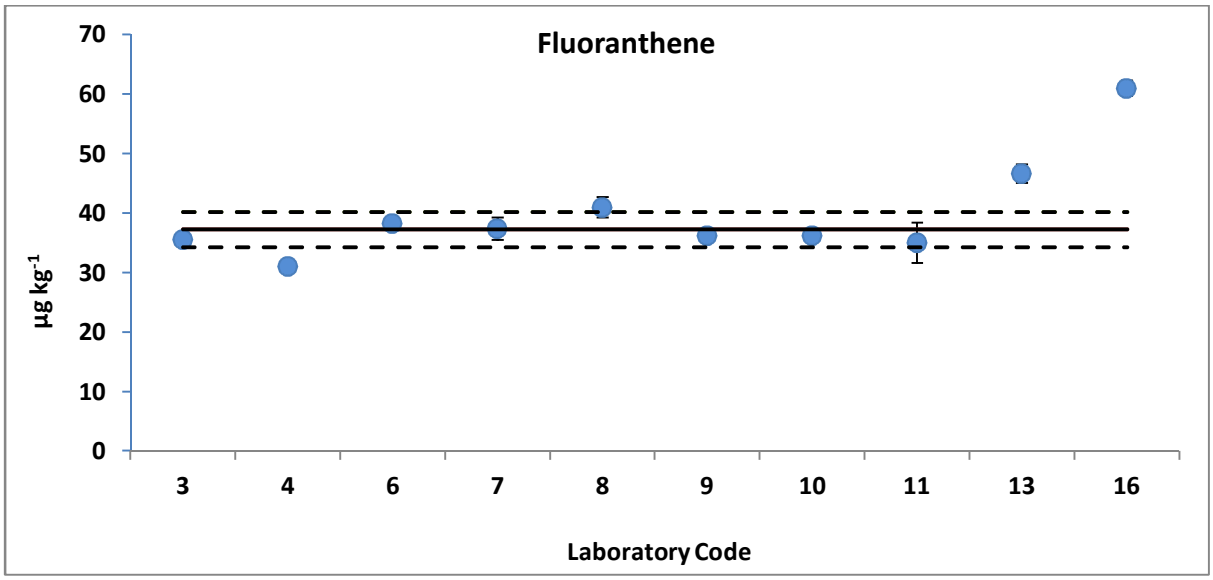


FIG. 13. Laboratory results for Fluoranthene in IAEA-459 (µg kg^{-1}).

TABLE 27. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF PYRENE REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
3	36.90		0.20	GC-MS	
4	33.56	1.34	1.00	GC-MS	SRM1941b
6	47.26	0.65	0.21	GC-MS/MS	IAEA-408
7	49.30	2.80	0.50	HPLC-FLD	Quasimeme QPH087
8	57.10	2.55	0.60	GC-MS	NIST 1944
9	40.08	1.03	<1	GC-MS	RM IAEA-159
10	43.69		0.50	GC-MS	IMR LRM
11	40.24	4.71	0.40	GC-HRMS	
13	53.89	0.99	0.10	GC-MS	NIST 1944
16	66.83	1.25	0.01	GC-MS	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

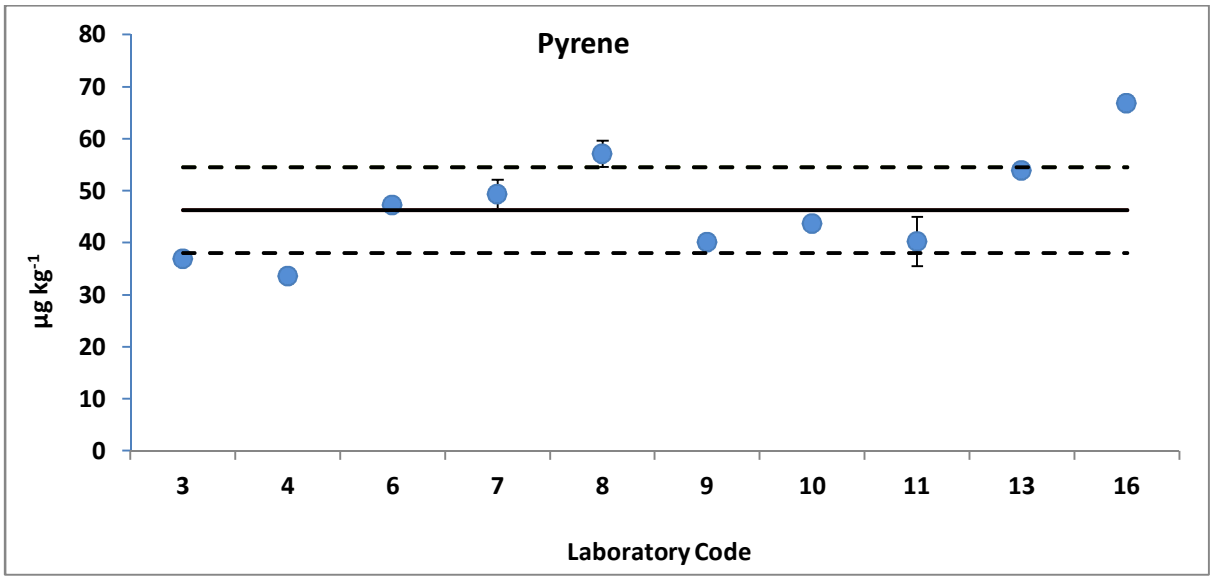


*FIG. 14. Laboratory results for Pyrene in IAEA-459 (*µg kg^{-1}*).*

TABLE 28. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF BENZ(*a*)ANTHRACENE REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
3	13.30		0.10	GC-MS	
4	13.83	0.56	1.00	GC-MS	SRM1941b
6	25.19	0.51	0.31	GC-MS/MS	IAEA-408
7	18.44	1.06	0.50	HPLC-FLD	Quasimeme QPH087
8	15.27	1.04	0.73	GC-MS	NIST 1944
9	18.53	0.55	<1	GC-MS	RM IAEA-159
10	19.73		0.50	GC-MS	IMR LRM
11	21.48	2.36	0.40	GC-HRMS	
13	21.28	3.01	0.10	GC-MS	NIST 1944
16	42.74	1.26	0.03	GC-MS	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

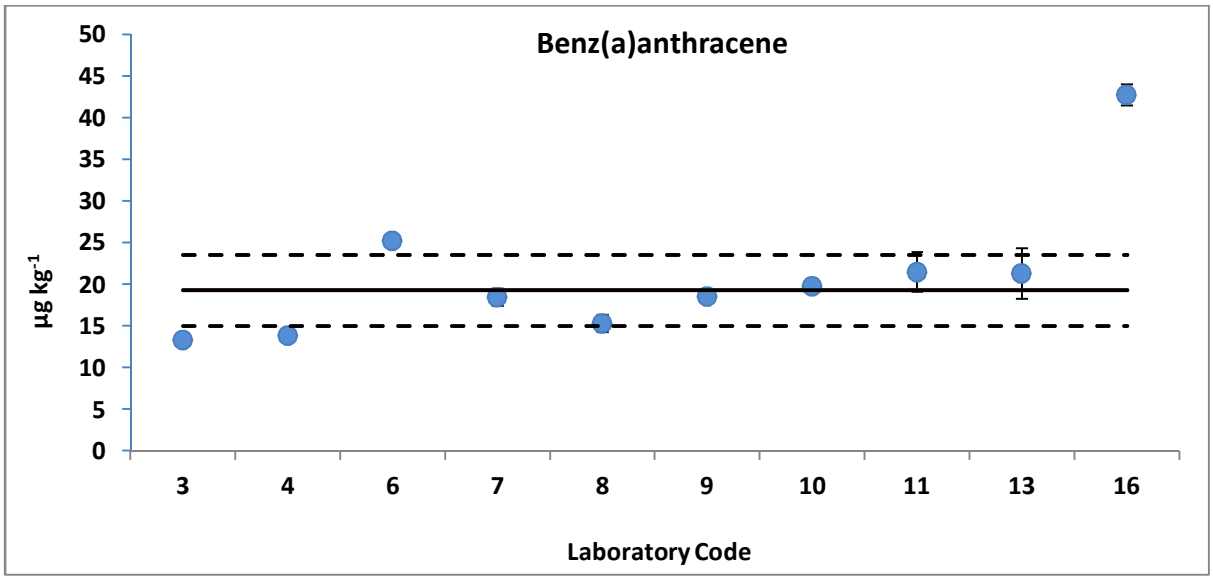


*FIG. 15. Laboratory results for Benz(a)anthracene in IAEA-459 (*µg kg^{-1}*).*

TABLE 29. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF BENZO(*k*)FLUORANTHENE REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
3	11.20		0.20	GC-MS	
4	13.88	0.35	2.00	GC-MS	SRM1941b
7	16.86	0.82	0.50	HPLC-FLD	Quasimeme QPH087
8	17.30	0.35	0.74	GC-MS	NIST 1944
9	18.38	1.07	<1	GC-MS	RM IAEA-159
10	17.50		0.50	GC-MS	IMR LRM
11	29.04	2.79	0.40	GC-HRMS	
13	24.34	0.33	0.10	GC-MS	NIST 1944
16	42.88	2.38	0.03	GC-MS	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

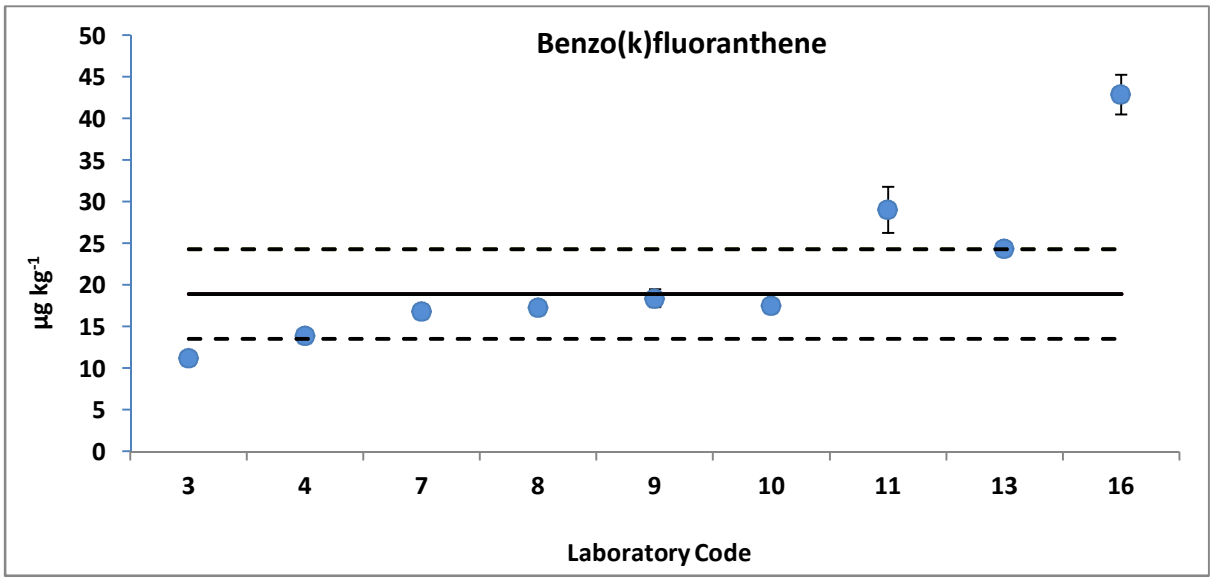


*FIG. 16. Laboratory results for Benzo(k)fluoranthene in IAEA-459 (*µg kg^{-1}*).*

TABLE 30. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF BENZO(*e*)PYRENE REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
4	28.96	0.70	1.00	GC-MS	SRM1941b
8	35.60	1.15	0.13	GC-MS	NIST 1944
10	34.11		0.50	GC-MS	IMR LRM
11	50.84	2.26	0.40	GC-HRMS	
13	42.42	0.68	0.10	GC-MS	NIST 1944
16	24.21	1.44	0.02	GC-MS	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

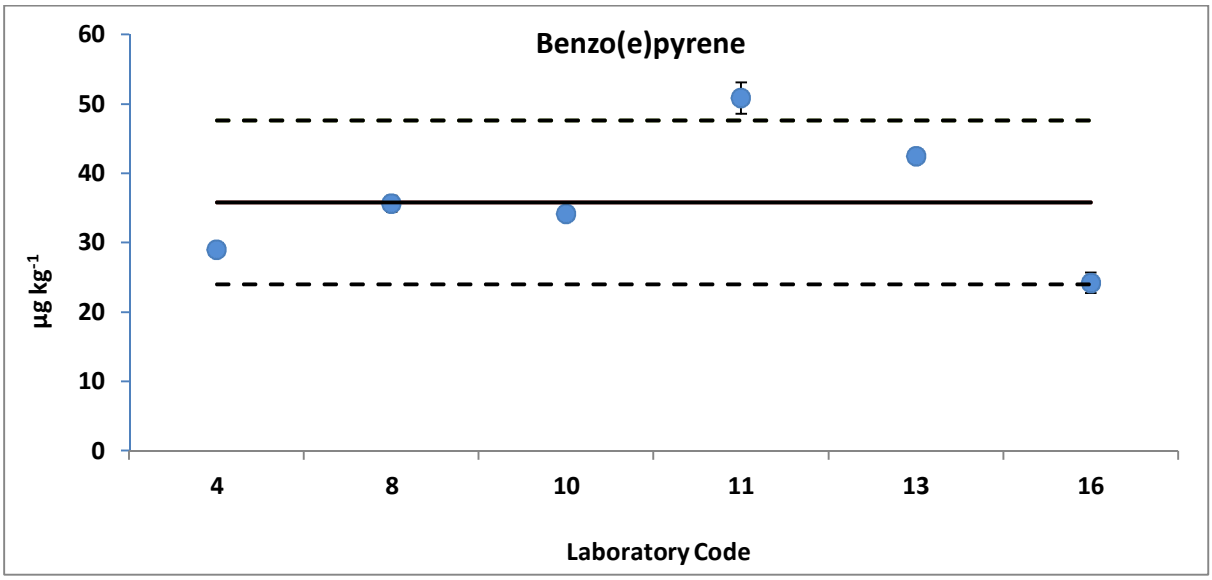


*FIG. 17. Laboratory results for Benzo(e)pyrene in IAEA-459 (*µg kg^{-1}*).*

TABLE 31. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF BENZO(*a*)PYRENE REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
3	16.40		0.10	GC-MS	
4	19.68	0.83	2.00	GC-MS	SRM1941b
6	22.54	0.59	0.35	GC-MS/MS	IAEA-408
7	20.90	1.23	0.50	HPLC-FLD	Quasimeme QPH087
8	18.63	0.87	0.76	GC-MS	NIST 1944
9	21.93	0.31	<1	GC-MS	RM IAEA-159
10	22.13		0.50	GC-MS	IMR LRM
11	27.94	2.99	0.40	GC-HRMS	
13	30.24	0.61	0.10	GC-MS	NIST 1944
16	47.98	1.10	0.02	GC-MS	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

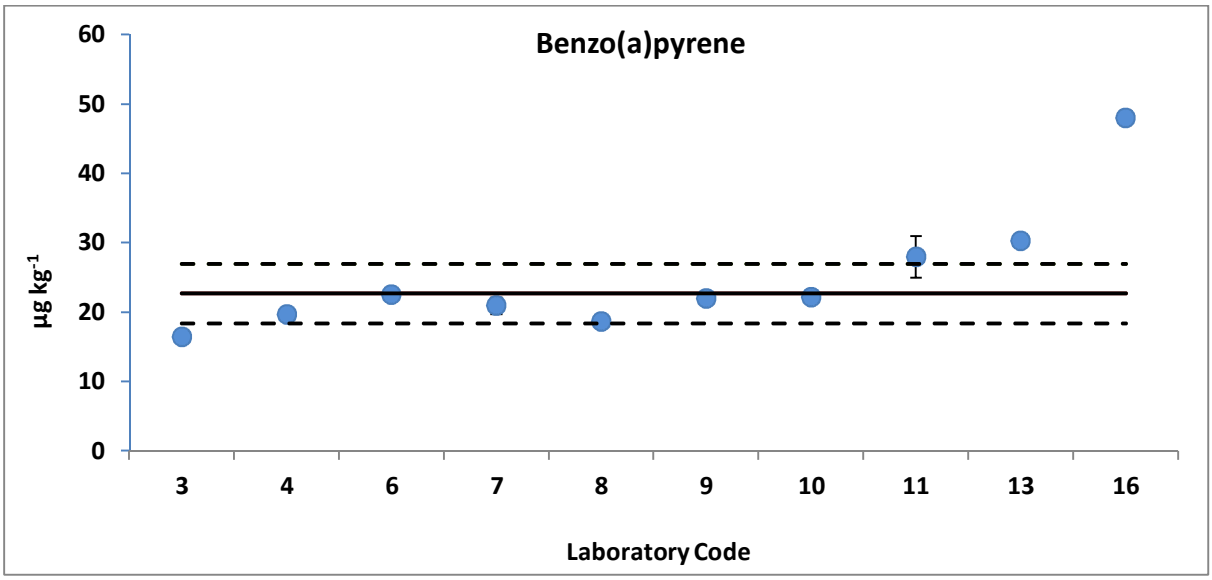


*FIG. 18. Laboratory results for Benzo(a)pyrene in IAEA-459 (*µg kg^{-1}*).*

TABLE 32. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF INDENO[1,2,3-*c,d*]PYRENE REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
3	16.80		0.10	GC-MS	
4	26.99	0.84	2.00	GC-MS	SRM1941b
7	36.69	2.03	0.50	HPLC-FLD	Quasimeme QPH087
8	27.03	0.93	0.73	GC-MS	NIST 1944
9	32.98	1.64	<1	GC-MS	RM IAEA-159
10	37.06		0.50	GC-MS	IMR LRM
11	42.28	4.59	0.40	GC-HRMS	
13	44.65	1.66	0.10	GC-MS	NIST 1944
16	100.29	5.83	0.09	GC-MS	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

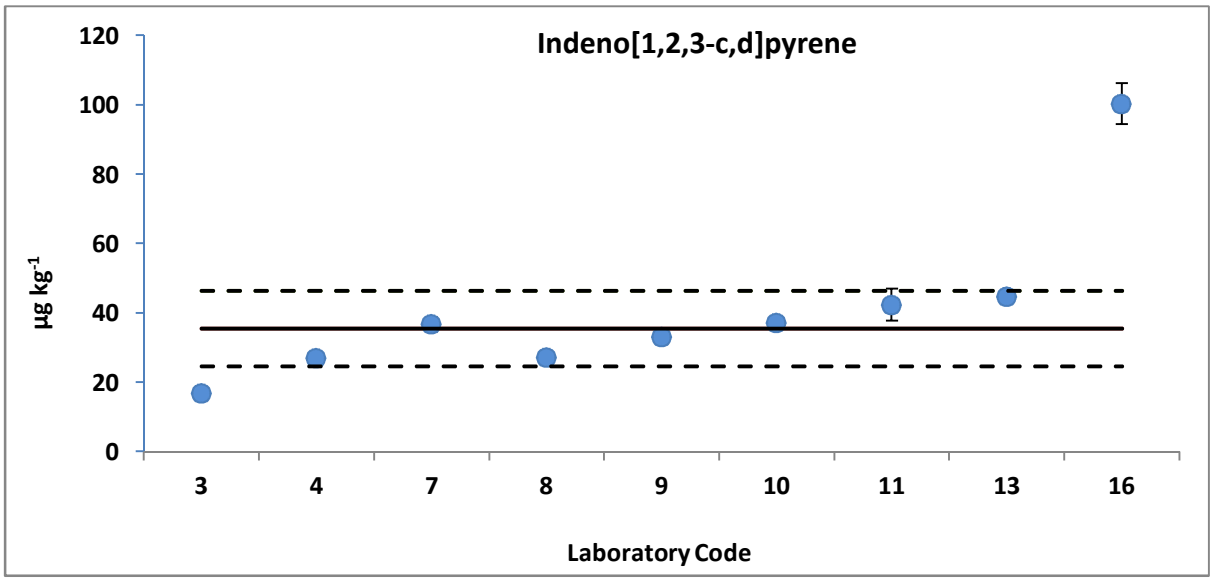


*FIG. 19. Laboratory results for Indeno[1,2,3-c,d]pyrene in IAEA-459 (*µg kg^{-1}*).*

TABLE 33. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF BENZO(*g,h,i*)PERYLENE REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
3	19.60		0.10	GC-MS	
4	32.82	0.63	2.00	GC-MS	SRM1941b
6	51.30	0.83	0.05	GC-MS/MS	IAEA-408
7	34.84	1.20	0.50	HPLC-FLD	Quasimeme QPH087
8	28.87	0.77	0.86	GC-MS	NIST 1944
9	34.86	0.54	<1	GC-MS	RM IAEA-159
10	37.78		0.50	GC-MS	IMR LRM
11	8.46	1.01	0.40	GC-HRMS	
13	45.39	1.39	0.10	GC-MS	NIST 1944
16	56.53	2.29	0.04	GC-MS	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

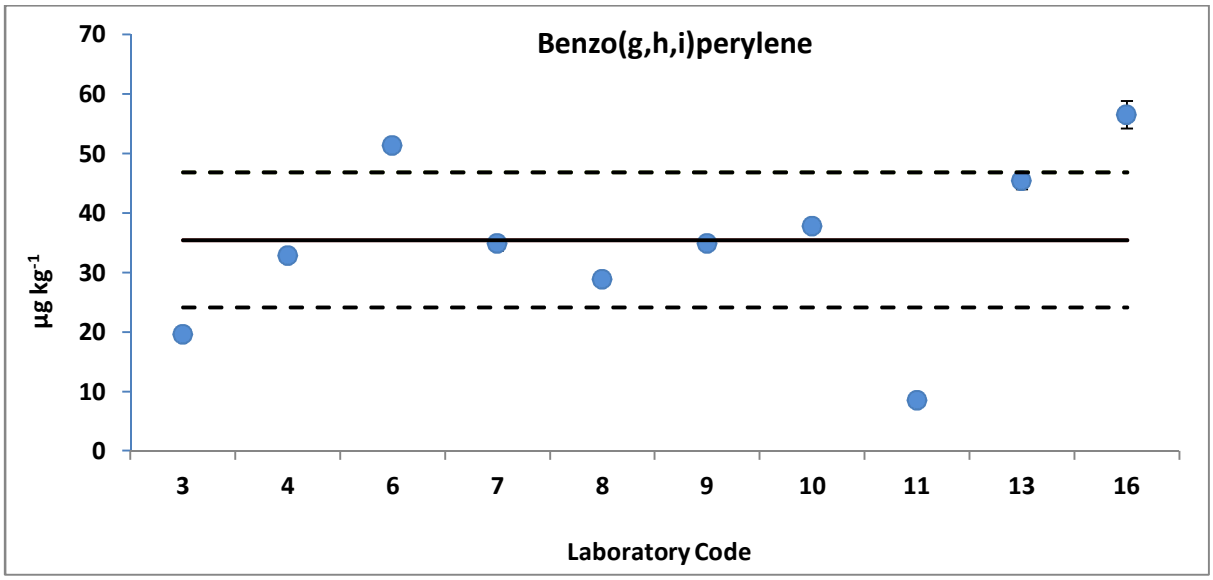


*FIG. 20. Laboratory results for Benzo(g,h,i)perylene in IAEA-459 (*µg kg^{-1}*).*

TABLE 34. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF CHRYSENE+TRIPHENYLENE REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
3	17.40		0.20	GC-MS	
4	24.51	0.58	1.00	GC-MS	SRM1941b
6	35.80	1.34	0.08	GC-MS/MS	IAEA-408
8	20.80	0.72	0.52	GC-MS	NIST 1944
9	24.12			GC-MS	RM IAEA-159
11	33.52			GC-HRMS	
16	36.24	1.10	0.01	GC-MS	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

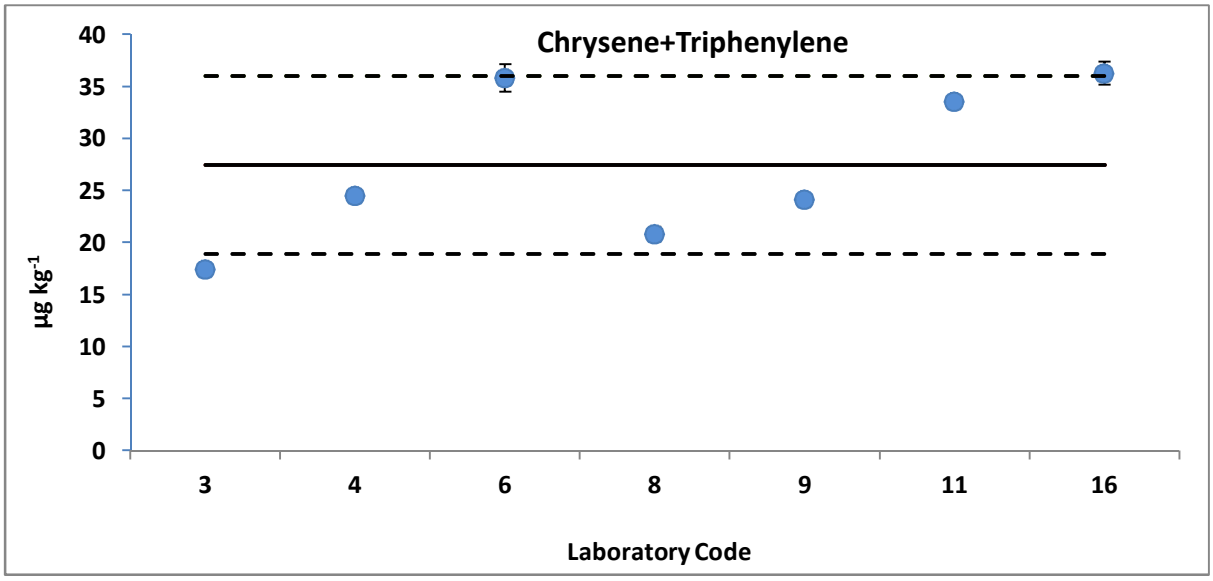


*FIG. 21. Laboratory results for Chrysene+Triphenylene in IAEA-459 (*µg kg^{-1}*).*

TABLE 35. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF BENZO(*b+j*)FLUORANTHENE REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
3	42.90		0.20	GC-MS	
4	50.17	0.58	2.00	GC-MS	SRM1941b
6	63.48	0.50	0.31	GC-MS/MS	IAEA-408
8	40.27	1.70	0.92	GC-MS	NIST 1944
9	59.86			GC-MS	RM IAEA-159
10	66.47			GC-MS	IMR LRM
11	69.85			GC-HRMS	
16	77.55	4.28	0.03	GC-MS	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

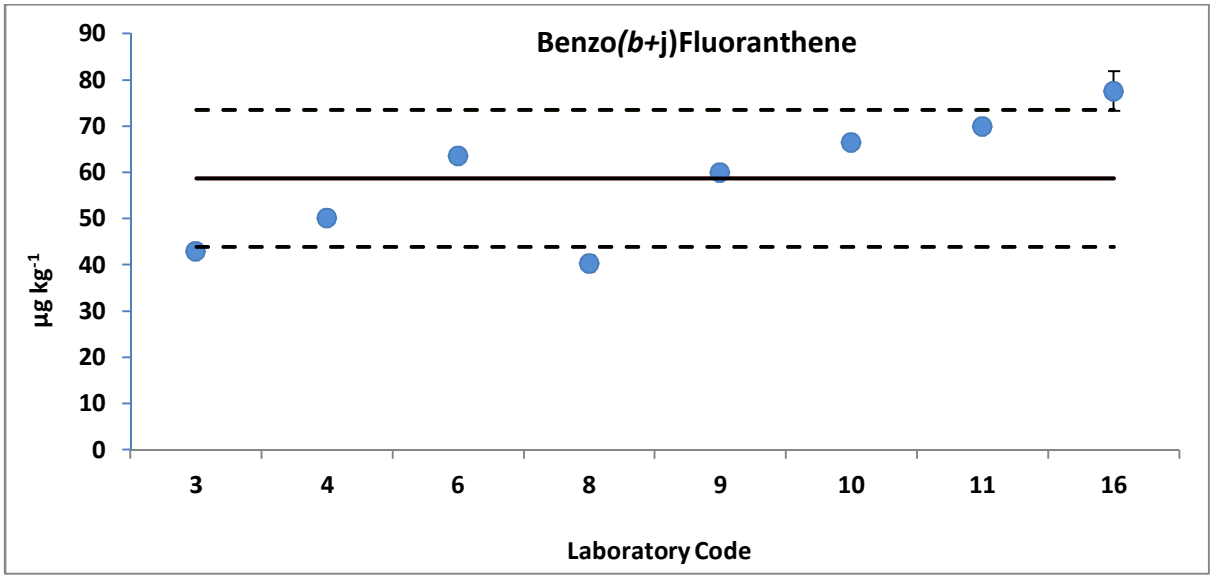


*FIG. 22. Laboratory results for Benzo(b+j)Fluoranthene in IAEA-459 (*µg kg^{-1}*).*

TABLE 36. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF BENZO(*b*)FLUORANTHENE REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
4	35.13	0.58	2.00	GC-MS	SRM1941b
7	44.10	1.55	0.50	HPLC-FLD	Quasimeme QPH087
10	47.65		0.50	GC-MS	IMR LRM
11	40.51	2.25	0.40	GC-HRMS	
13	56.79	2.24	0.10	GC-MS	NIST 1944

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

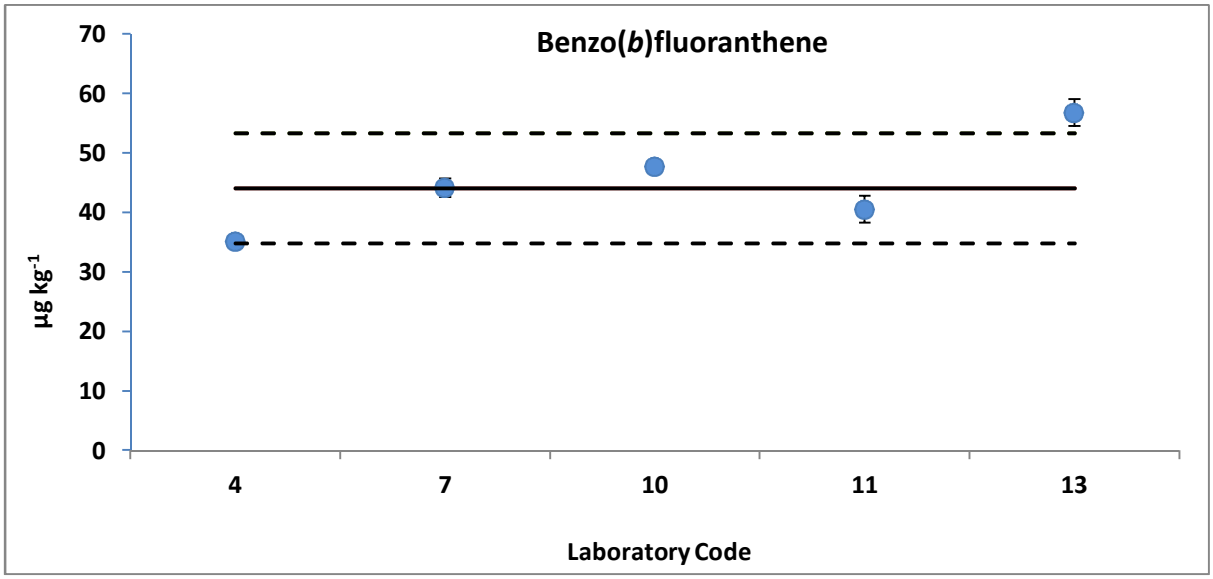


*FIG. 23. Laboratory results for Benzo(b)fluoranthene in IAEA-459 (*µg kg^{-1}*).*

APPENDIX II

RESULTS FOR THE CERTIFIED MEASUREMENTS OF POLYCHORINATED BIPHENYLS (PCBs)

TABLE 37. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF PCB 28 REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
3	2.98		0.001	GC-MS/MS	
4	2.08	0.04	0.100	GC-ECD	SRM1941b
5	2.09	0.31	0.003	GC-HRMS	NIST 1944
6	3.16	0.31	0.004	GC-MS/MS	IAEA-408
7	1.80	0.10	0.050	GC-MS/MS	Quasi QOR123MS
9	2.21	0.05	0.250	GC-MS	
10	2.52	0.14	0.020	GC-ECD	
11	2.69	0.08	0.050	GC-HRMS	
13	1.95	0.34		GC-MS/MS	NIST1944
15	1.36	0.11	0.100	GC-ECD	Quasi MS-2
16	2.28	0.11	0.024	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

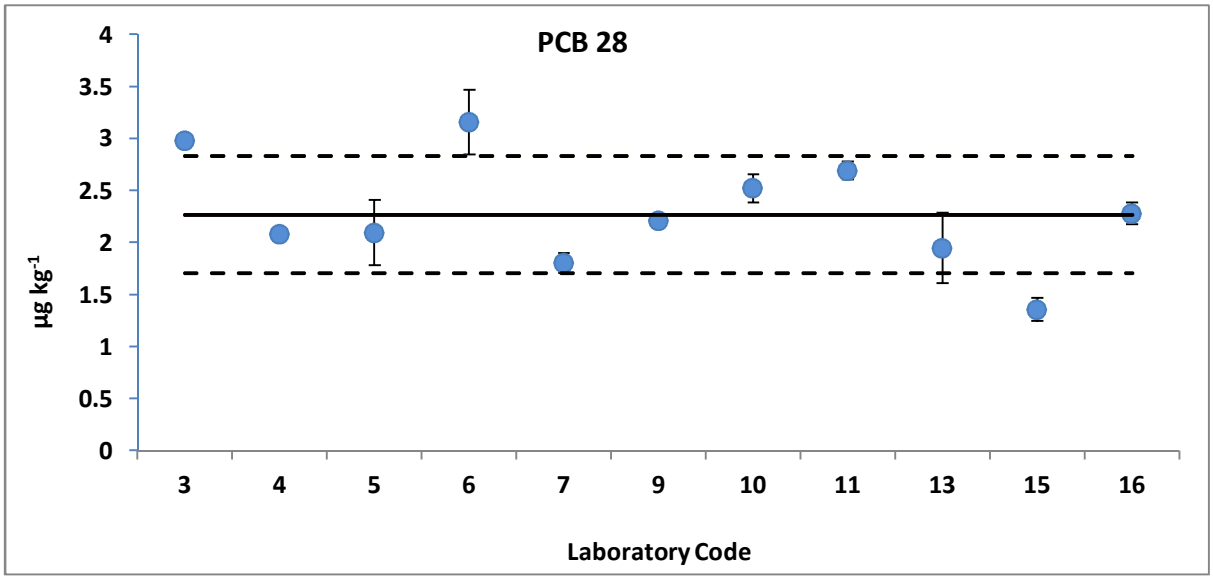


*FIG. 24. Laboratory results for PCB 28 in IAEA-459 (*µg kg^{-1}*).*

TABLE 38. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF PCB 31 REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
5	2.35	0.36	0.003	GC-HRMS	NIST 1944
9	2.19	0.05	0.250	GC-MS	
10	2.43	0.07	0.020	GC-ECD	
11	3.05	0.08	0.050	GC-HRMS	
13	2.49	0.44		GC-MS/MS	NIST1944
15	1.76	0.16	0.100	GC-ECD	Quasi MS-2
16	2.57	0.11	0.060	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

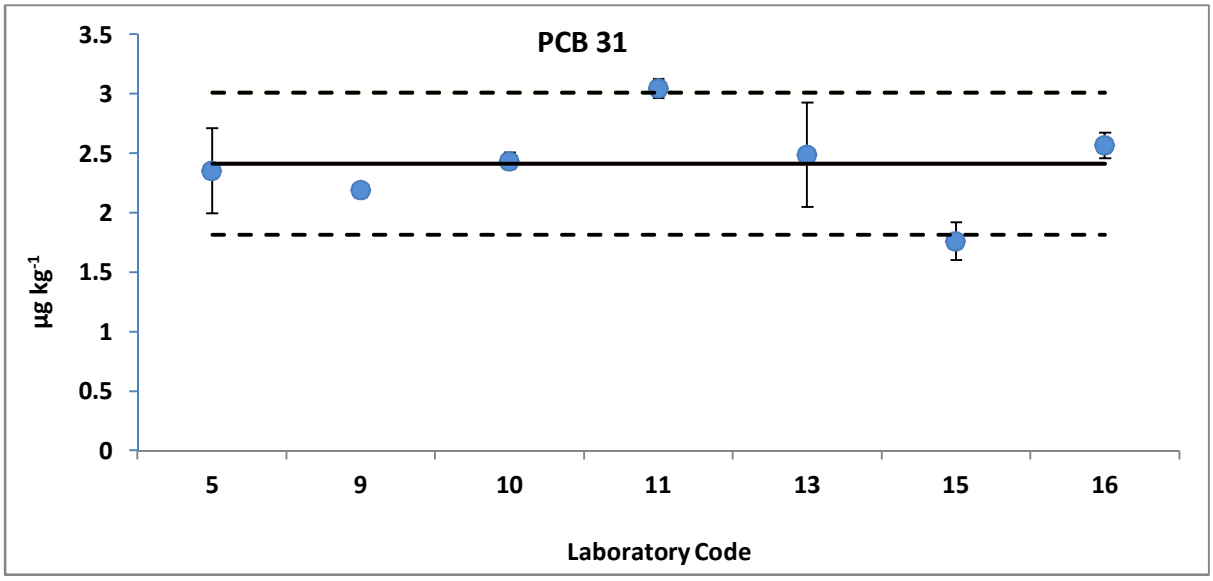


*FIG. 25. Laboratory results for PCB 31 in IAEA-459 (*µg kg^{-1}*).*

TABLE 39. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF PCB 44 REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
2	2.24	0.22	1.000	GCxGC-ECD	
4	1.75	0.09	0.100	GC-ECD	SRM1941b
5	1.66	0.07	0.005	GC-HRMS	NIST 1944
9	1.78	0.10	0.250	GC-MS	RM IAEA-159
11	1.51	0.16	0.030	GC-HRMS	
13	1.87	0.32		GC-MS/MS	NIST1944
16	1.40	0.05	0.019	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

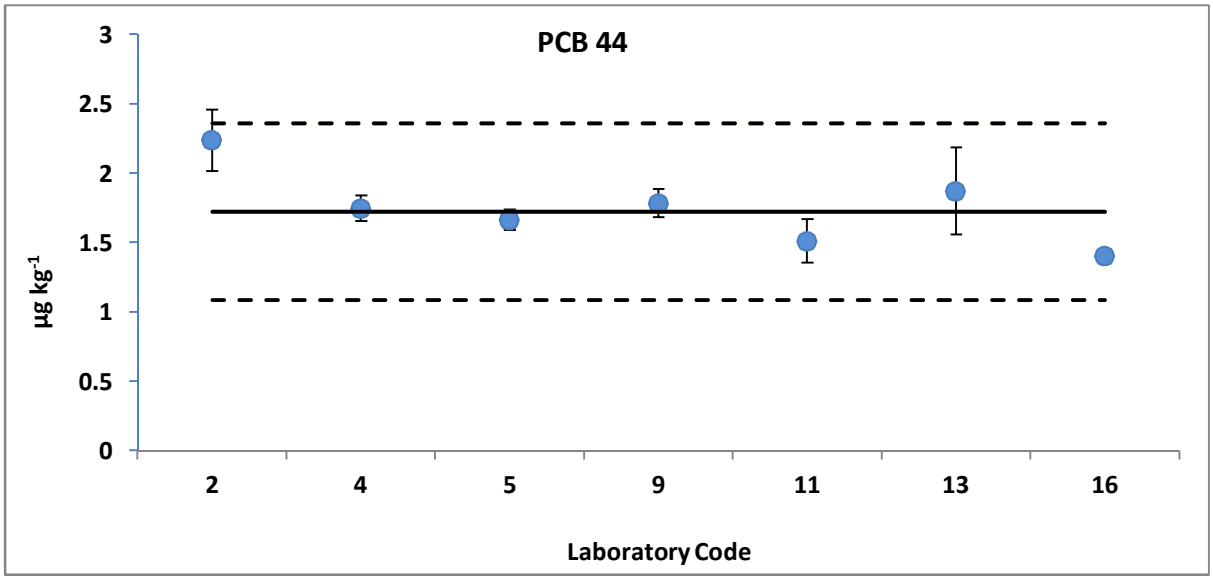


*FIG. 26. Laboratory results for PCB 44 in IAEA-459 (*µg kg^{-1}*).*

TABLE 40. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF PCB 49 REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
2	3.04	0.42	1.000	GCxGC-ECD	
5	2.64	0.10	0.005	GC-HRMS	NIST 1944
11	2.67	0.20	0.030	GC-HRMS	
13	2.62	0.43		GC-MS/MS	NIST1944
16	1.80	0.04	0.063	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

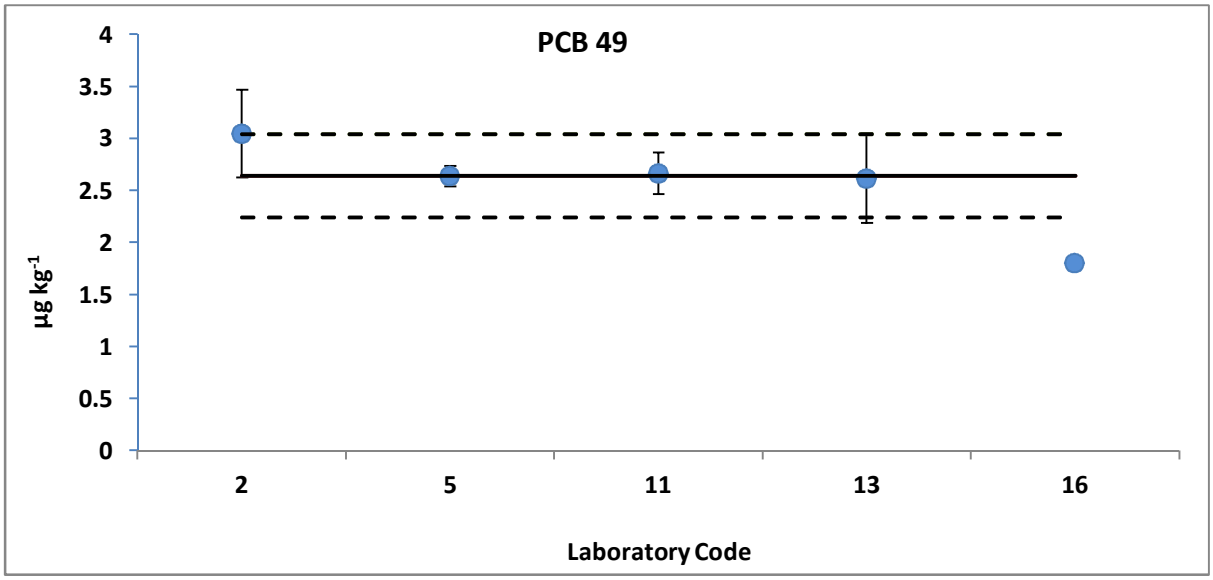


*FIG. 27. Laboratory results for PCB 49 in IAEA-459 (*µg kg^{-1}*).*

TABLE 41. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF PCB 52 REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
2	1.98	0.32	1.000	GCxGC-ECD	
3	2.06		0.001	GC-MS/MS	
4	2.18	0.02	0.100	GC-ECD	SRM1941b
5	2.60	0.11	0.005	GC-HRMS	NIST 1944
6	2.98	0.20	0.001	GC-MS/MS	IAEA-408
7	2.59	0.15	0.050	GC-MS/MS	Quasi QOR123MS
9	3.63	0.14	0.250	GC-MS	
10	1.71	0.39	0.020	GC-ECD	
11	2.80	0.21	0.080	GC-HRMS	
13	2.54	0.38		GC-MS/MS	NIST1944
15	1.90	0.06	0.100	GC-ECD	Quasi MS-2
16	2.06	0.06	0.077	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

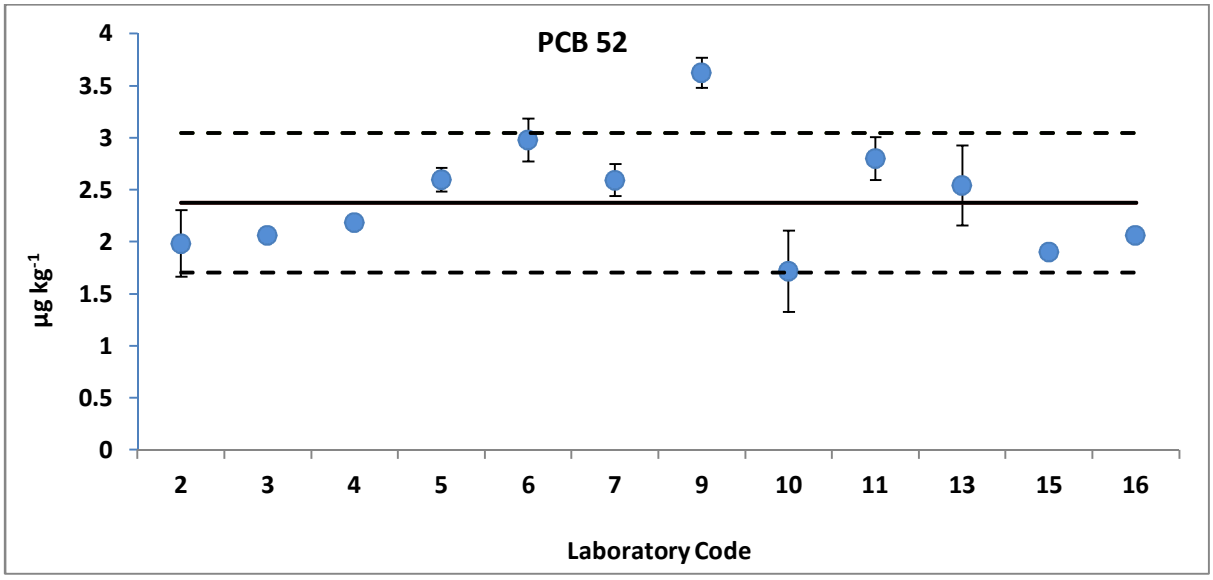


*FIG. 28. Laboratory results for PCB 52 in IAEA-459 (*µg kg^{-1}*).*

TABLE 42. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF PCB 66 REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
2	2.87	0.20	1.000	GCxGC-ECD	
4	2.28	0.06	0.100	GC-ECD	SRM1941b
5	3.60	0.12	0.005	GC-HRMS	NIST 1944
11	3.82	0.34	0.040	GC-HRMS	
13	2.94	0.54		GC-MS/MS	NIST1944

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

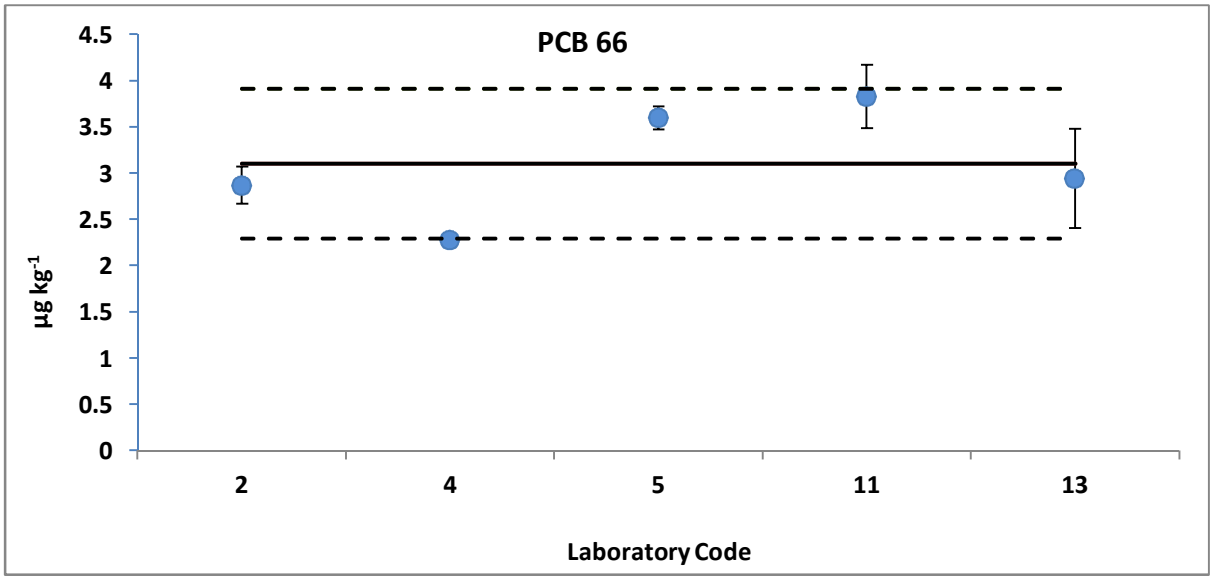


*FIG. 29. Laboratory results for PCB 66 in IAEA-459 (*µg kg^{-1}*).*

TABLE 43. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF PCB 87 REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
2	1.99	0.32	1.000	GCxGC-ECD	
4	1.23	0.06	0.100	GC-ECD	SRM1941b
5	1.11	0.05	0.005	GC-HRMS	NIST 1944
11	1.30	0.05	0.020	GC-HRMS	
16	1.16	0.02	0.017	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

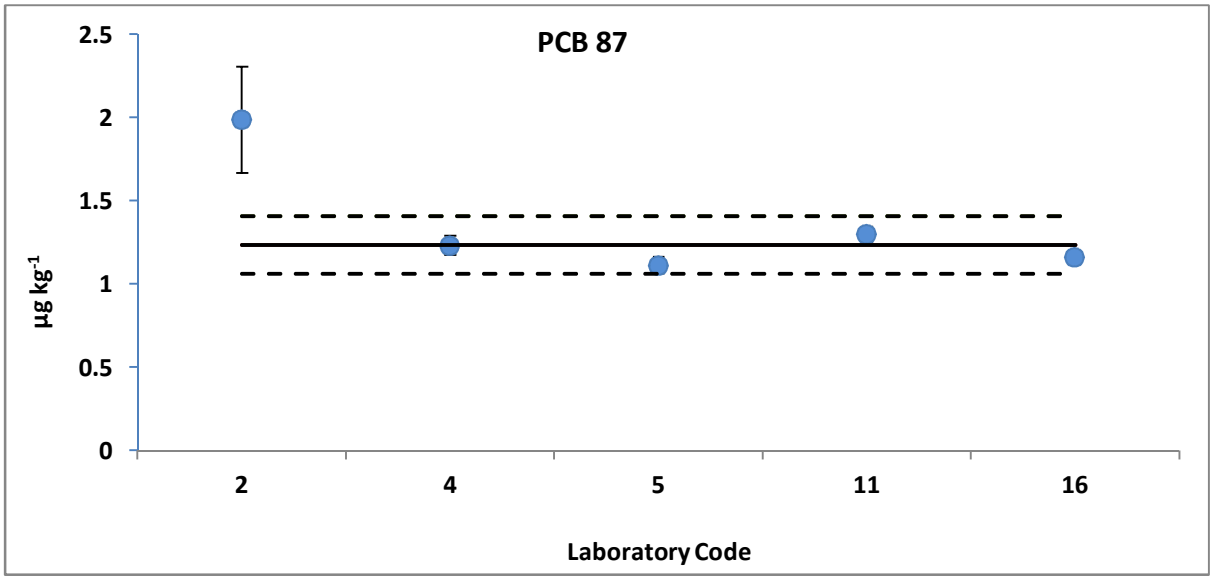


*FIG. 30. Laboratory results for PCB 87 in IAEA-459 (*µg kg^{-1}*).*

TABLE 44. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF PCB 101 REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
2	4.02	0.44	1.000	GCxGC-ECD	
3	2.42		0.001	GC-MS/MS	
4	3.48	0.08	0.100	GC-ECD	SRM1941b
5	4.10	0.16	0.005	GC-HRMS	NIST 1944
6	4.82	0.32	0.005	GC-MS/MS	IAEA-408
7	3.59	0.16	0.050	GC-MS/MS	Quasi QOR123MS
9	3.57	0.07	0.250	GC-MS	RM IAEA-159
10	3.46	0.04	0.020	GC-ECD	
11	5.19	0.33	0.050	GC-HRMS	
13	3.99	0.66		GC-MS/MS	NIST1944
15	3.61	0.89	0.100	GC-ECD	Quasi MS-2
16	3.76	0.06	0.026	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

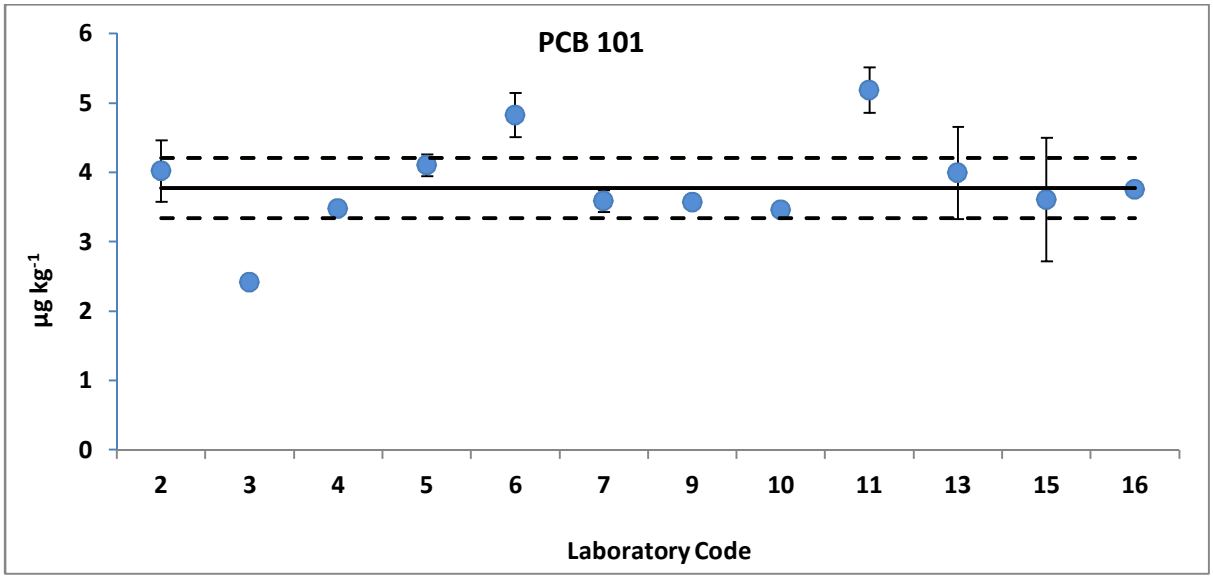


*FIG. 31. Laboratory results for PCB 101 in IAEA-459 (*µg kg^{-1}*).*

TABLE 45. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF PCB 105 REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
2	1.58	0.18	1.000	GCxGC-ECD	
4	1.15	0.07	0.100	GC-ECD	SRM1941b
5	1.32	0.01	0.010	GC-HRMS	NIST 1944
7	1.28	0.04	0.050	GC-MS/MS	Quasi QOR123MS
10	0.976	0.003	0.020	GC-ECD	
11	1.70	0.04	0.020	GC-HRMS	
13	1.51	0.24		GC-MS/MS	NIST1944
15	0.87	0.14	0.100	GC-ECD	Quasi MS-2
16	1.22	0.04	0.018	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

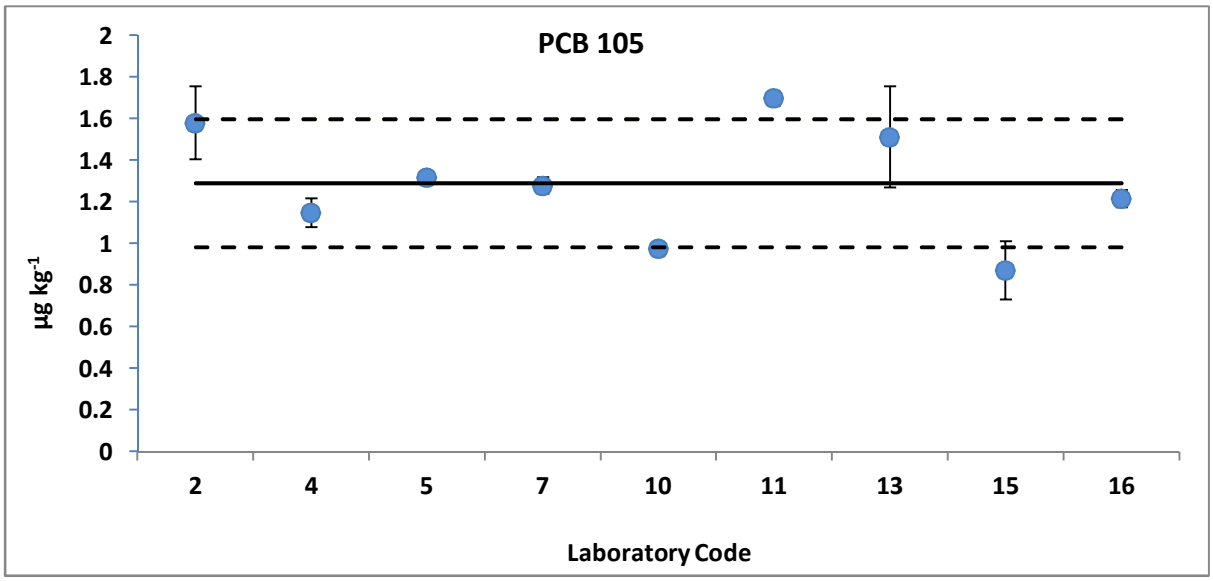


*FIG. 32. Laboratory results for PCB 105 in IAEA-459 (*µg kg^{-1}*).*

TABLE 46. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF PCB 110 REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
2	3.37	0.33	1.000	GCxGC-ECD	
5	4.31	0.16	0.005	GC-HRMS	NIST 1944
11	5.37	0.22	0.050	GC-HRMS	
13	3.11	0.47		GC-MS/MS	NIST1944
15	3.33	0.71	0.100	GC-ECD	
16	3.76	0.05	0.032	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

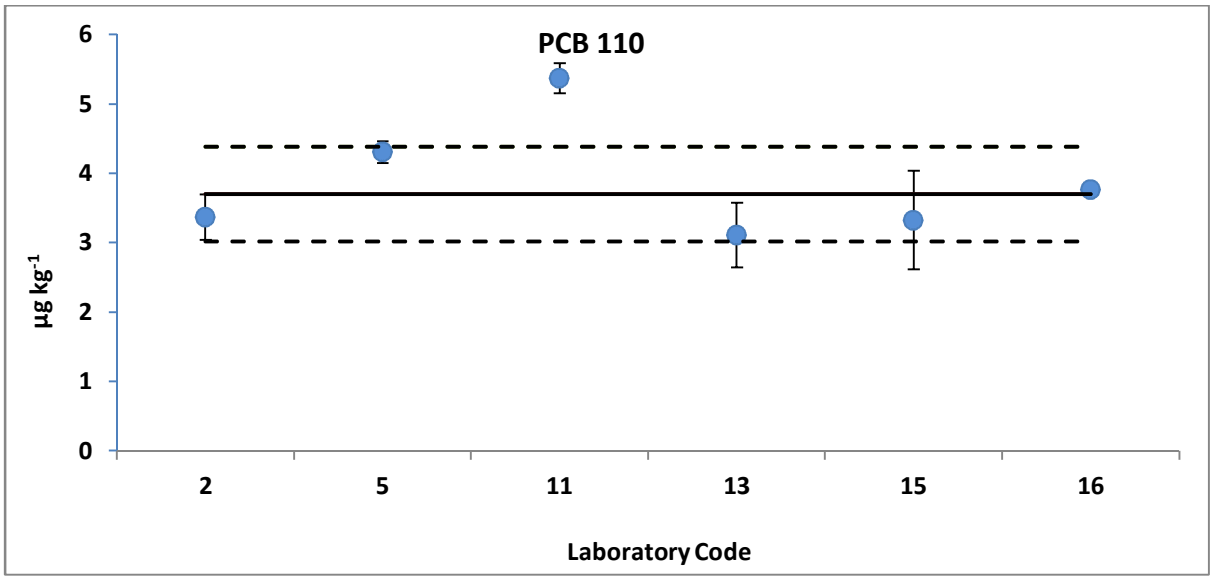


*FIG. 33. Laboratory results for PCB 110 in IAEA-459 (*µg kg^{-1}*).*

TABLE 47. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF PCB 118 REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
2	3.07	0.29	1.000	GCxGC-ECD	
4	2.74	0.11	0.100	GC-ECD	SRM1941b
5	3.00	0.02	0.005	GC-HRMS	NIST 1944
6	3.49	0.14	0.003	GC-MS/MS	IAEA-408
7	3.26	0.10	0.050	GC-MS/MS	Quasi QOR123MS
9	2.59	0.14	0.250	GC-MS	RM IAEA-159
10	2.63	0.02	0.020	GC-ECD	
11	4.41	0.14	0.050	GC-HRMS	
13	3.01	0.45		GC-MS/MS	NIST1944
15	2.49	0.40	0.100	GC-ECD	Quasi MS-2
16	2.88	0.04	0.059	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

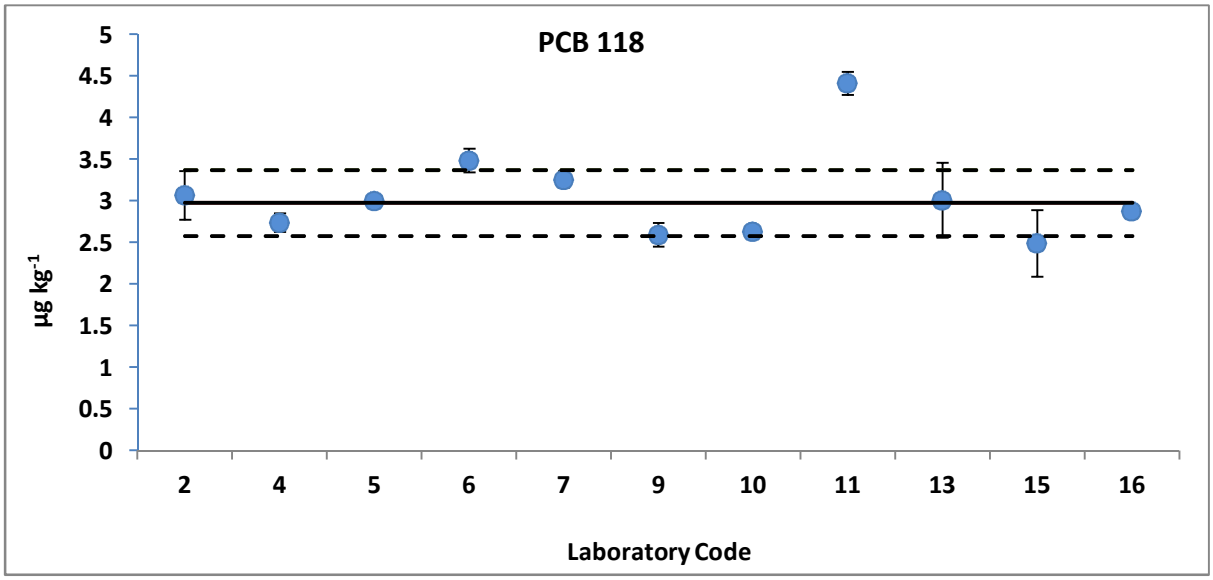


*FIG. 34. Laboratory results for PCB 118 in IAEA-459 (*µg kg^{-1}*).*

TABLE 48. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF PCB 128 REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
2**	0.55	0.19	1.000	GCxGC-ECD	
4	0.66	0.02	0.100	GC-ECD	SRM1941b
11	0.69	0.04	0.005	GC-HRMS	
13	0.61	0.10		GC-MS/MS	NIST1944
15	0.54	0.09	0.100	GC-ECD	
16	0.59	0.02	0.006	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

** Not taken into account for assigned value calculation

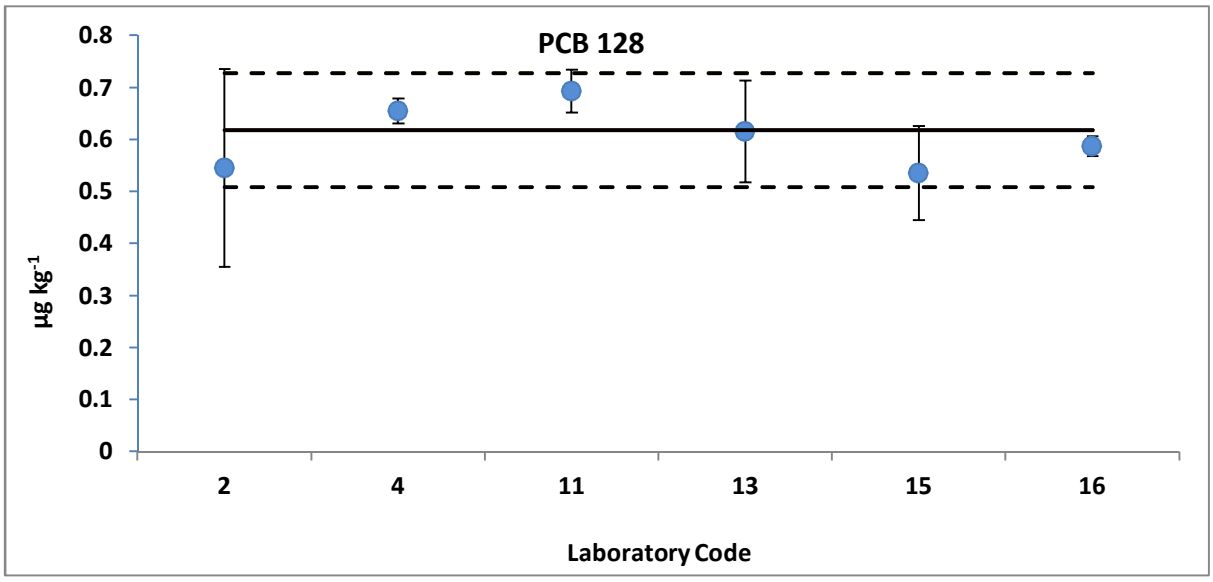


*FIG. 35. Laboratory results for PCB 128 in IAEA-459 (*µg kg^{-1}*).*

TABLE 49. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF PCB 138 REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
2	4.44	0.52	1.000	GCxGC-ECD	
3	1.29		0.001	GC-MS/MS	
4	3.66	0.19	0.100	GC-ECD	SRM1941b
5	3.08	0.12	0.005	GC-HRMS	NIST 1944
6	3.00	0.14	0.002	GC-MS/MS	IAEA-408
7	4.75	0.16	0.050	GC-MS/MS	Quasi QOR123MS
9	2.47	0.15	0.250	GC-MS	RM IAEA-159
10	2.23	0.06	0.020	GC-ECD	
11	3.59	0.10	0.020	GC-HRMS	
13	4.65	0.80		GC-MS/MS	NIST1944
15	2.19	0.29	0.100	GC-ECD	Quasi MS-2
16	3.42	0.06	0.030	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

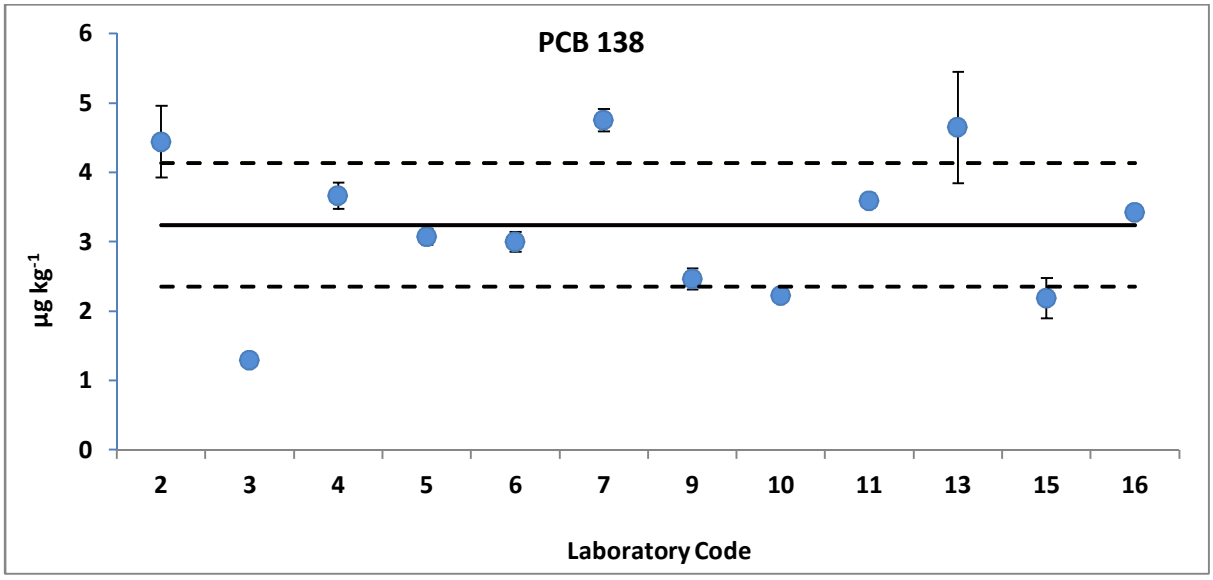


*FIG. 36. Laboratory results for PCB 138 in IAEA-459 (*µg kg^{-1}*).*

TABLE 50. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF PCB 149 REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
2	3.31	0.39	1.000	GCxGC-ECD	
5	2.78	0.10	0.005	GC-HRMS	NIST 1944
9	2.54	0.08	0.250	GC-MS	RM IAEA-159
11	4.09	0.43	0.020	GC-HRMS	
13	2.79	0.43		GC-MS/MS	NIST1944
16	2.44	0.03	0.053	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

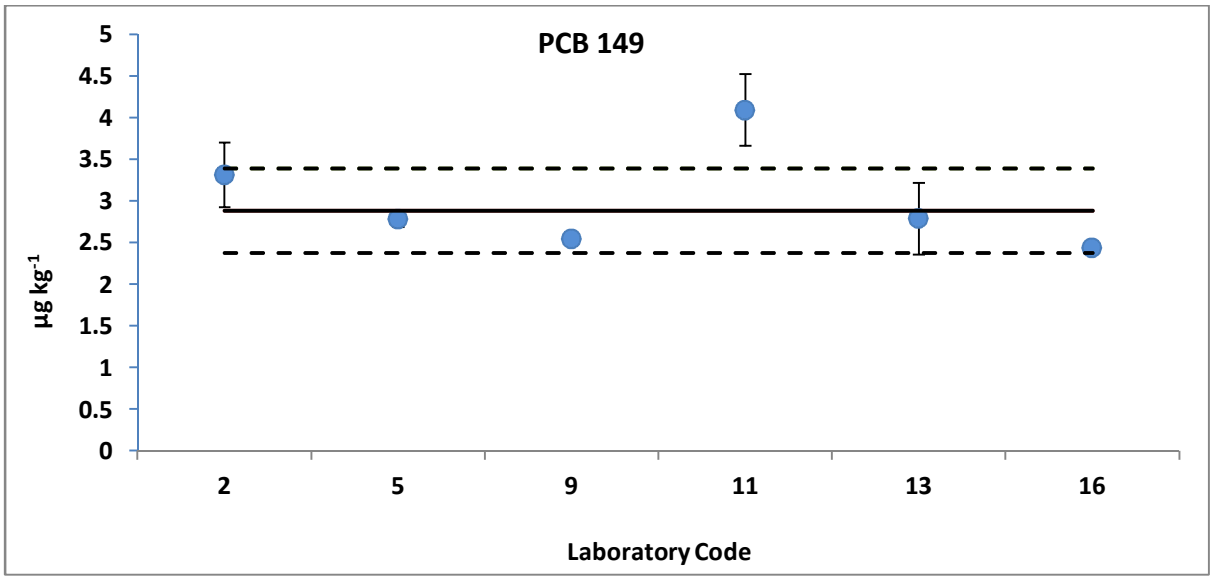


*FIG. 37. Laboratory results for PCB 149 in IAEA-459 (*µg kg^{-1}*).*

TABLE 51. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF PCB 151 REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
2	1.19	0.36	1.000	GCxGC-ECD	
5	0.52	0.02	0.005	GC-HRMS	NIST 1944
11	0.78	0.09	0.005	GC-HRMS	
13	0.61	0.09		GC-MS/MS	NIST1944
16	0.60	0.02	0.008	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

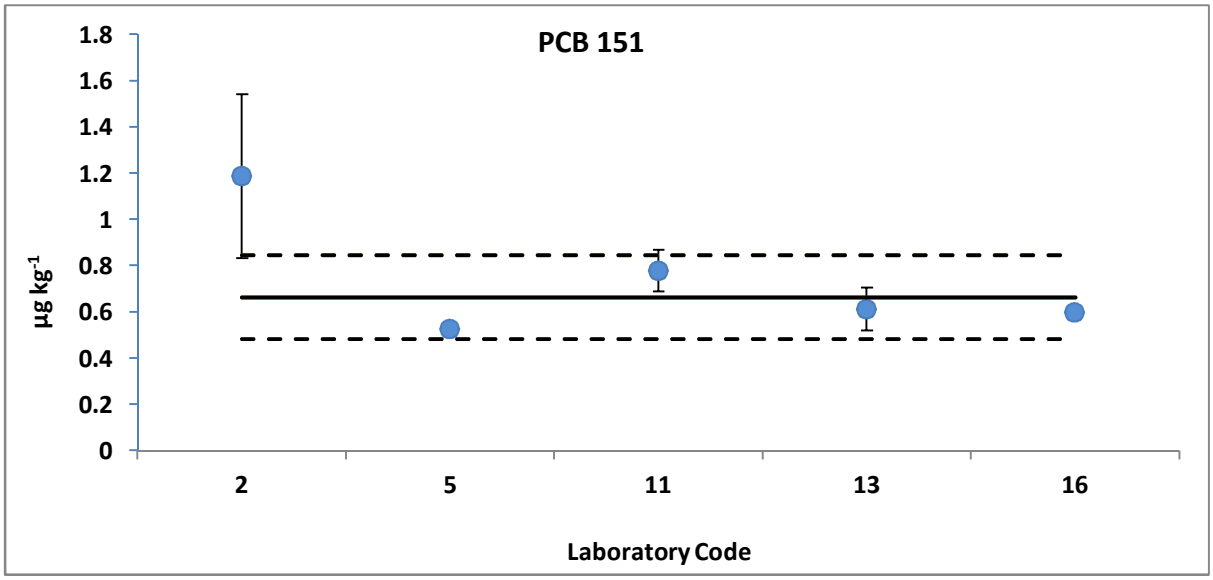


*FIG. 38. Laboratory results for PCB 151 in IAEA-459 (*µg kg^{-1}*).*

TABLE 52. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF PCB 153 REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
2	3.27	0.38	1.000	GCxGC-ECD	
3	1.53		0.001	GC-MS/MS	
4	4.75	0.23	0.100	GC-ECD	SRM1941b
5	4.08	0.13	0.005	GC-HRMS	NIST 1944
6	4.38	0.21	0.006	GC-MS/MS	IAEA-408
7	4.32	0.08	0.050	GC-MS/MS	Quasi QOR123MS
9	3.09	0.15	0.250	GC-MS	RM IAEA-159
10	3.22	0.05	0.020	GC-ECD	
11	4.61	0.12	0.020	GC-HRMS	
13	4.13	0.62		GC-MS/MS	NIST1944
15	3.01	0.27	0.100	GC-ECD	Quasi MS-2
16	3.60	0.08	0.022	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

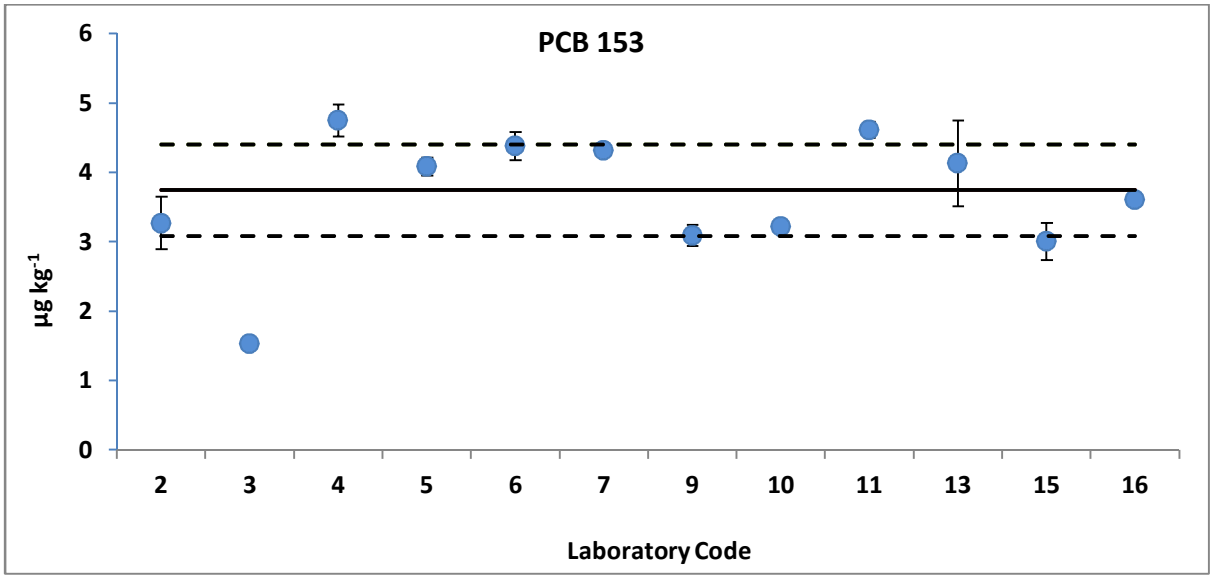


*FIG. 39. Laboratory results for PCB 153 in IAEA-459 (*µg kg^{-1}*).*

TABLE 53. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF PCB 156 REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
2	0.732	0.145	1.000	GCxGC-ECD	
5	0.322	0.007	0.003	GC-HRMS	NIST 1944
7	0.349	0.022	0.050	GC-MS/MS	Quasi QOR123MS
10	0.248	0.021	0.020	GC-ECD	
11	0.373	0.019	0.005	GC-HRMS	
13	0.402	0.065		GC-MS/MS	NIST1944
15	0.255	0.050	0.100	GC-ECD	Quasi MS-2
16	0.349	0.032	0.007	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

** Not taken into account for assigned value calculation

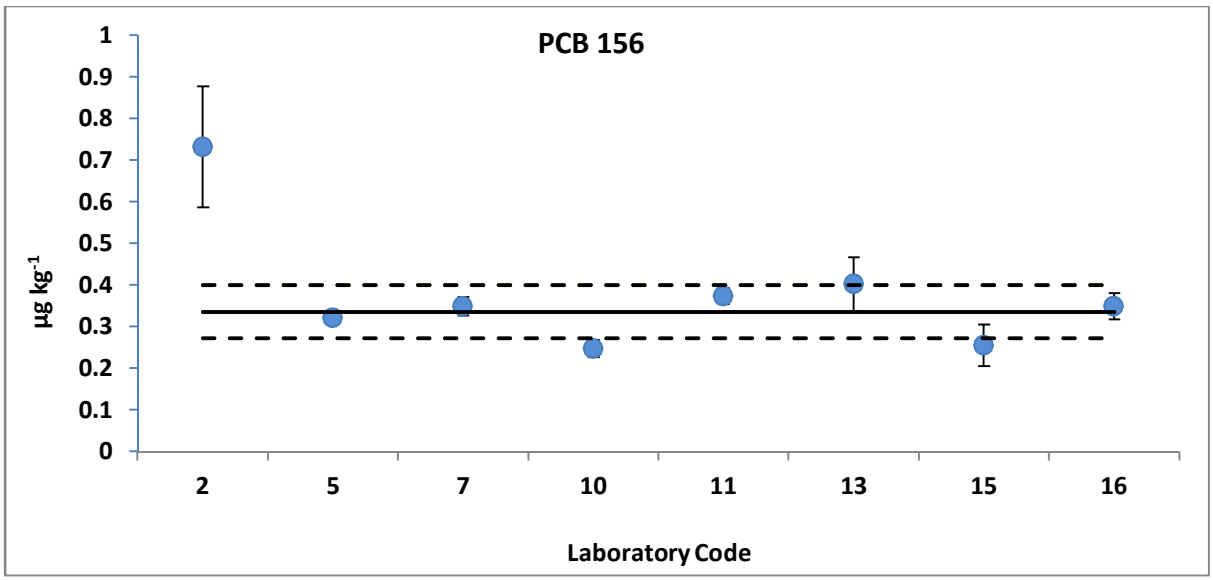


*FIG. 40. Laboratory results for PCB 156 in IAEA-459 (*µg kg^{-1}*).*

TABLE 54. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF PCB 170 REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
2	1.20	0.12	1.000	GCxGC-ECD	
4	0.93	0.06	0.100	GC-ECD	SRM1941b
5	0.80	0.03	0.003	GC-HRMS	NIST 1944
7	1.30	0.05	0.050	GC-MS/MS	
11	0.82	0.04	0.005	GC-HRMS	
13	1.04	0.20		GC-MS/MS	NIST1944
16	1.06	0.03	0.009	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

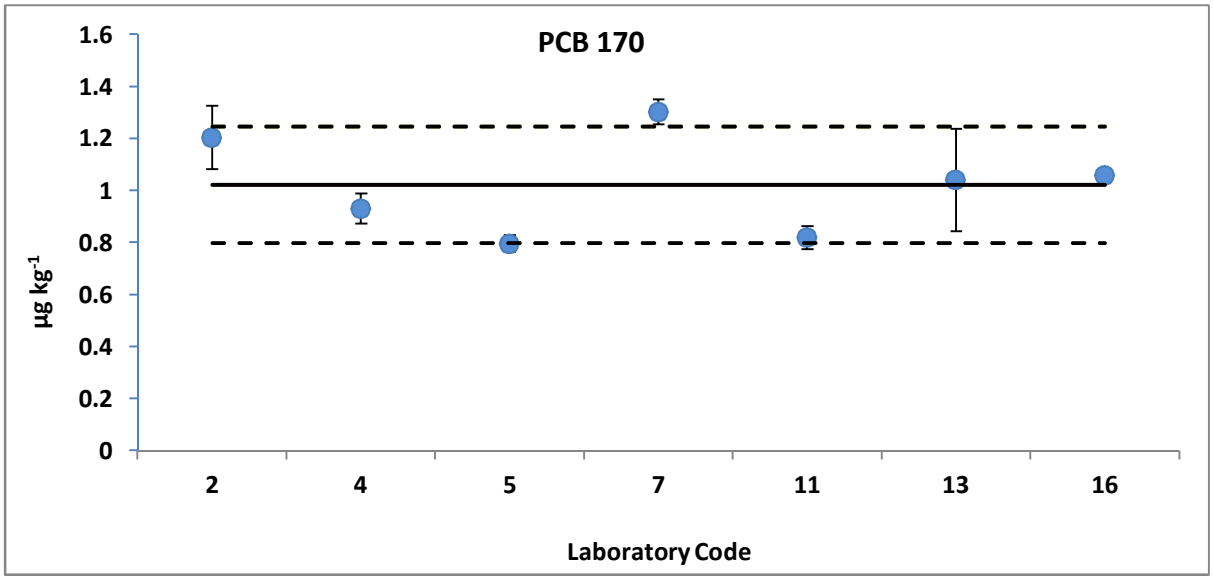


*FIG. 41. Laboratory results for PCB 170 in IAEA-459 (*µg kg^{-1}*).*

TABLE 55. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF PCB 180 REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
2	2.53	0.23	1.000	GCxGC-ECD	
3	1.07		0.001	GC-MS/MS	
4	2.16	0.09	0.100	GC-ECD	SRM1941b
5	2.50	0.11	0.003	GC-HRMS	NIST 1944
6	2.54	0.16	0.005	GC-MS/MS	IAEA-408
7	2.55	0.18	0.050	GC-MS/MS	Quasi QOR123MS
9	1.77	0.10	0.250	GC-MS	RM IAEA-159
10	1.86	0.03	0.020	GC-ECD	
11	2.74	0.09	0.005	GC-HRMS	
13	2.38	0.40		GC-MS/MS	NIST1944
15	1.72	0.02	0.100	GC-ECD	Quasi MS-2
16	2.19	0.06	0.016	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

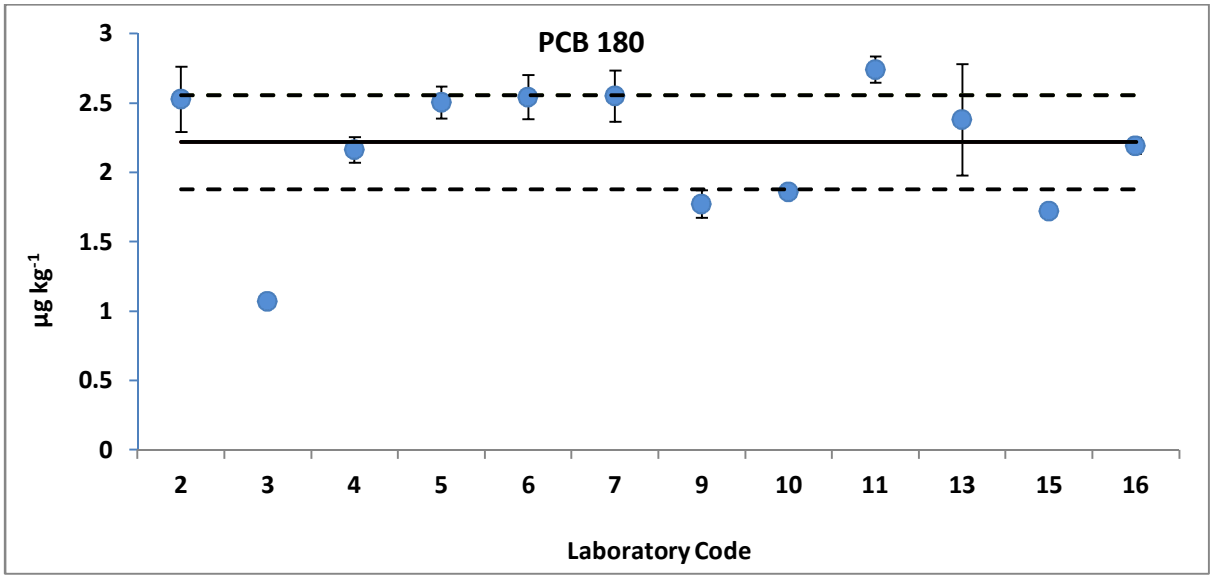


*FIG. 42. Laboratory results for PCB 180 in IAEA-459 (*µg kg^{-1}*).*

TABLE 56. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF PCB 183 REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
2	0.98	0.11	1.000	GCxGC-ECD	
5	0.67	0.04	0.003	GC-HRMS	NIST 1944
11	0.57	0.03	0.005	GC-HRMS	
13	0.49	0.08		GC-MS/MS	NIST1944
16	0.87	0.03	0.007	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

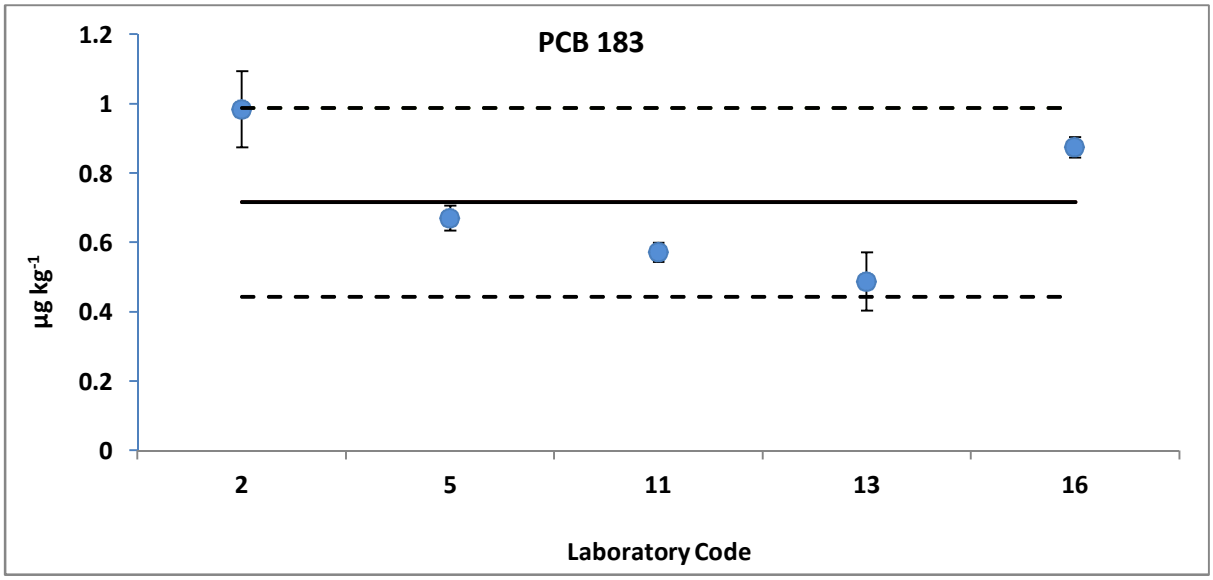


*FIG. 43. Laboratory results for PCB 183 in IAEA-459 (*µg kg^{-1}*).*

TABLE 57. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF PCB 187 REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
2	1.49	0.20	1.000	GCxGC-ECD	
4	1.28	0.05	0.100	GC-ECD	SRM1941b
5	1.53	0.07	0.003	GC-HRMS	NIST 1944
11	1.35	0.07	0.005	GC-HRMS	
13	1.04	0.14		GC-MS/MS	NIST1944
16	1.48	0.03	0.009	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

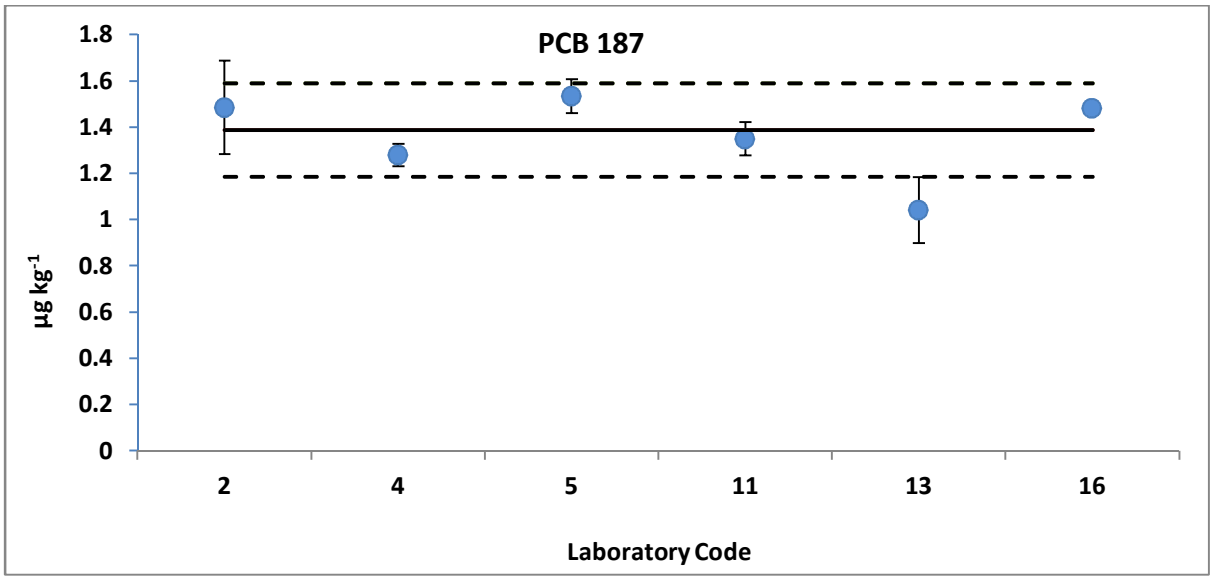


*FIG. 44. Laboratory results for PCB 187 in IAEA-459 (*µg kg^{-1}*).*

TABLE 58. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF PCB 209 REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
4	0.193	0.020	0.100	GC-ECD	SRM1941b
5	0.122	0.007	0.003	GC-HRMS	NIST 1944
7	0.198	0.004	0.050	GC-MS/MS	
11	0.210	0.002	0.010	GC-HRMS	
16	0.256	0.014	0.008	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

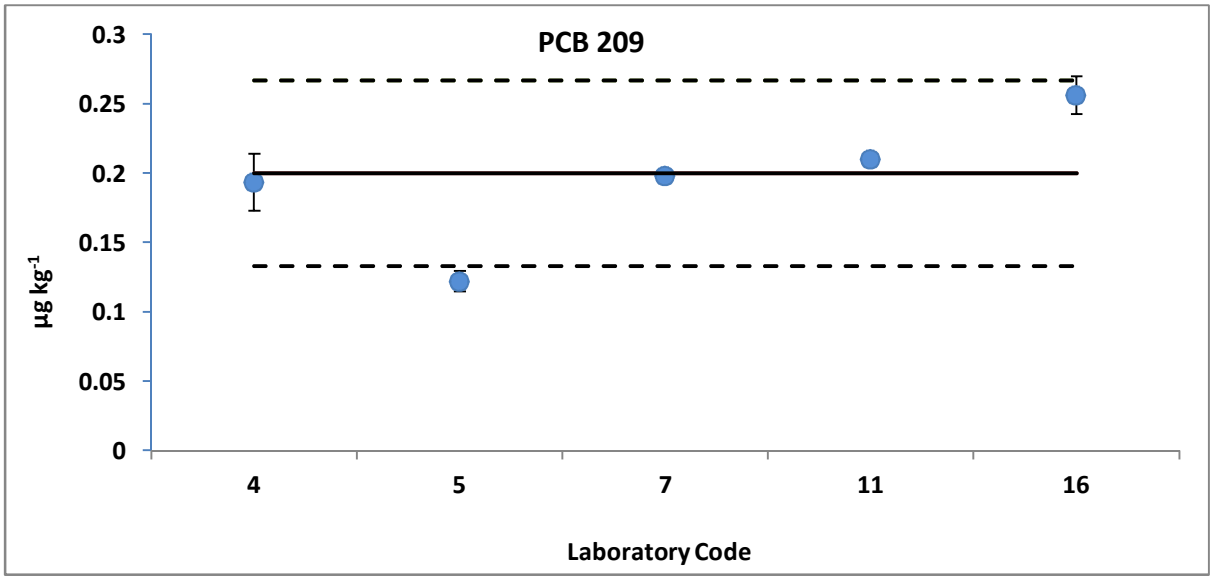


*FIG. 45. Laboratory results for PCB 209 in IAEA-459 (*µg kg^{-1}*).*

APPENDIX III

RESULTS FOR THE CERTIFIED MEASUREMENTS OF ORGANOCHLORINATED PESTICIDES

TABLE 59. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF op'- DDD REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
3	0.59		0.010	GC-MS/MS	
4	0.98	0.07	0.100	GC-ECD	
5	0.88	0.01	0.010	GC-HRMS	NIST 1944
7	0.95	0.07	0.035	GC-MS/MS	
11	0.97	0.11	0.010	GC-HRMS	
13	0.72	0.15		GC-MS/MS	NIST1944
15	0.28	0.06	0.100	GC-ECD	
16	0.46	0.02	0.007	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

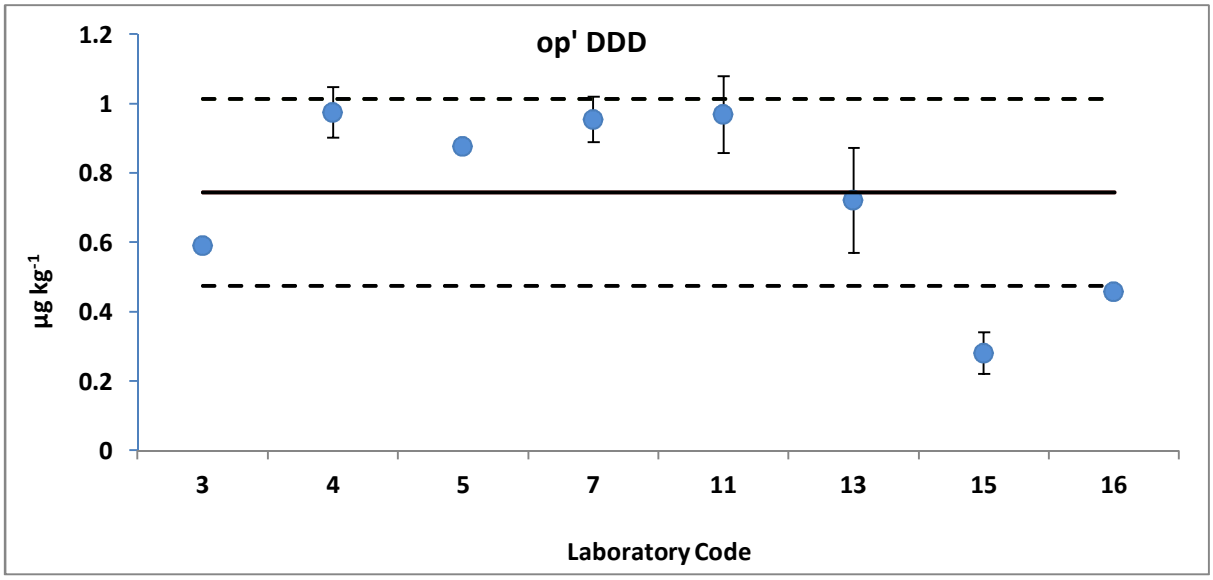


*FIG. 46. Laboratory results for op'-DDD in IAEA-459 (*µg kg^{-1}*).*

TABLE 60. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF op'- DDE REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
3	0.36		0.010	GC-MS/MS	
4	0.38	0.01	0.100	GC-ECD	
7	0.43	0.01	0.035	GC-MS/MS	
11	0.41	0.01	0.010	GC-HRMS	
13	0.67	0.13		GC-MS/MS	NIST1944
15	0.53	0.01	0.100	GC-ECD	
16	0.66	0.03	0.011	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

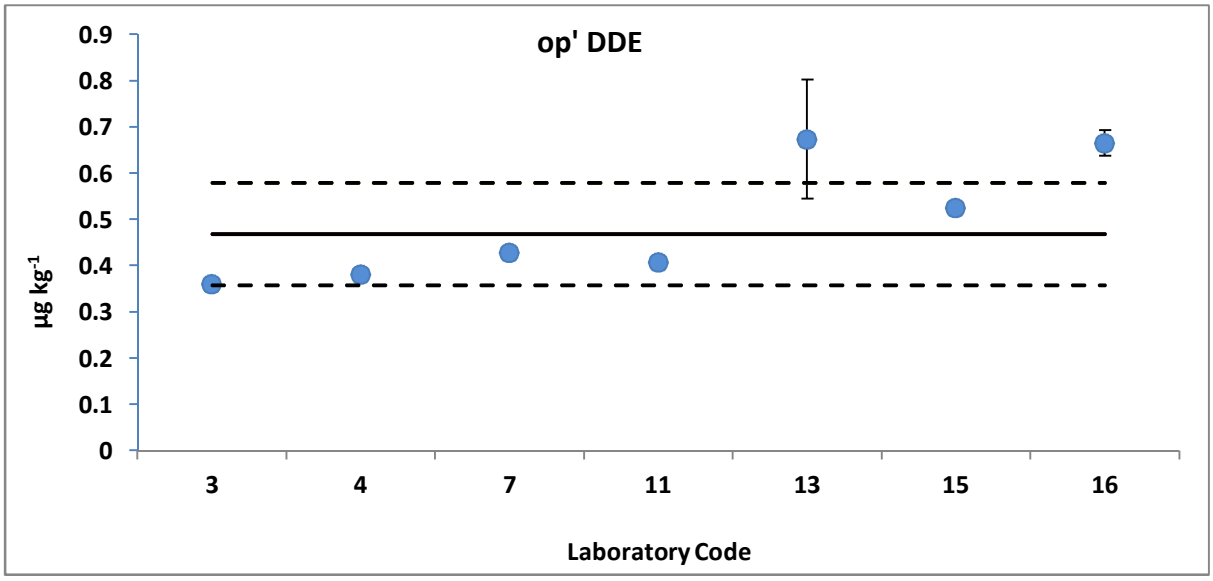


*FIG. 47. Laboratory results for op'-DDE in IAEA-459 (*µg kg^{-1}*).*

TABLE 61. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF op'- DDT REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
2**	0.58	0.05	1.000	GCxGC-ECD	
3	0.88		0.010	GC-MS/MS	
4	0.37	0.04	0.100	GC-ECD	
5	0.26	0.01	0.100	GC-HRMS	
7	0.36	0.04	0.035	GC-MS/MS	Quasi QOR123MS
11	0.28	0.02	0.020	GC-HRMS	
13	0.31	0.06		GC-MS/MS	NIST1944
16	0.41	0.05	0.020	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

** Not taken into account for assigned value calculation

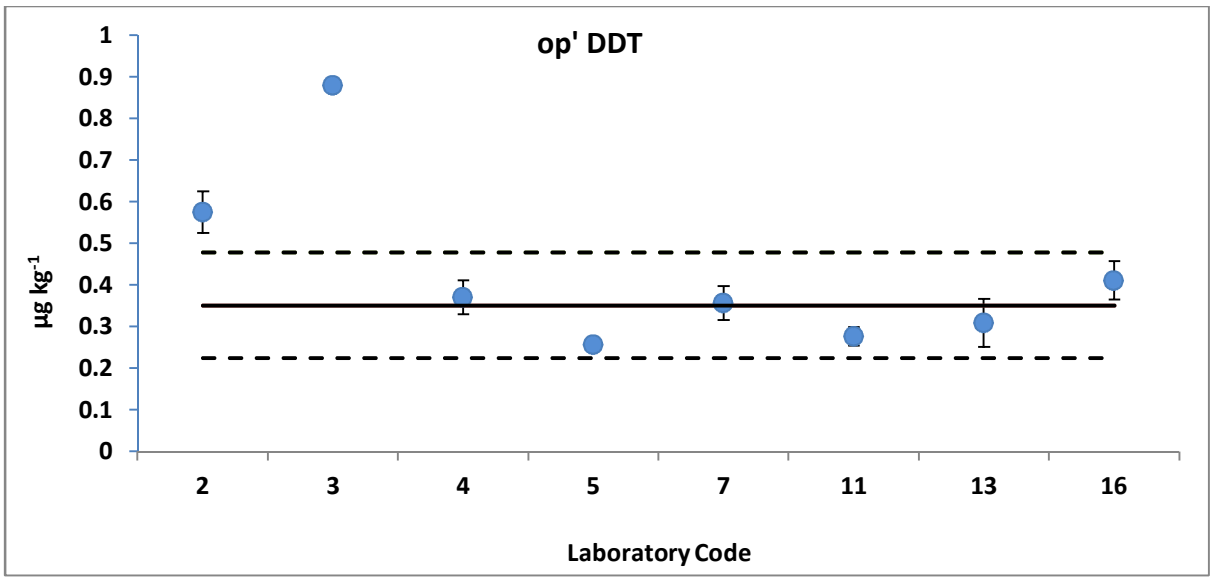


*FIG. 48. Laboratory results for op'-DDT in IAEA-459 (*µg kg^{-1}*).*

TABLE 62. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF pp'- DDE REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
2	3.76	0.42	1.000	GCxGC-ECD	
3	3.09		0.010	GC-MS/MS	
4	4.87	0.13	0.100	GC-ECD	SRM1941b
5	3.76	0.08	0.020	GC-HRMS	NIST 1944
6	3.68	0.11	0.002	GC-MS/MS	IAEA-408
7	3.91	0.12	0.035	GC-MS/MS	Quasi QOR123MS
9	2.76	0.05	0.100	GC-ECD	RM IAEA-159
10	3.703	0.004	0.020	GC-ECD	
11	3.69	0.17	0.040	GC-HRMS	
13	3.17	0.51		GC-MS/MS	NIST1944
15	2.36	0.38	0.100	GC-ECD	Quasi MS-2
16	4.26	0.10	0.015	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

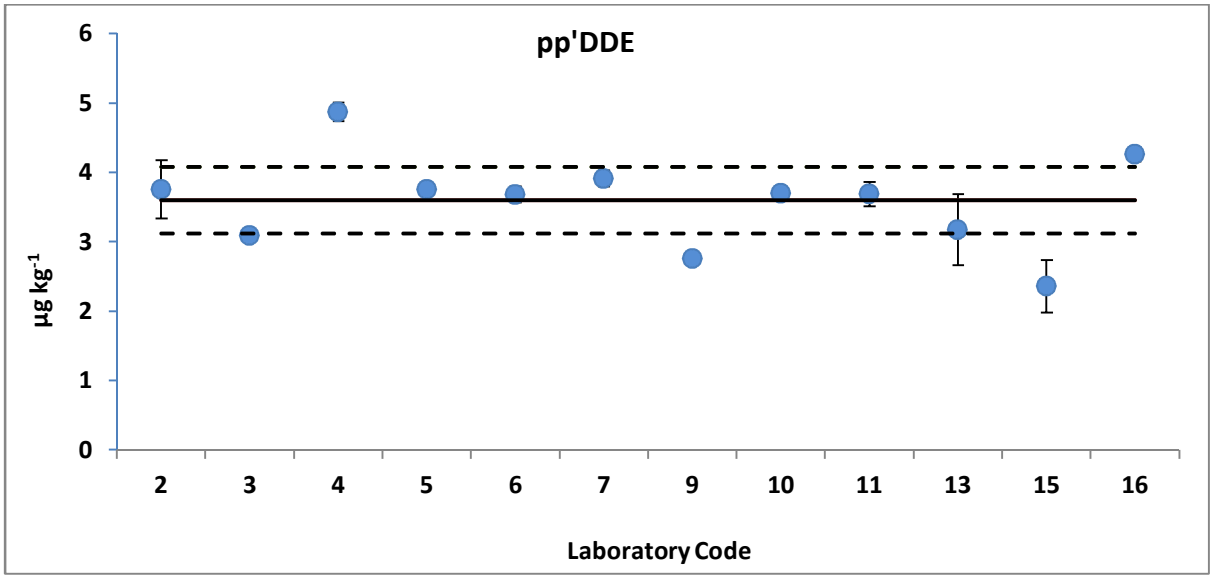


FIG. 49. Laboratory results for pp'-DDE in IAEA-459 (µg kg^{-1}).

TABLE 53. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF pp'- DDD REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
2	3.17	0.28	1.000	GCxGC-ECD	
3	1.86		0.010	GC-MS/MS	
4	4.45	0.22	0.100	GC-ECD	SRM1941b
5	3.47	0.05	0.010	GC-HRMS	NIST 1944
6	4.58	0.07	0.007	GC-MS/MS	IAEA-408
7	3.88	0.21	0.035	GC-MS/MS	Quasi QOR123MS
9	2.35	0.03	0.100	GC-ECD	RM IAEA-159
10	2.49	0.05	0.020	GC-ECD	
11	3.32	0.36	0.005	GC-HRMS	
13	2.89	0.74		GC-MS/MS	NIST1944
15	1.15	0.15	0.100	GC-ECD	Quasi MS-2
16	2.18	0.11	0.008	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

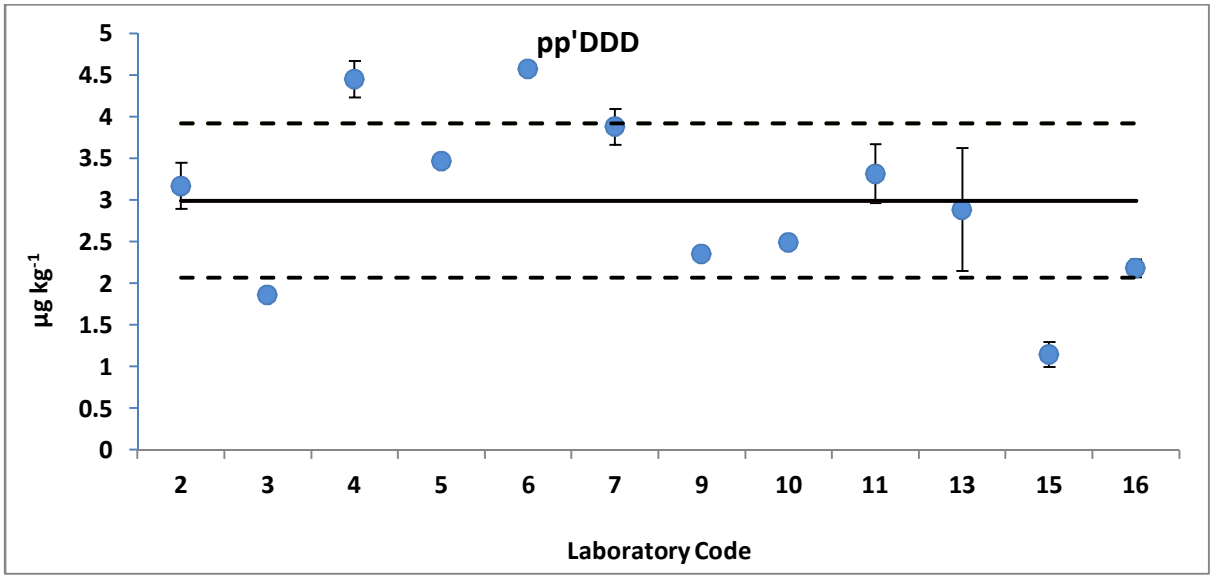


*FIG. 50. Laboratory results for pp'-DDD in IAEA-459 (*µg kg^{-1}*).*

TABLE 54. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF pp'- DDT REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
2	1.39	0.06	1.000	GCxGC-ECD	
3	2.56		0.010	GC-MS/MS	
4	1.01	0.09	0.100	GC-ECD	
5	1.16	0.05	0.020	GC-HRMS	NIST 1944
6	1.02	0.11	0.008	GC-MS/MS	IAEA-408
7	1.29	0.08	0.035	GC-MS/MS	Quasi QOR123MS
9	0.50	0.01	0.100	GC-ECD	
10	0.50	0.06	0.020	GC-ECD	
11	1.46	0.16	0.040	GC-HRMS	
13	1.52	0.34		GC-MS/MS	NIST1944
15	2.86	0.30	0.100	GC-ECD	Quasi MS-2
16	1.67	0.12	0.015	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

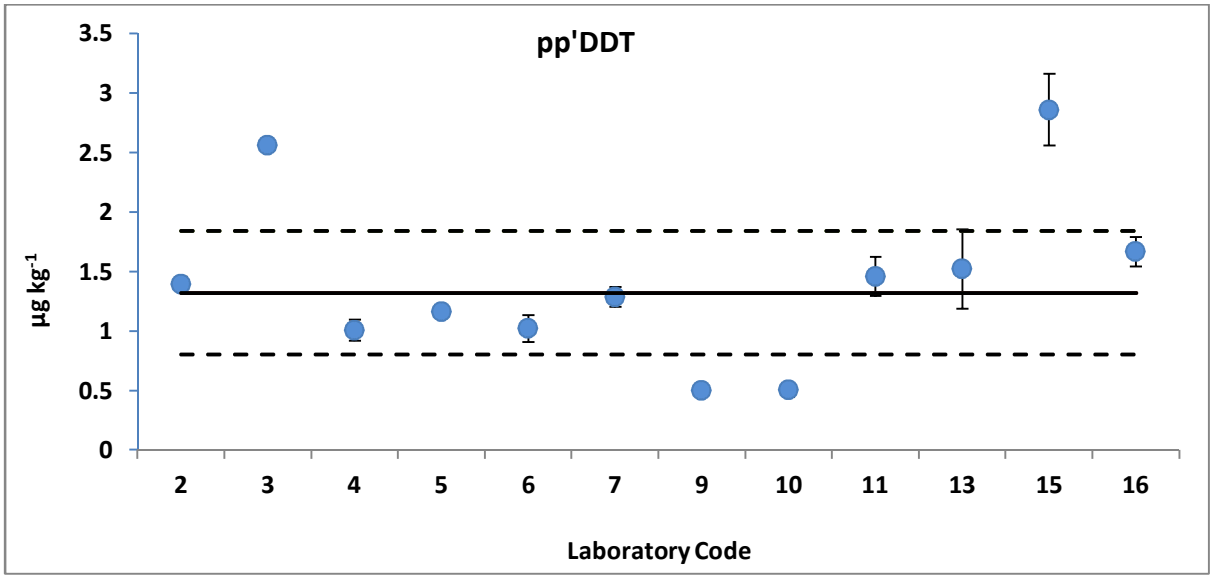


*FIG. 51. Laboratory results for pp'-DDT in IAEA-459 (*µg kg^{-1}*).*

APPENDIX IV

RESULTS FOR THE CERTIFIED MEASUREMENTS OF POLYBROMINATED DIPHENYL ETHERS (PBDEs)

TABLE 55. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF BDE 47 REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
2	0.634	0.293	0.401	GC-HRMS	In House Spike Solution
3	0.136	0.014	0.040	GC-MS-EI	
4	0.142	0.006	<0.02	GC-MS-NICI	
5	0.167	0.021	0.001	GC-HRMS	NIST 1944
10	0.188		0.020	MS-NICI	
11	0.138	0.005	0.003	GC-HRMS	
16	0.268	0.019	0.084	GC-ECD	

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

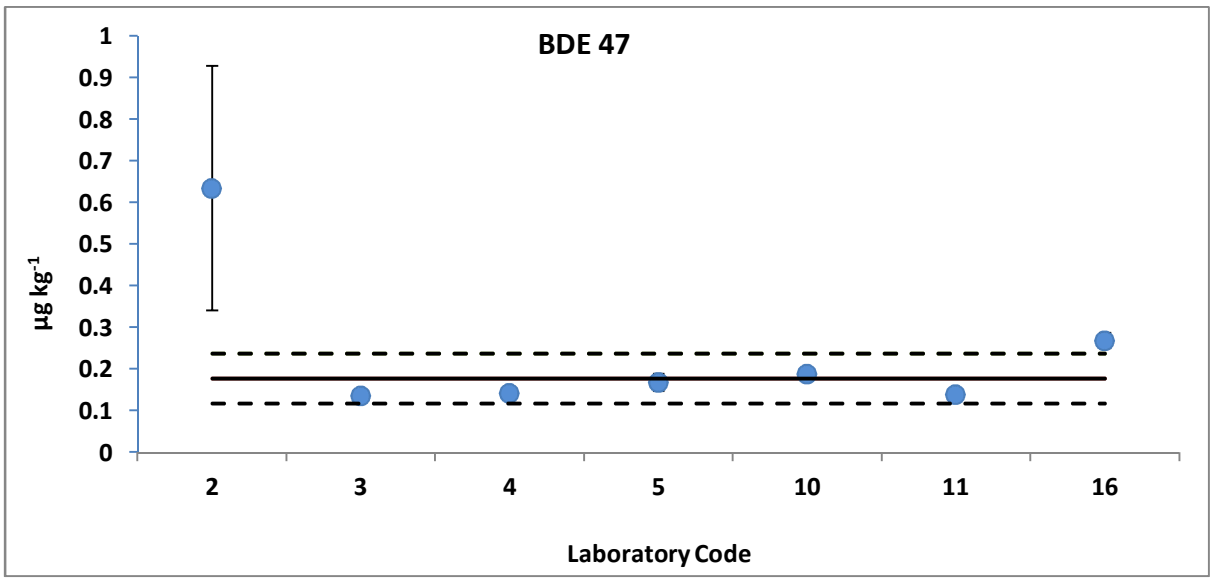


*FIG. 52. Laboratory results for BDE 47 in IAEA-459 (*µg kg^{-1}*).*

TABLE 56. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF BDE 99 REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
2	0.703	0.261	0.304	GC-HRMS	In House Spike Solution
3	0.231	0.062	0.030	GC-MS-EI	
4	0.198	0.030	<0.02	GC-MS-NICI	
5	0.224	0.025	0.002	GC-HRMS	NIST 1944
10	0.235		0.020	MS-NICI	
11	0.201	0.004	0.004	GC-HRMS	
16	0.293	0.017	0.063	GC-ECD	

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

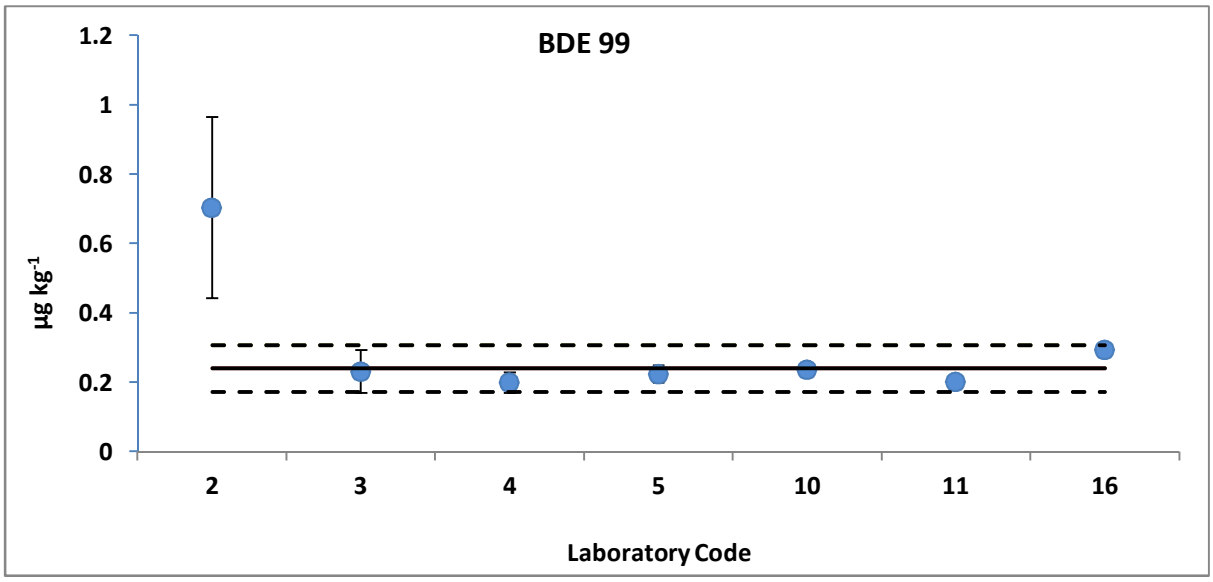


*FIG. 53. Laboratory results for BDE 99 in IAEA-459 (*µg kg^{-1}*).*

TABLE 67. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF BDE 153 REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
2	0.123	0.023	0.018	GC-HRMS	In House Spike Solution
3	0.093	0.009	0.040	GC-MS-EI	
4	0.095	0.020	<0.02	GC-MS-NICI	
5	0.091	0.011	0.002	GC-HRMS	NIST 1944
10	0.092		0.020	MS-NICI	
11	0.101	0.005	0.007	GC-HRMS	
16	0.195	0.016	0.001	GC-ECD	

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

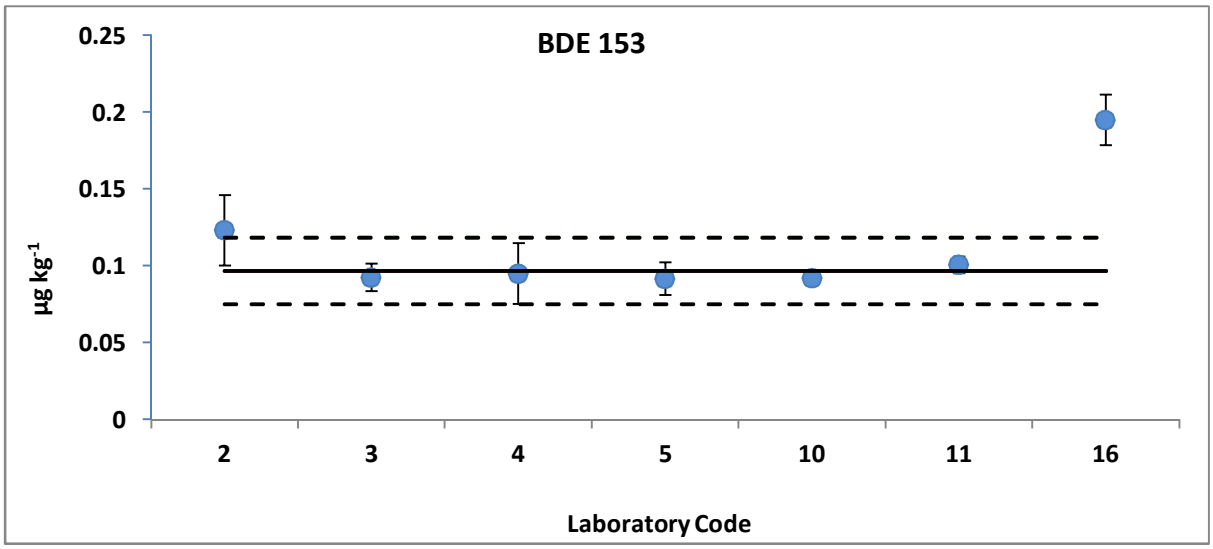


*FIG. 54. Laboratory results for BDE 153 in IAEA-459 (*µg kg^{-1}*).*

TABLE 68. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF BDE 183 REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
2	0.290	0.029	0.027	GC-HRMS	In House Spike Solution
3	0.183	0.063	0.007	GC-MS-EI	
4	0.418	0.042	<0.02	GC-MS-NICI	
5	0.275	0.017	0.003	GC-HRMS	NIST 1944
10	0.299		0.020	MS-NICI	
11	0.286	0.023	0.010	GC-HRMS	
16	0.181	0.033	0.003	GC-ECD	

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

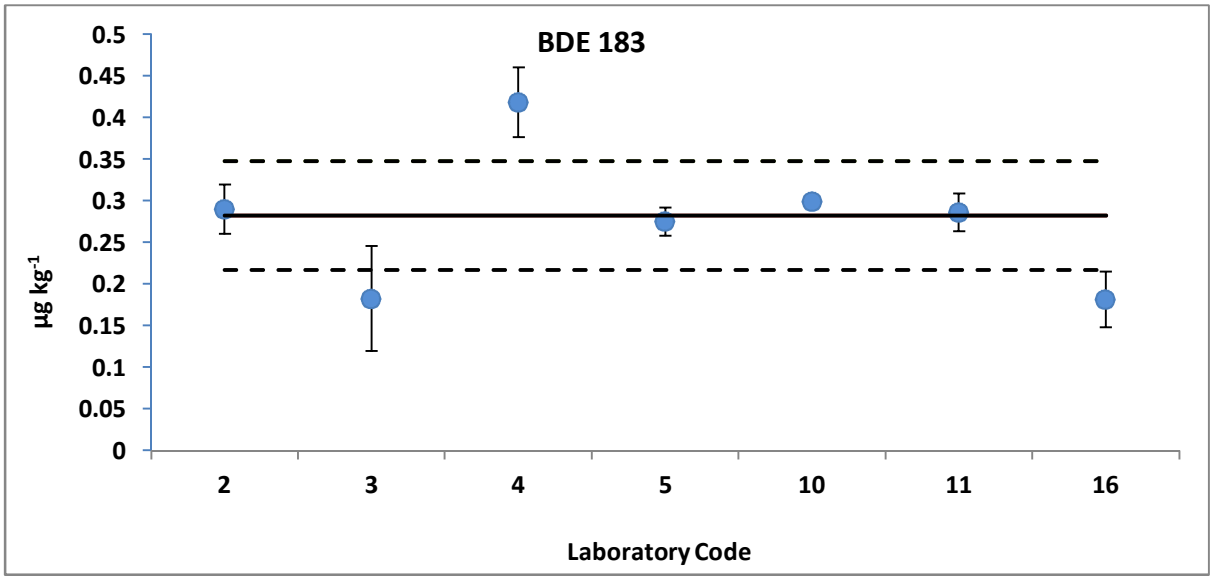


*FIG. 55. Laboratory results for BDE 183 in IAEA-459 (*µg kg^{-1}*).*

TABLE 69. RESULTS USED FOR THE CALCULATION OF CERTIFIED MASS FRACTION VALUES OF BDE 209 REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
2	11.75	0.81	0.51	GC-HRMS	In House Spike Solution
3	16.48	2.91	0.61	GC-MS-EI	
4	8.79	1.63	<0.02	GC-MS-NICI	
5	11.91	2.54	0.10	GC-HRMS	NIST 1944
10	10.49		0.02	MS-NICI	
11	10.18	0.24	0.07	GC-HRMS	
16	9.29	2.76	0.04	GC-ECD	

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

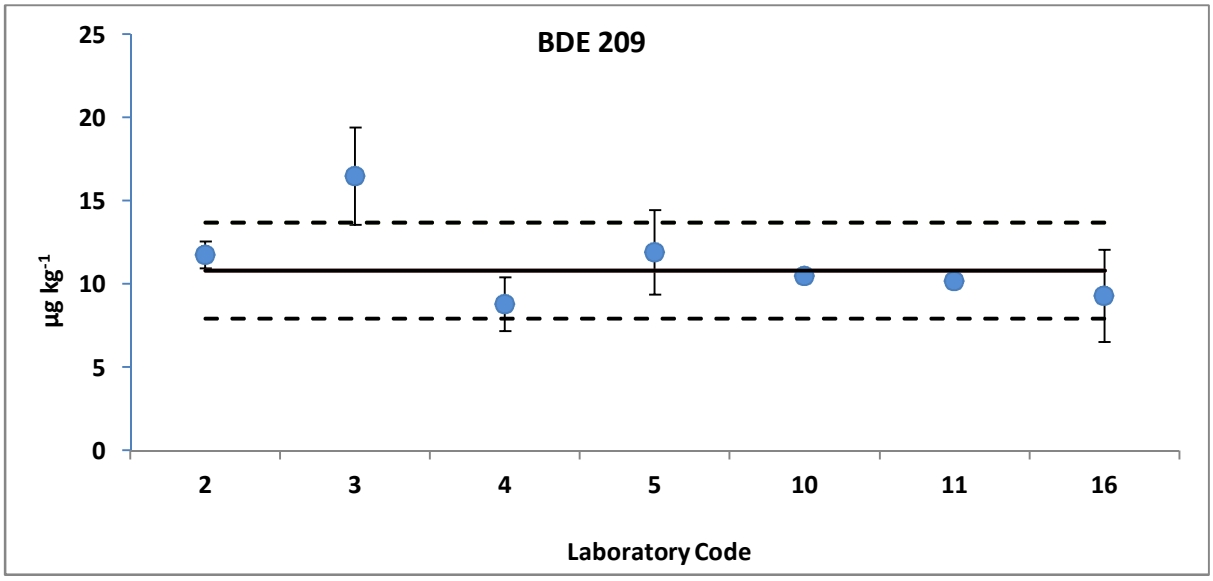


*FIG. 56. Laboratory results for BDE 209 in IAEA-459 (*µg kg^{-1}*).*

APPENDIX V

RESULTS FOR THE INFORMATION MEASUREMENTS OF POLYCYCLIC AROMATIC HYDROCARBONS (PAHs)

TABLE 70. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF NAPHTHALENE REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
3	48.02		0.10	GC-MS	
4	9.20	0.63	5.00	GC-MS	SRM1941b
6	16.78	0.98	0.26	GC-MS/MS	IAEA-408
7	11.59	0.99	0.50	HPLC-FLD	Quasimeme QPH087
8	34	11	3.36	GC-MS	NIST 1944
9	12.28	0.34	1.00	GC-MS	RM IAEA-159
10	23.61		0.50	GC-MS	IMR LRM
11	22.52	1.98	0.40	GC-HRMS	
13	19.40	2.41	0.10	GC-MS	NIST 1944
16	24.64	3.92	0.02	GC-MS	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants

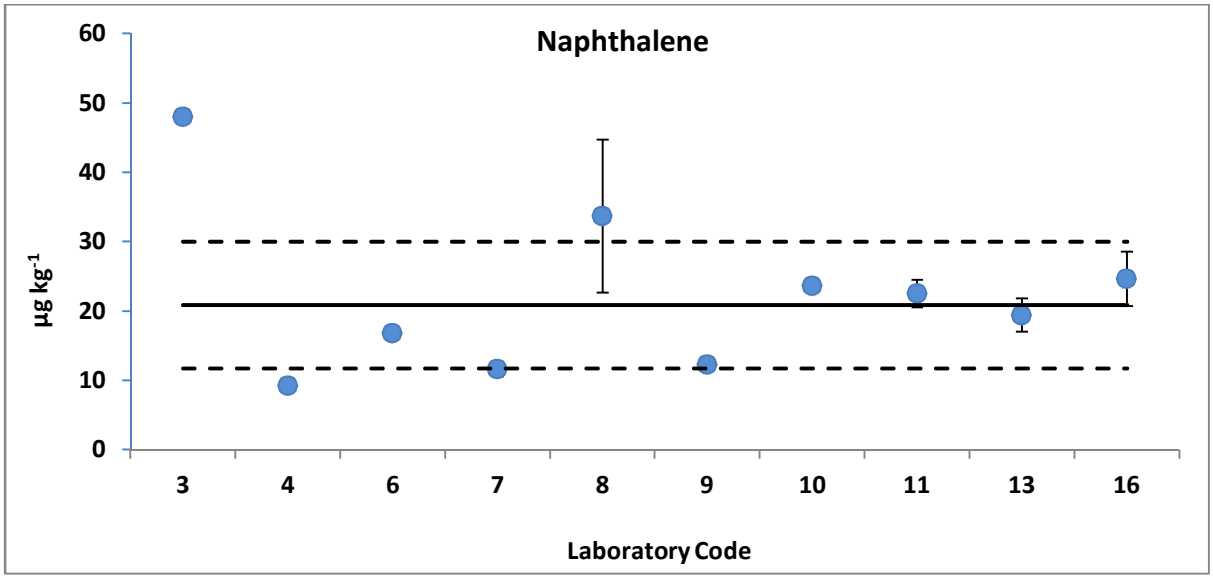


*FIG. 57. Laboratory results for Naphthalene in IAEA-459 (*µg kg^{-1}*).*

TABLE 71. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF C2-NAPHTHALENES REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
4	30.58	1.88	1.00	GC-MS	SRM1941b
6	47.97	2.70	0.07	GC-MS/MS	IAEA-408
11	75.31	2.00	0.60	GC-HRMS	
13	37.89	1.33	0.10	GC-MS	NIST 1944
16	85.23	7.33	0.03	GC-MS	NIST1941B

*Calculated as: 2 x $\frac{S}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

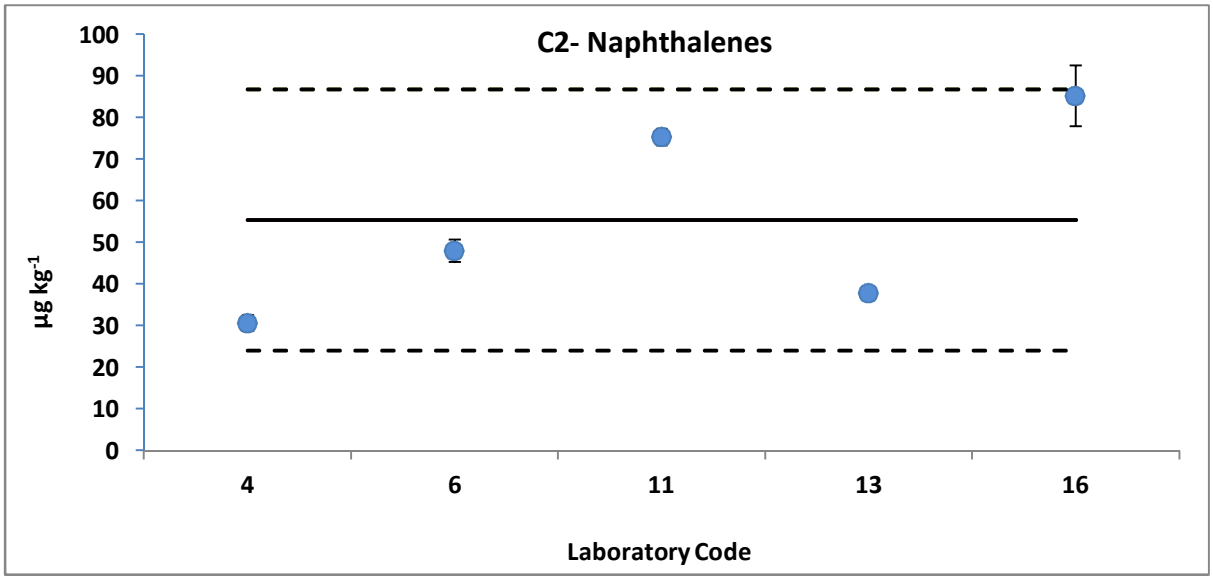


*FIG. 58. Laboratory results for C2- Naphthalenes in IAEA-459 (*µg kg^{-1}*).*

TABLE 72. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF C3-NAPHTHALENES REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
4	45.83	4.10	1.00	GC-MS	SRM1941b
6	50.29	1.52	0.25	GC-MS/MS	IAEA-408
11	60.83	1.69	0.60	GC-HRMS	
13	79.54	4.71	0.10	GC-MS	NIST 1944
16	108	12	0.03	GC-MS	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

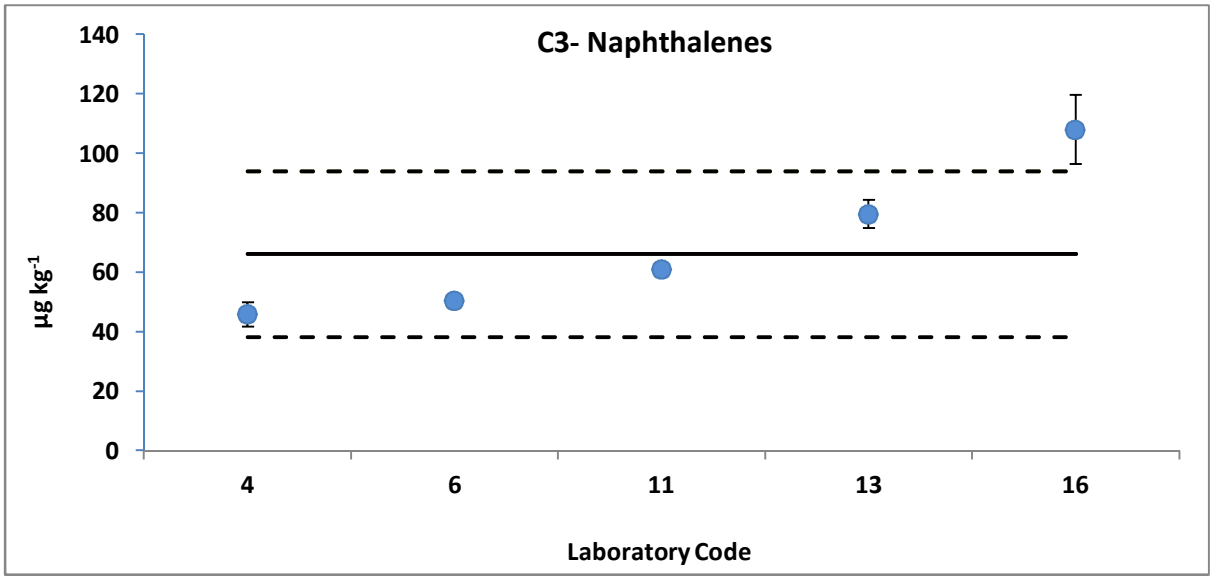


*FIG. 59. Laboratory results for C3- Naphthalenes in IAEA-459 (*µg kg^{-1}*).*

TABLE 73. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF BIPHENYL REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
10	10.33		0.50	GC-MS	IMR LRM
11	13.09	1.55	0.40	GC-HRMS	
16	9.85	0.58	0.01	GC-MS	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

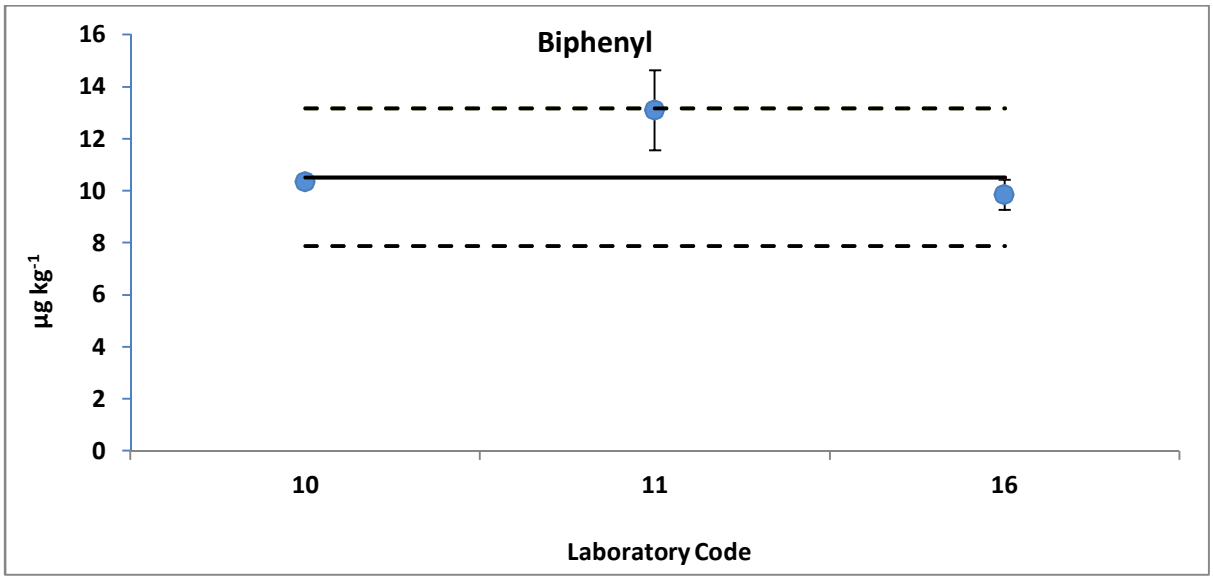


*FIG. 60. Laboratory results for Biphenyl in IAEA-459 (*µg kg^{-1}*).*

TABLE 74. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF C1-FLUORENES REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
11	6.52	0.75	0.60	GC-HRMS	
16	15.65	0.95	0.02	GC-MS	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

TABLE 75. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF C2-FLUORENES REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
11	6.96	1.11	0.60	GC-HRMS	
16	36.75	1.89	0.02	GC-MS	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

TABLE 76. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF C3-FLUORENES REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
11	7.94	0.38	0.60	GC-HRMS	
16	52.23	2.50	0.02	GC-MS	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

TABLE 77. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF C1-DIBENZOTHIOPHENES REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
4	19.58	1.48	1.00	GC-MS	SRM1941b
11	33.38	3.74	0.40	GC-HRMS	
13	38.37	1.00	0.20	GC-MS	NIST 1944
16	41.88	1.42	0.01	GC-MS	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

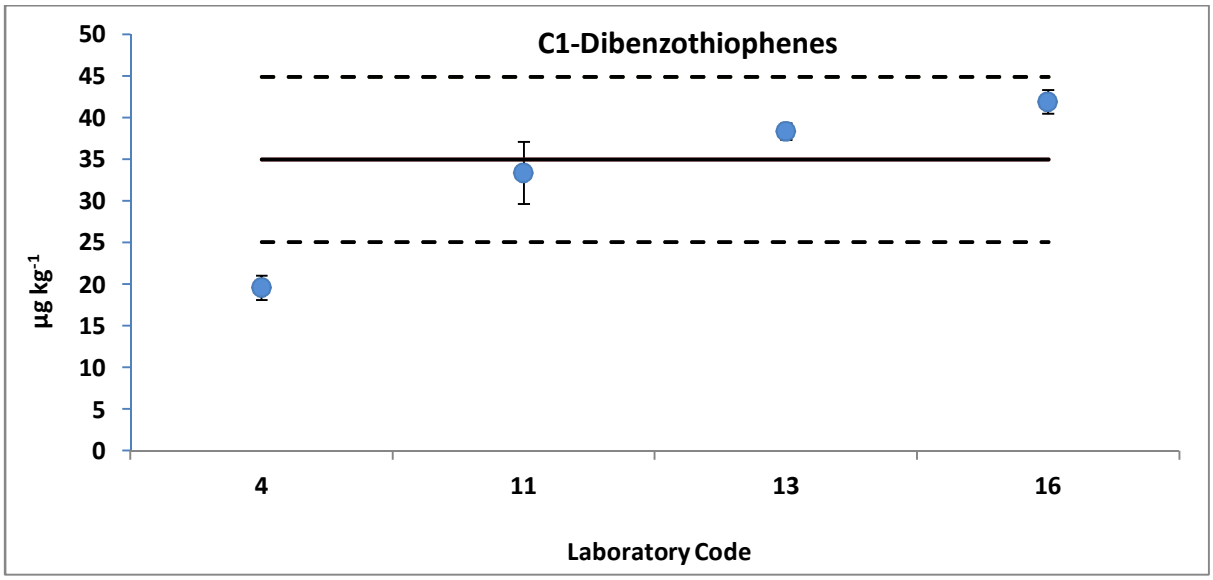


*FIG. 61. Laboratory results for C1-Dibenzothiophenes in IAEA-459 (*µg kg^{-1}*).*

TABLE 78. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF C2-DIBENZOTHIOPHENES REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
4	53.61	4.24	4.00	GC-MS	SRM1941b
11	45.80	5.78	0.60	GC-HRMS	
13	69.86	4.07	0.20	GC-MS	NIST 1944
16	81.91	1.43	0.01	GC-MS	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

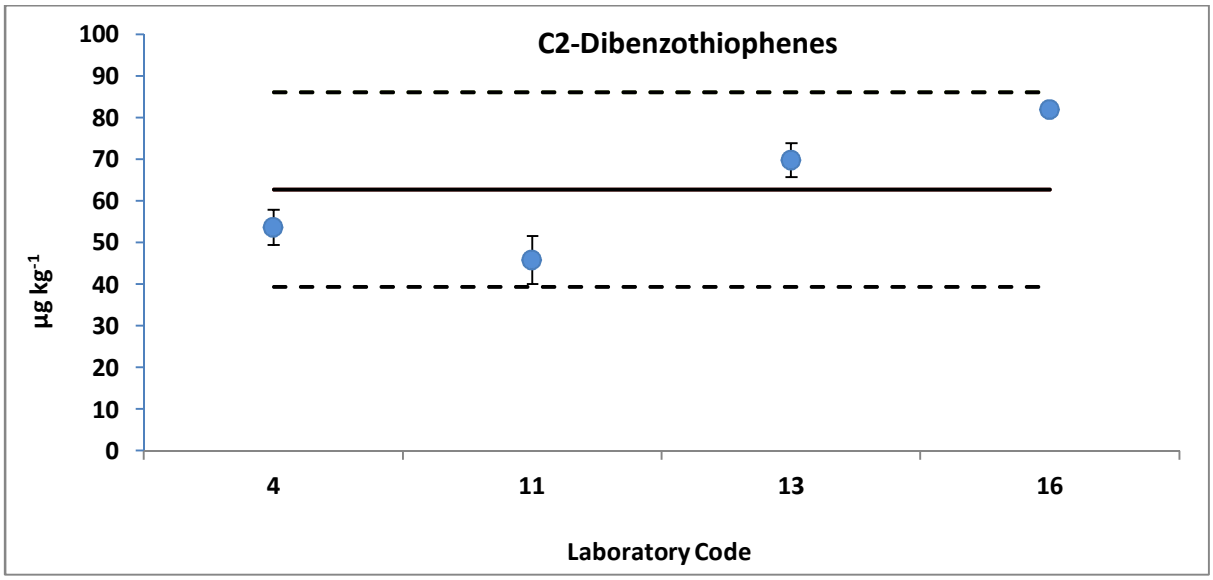


*FIG. 62. Laboratory results for C2-Dibenzothiophenes in IAEA-459 (*µg kg^{-1}*).*

TABLE 79. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF C3-DIBENZOTHIOPHENES REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
4	69.67	5.30	4.00	GC-MS	SRM1941b
11	128.80	4.79	0.60	GC-HRMS	
13	80.46	4.09	0.20	GC-MS	NIST 1944
16	116.29	3.46	0.01	GC-MS	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

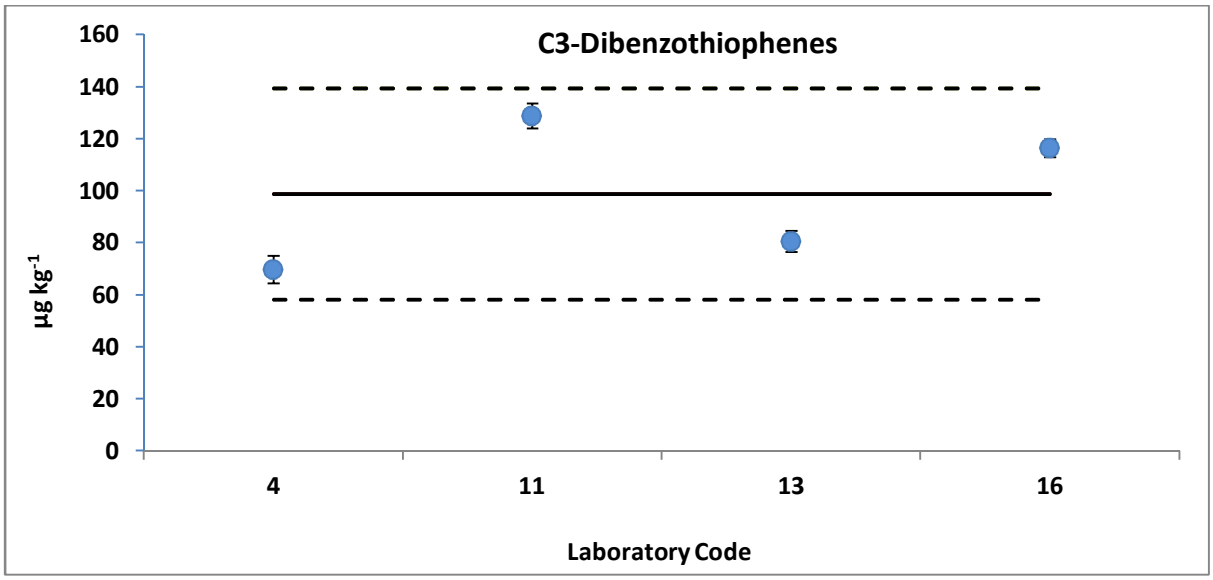


*FIG. 63. Laboratory results for C3-Dibenzothiophenes in IAEA-459 (*µg kg^{-1}*).*

TABLE 80. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF 1-METHYLPHENANTHRENE REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
4	5.55	0.28	2.00	GC-MS	SRM1941b
8	3.50	0.23	0.16	GC-MS	NIST 1944
10	7.97		0.50	GC-MS	IMR LRM
11	5.55	0.37	0.40	GC-HRMS	
13	19.03	0.78	0.10	GC-MS	NIST 1944
16	16.08	0.56	0.02	GC-MS	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported by participants.

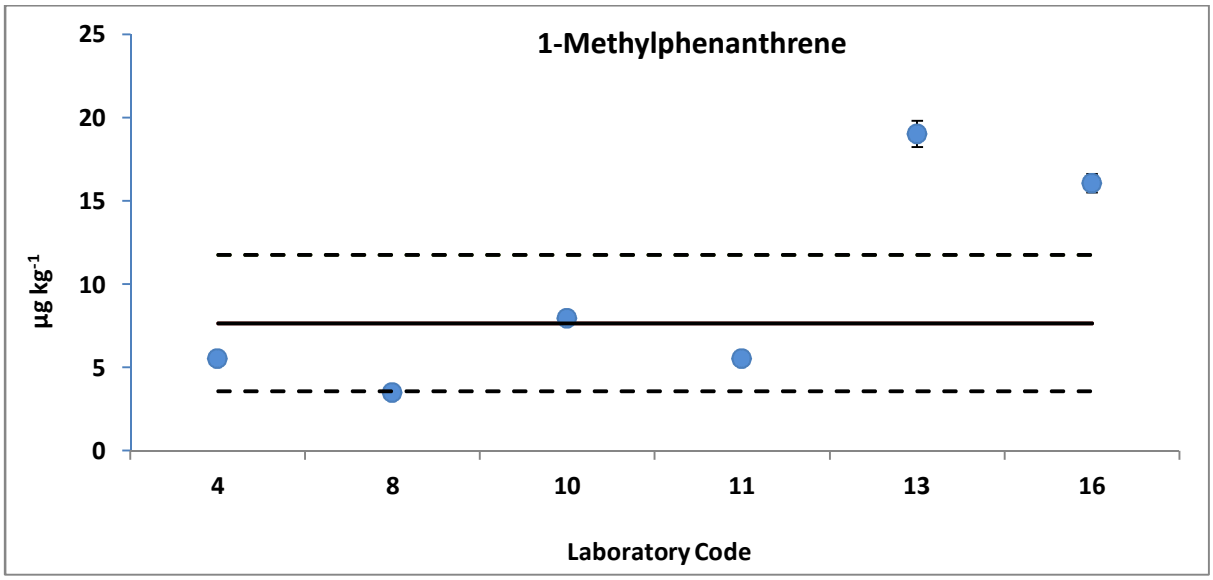


*FIG. 64. Laboratory results for 1-Methylphenanthrene in IAEA-459 (*µg kg^{-1}*).*

TABLE 81. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF 2-METHYLPHENANTHRENE REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
4	12.73	0.49	2.00	GC-MS	SRM1941b
8	24.80	1.53	0.16	GC-MS	NIST 1944
10	20.27		0.50	GC-MS	IMR LRM
11	11.28	1.59	0.40	GC-HRMS	
16	32.49	0.95	0.02	GC-MS	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported by participants.

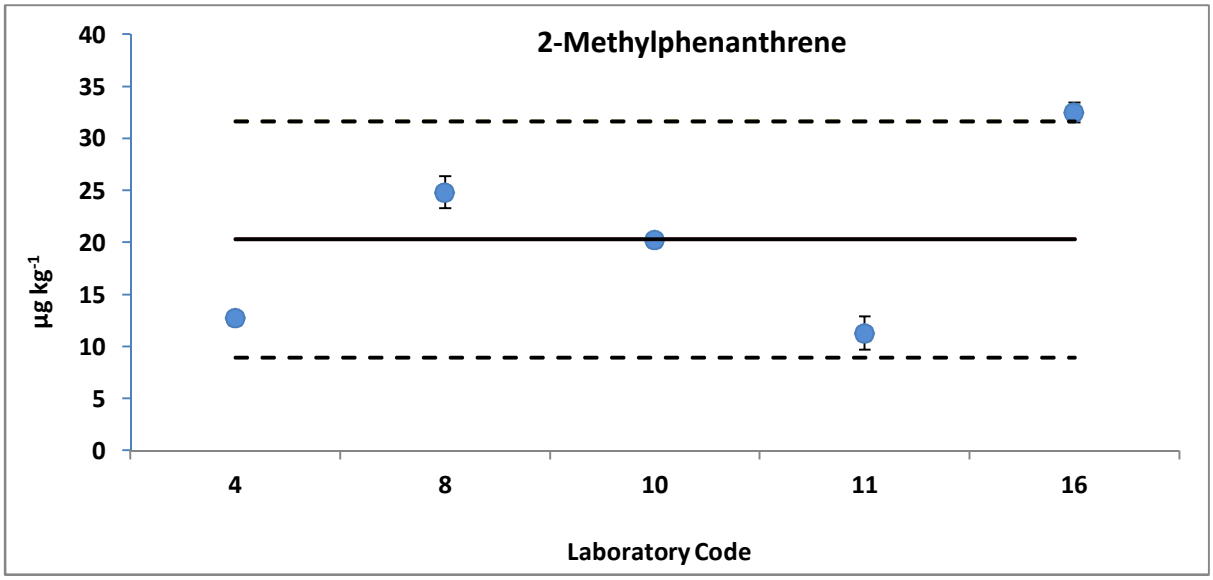


*FIG. 65. Laboratory results for 2-Methylphenanthrene in IAEA-459 (*µg kg^{-1}*).*

TABLE 82. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF C1-PHEN/ANTH REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
4	32.46	1.51	2.00	GC-MS	SRM1941b
6	40.28	1.27	0.45	GC-MS/MS	IAEA-408
11	29.24	3.00	0.40	GC-HRMS	
13	59.72	4.55	0.10	GC-MS	NIST 1944
16	64.35	1.30	0.02	GC-MS	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

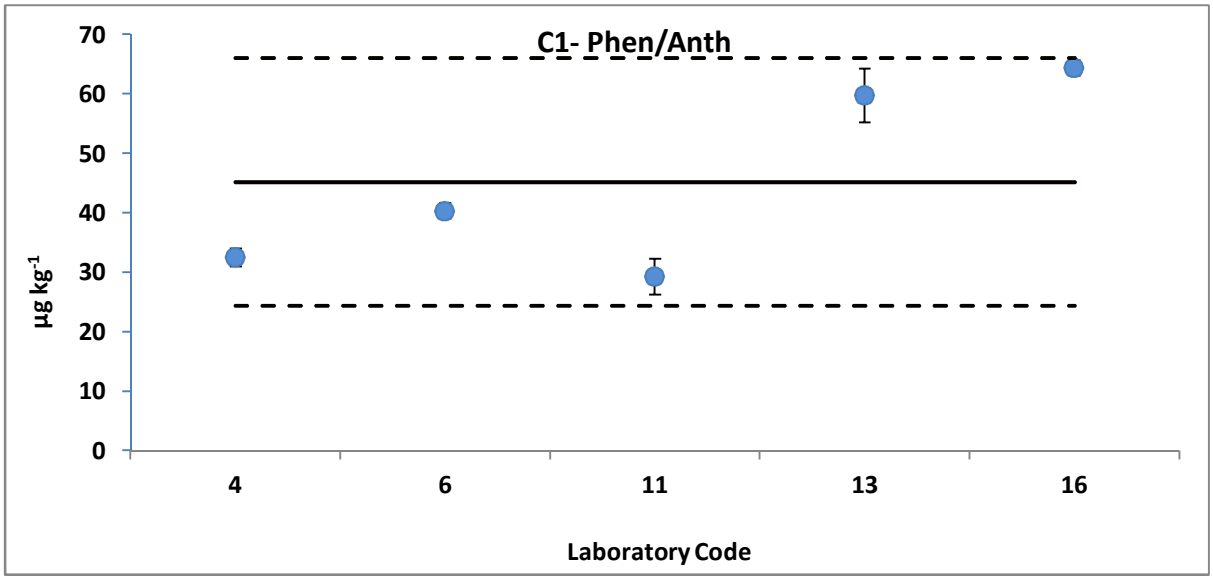


*FIG. 66. Laboratory results for C1- Phen/Anth in IAEA-459 (*µg kg^{-1}*).*

TABLE 83. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF C2-PHEN/ANTH REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
4	41.57	2.60	2.00	GC-MS	SRM1941b
6	52.64	1.90	0.29	GC-MS/MS	IAEA-408
11	32.32	2.25	0.50	GC-HRMS	
13	62.90	2.21	0.10	GC-MS	NIST 1944
16	46.95	0.96	0.01	GC-MS	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

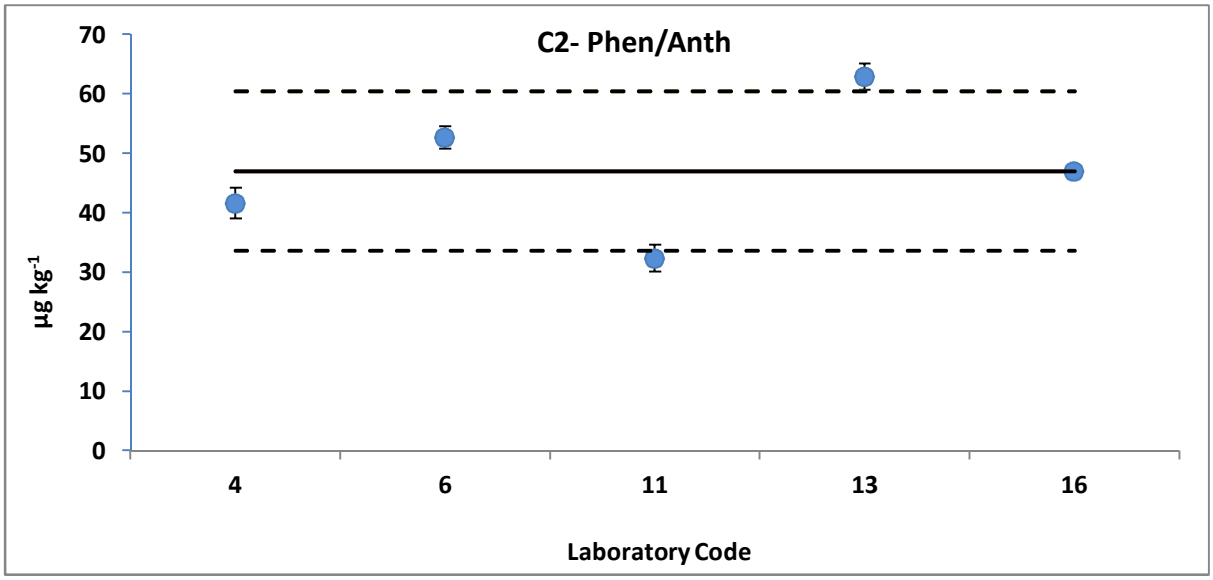


*FIG. 67. Laboratory results for C2- Phen/Anth in IAEA-459 (*µg kg^{-1}*).*

TABLE 84. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF C3-PHEN/ANTH REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
4	34.53	2.05	2.00	GC-MS	SRM1941b
6	35.90	0.51	0.09	GC-MS/MS	IAEA-408
11	40.77	1.08	0.50	GC-HRMS	
16	48.28	0.76	0.01	GC-MS	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

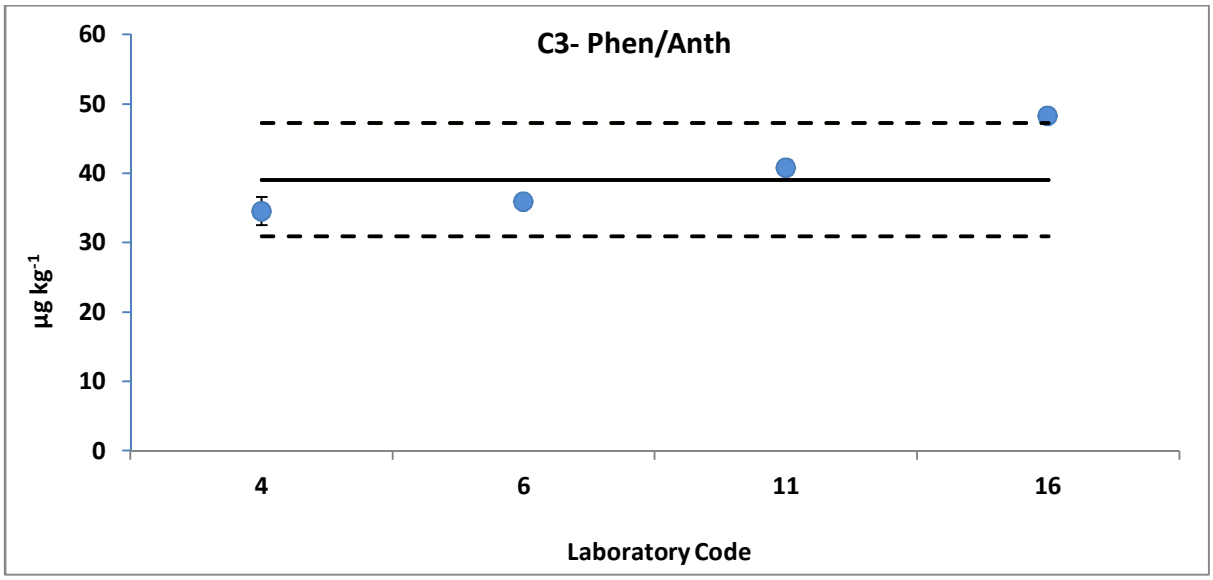


FIG. 68. Laboratory results for C3- Phen/Anth in IAEA-459 (µg kg^{-1}).

TABLE 85. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF C4-PHEN/ANTH REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
4	32.39	1.93	2.00	GC-MS	SRM1941b
11	28.49	3.79	0.50	GC-HRMS	
16	59.48	1.87	0.01	GC-MS	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

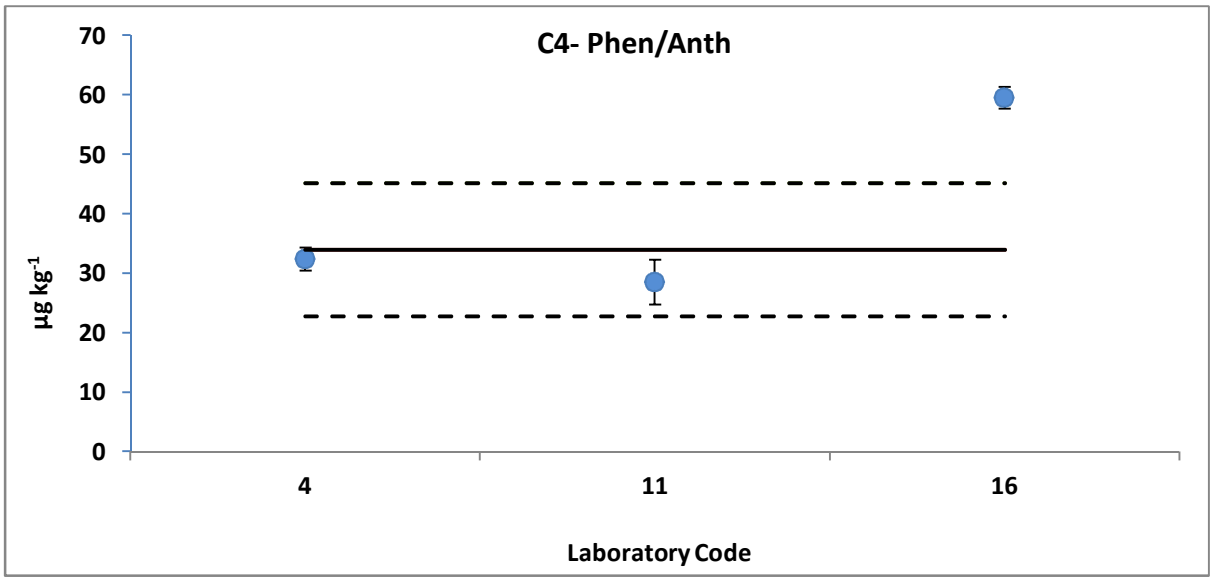


*FIG. 69. Laboratory results for C4- Phen/Anth in IAEA-459 (*µg kg^{-1}*).*

TABLE 86. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF 1-METYLPYRENE REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
11	10.38	0.82	0.40	GC-HRMS	
13	8.39	0.18	0.10	GC-MS	NIST 1944
16	8.65	0.51	0.01	GC-MS	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

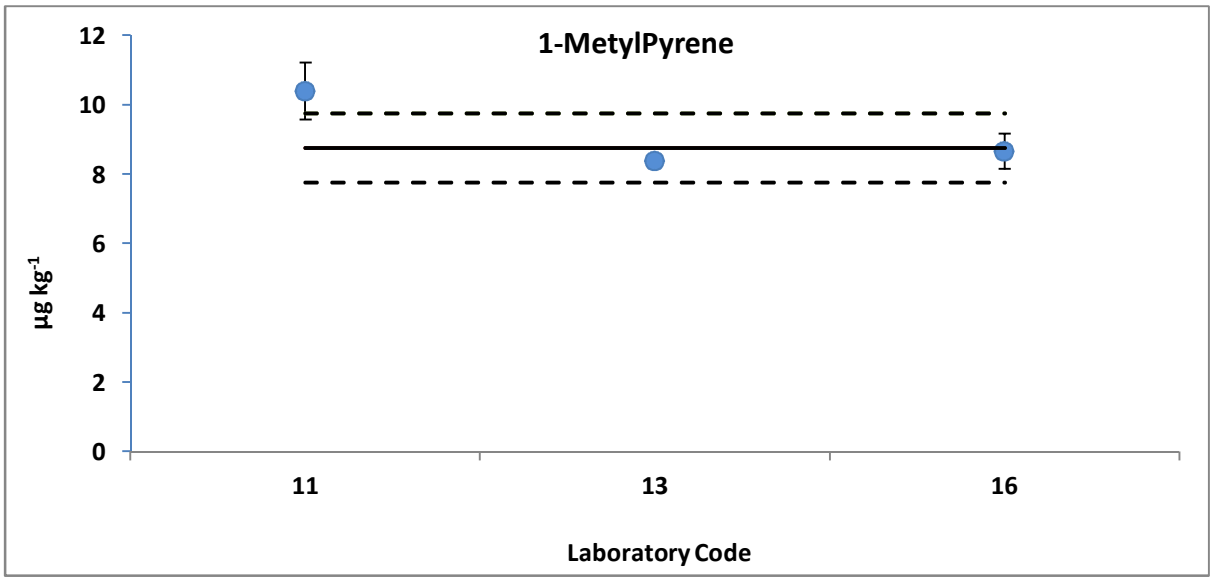


FIG. 70. Laboratory results for 1-MetylPyrene in IAEA-459 (µg kg^{-1}).

TABLE 87. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF C1-FLUORANTHENES/PYRENES REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
4	29.87	1.81	1.00	GC-MS	SRM1941b
11	49.59	4.99	0.50	GC-HRMS	
13	46.65	1.56	0.10	GC-MS	NIST 1944
16	42.09	1.92	0.01	GC-MS	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

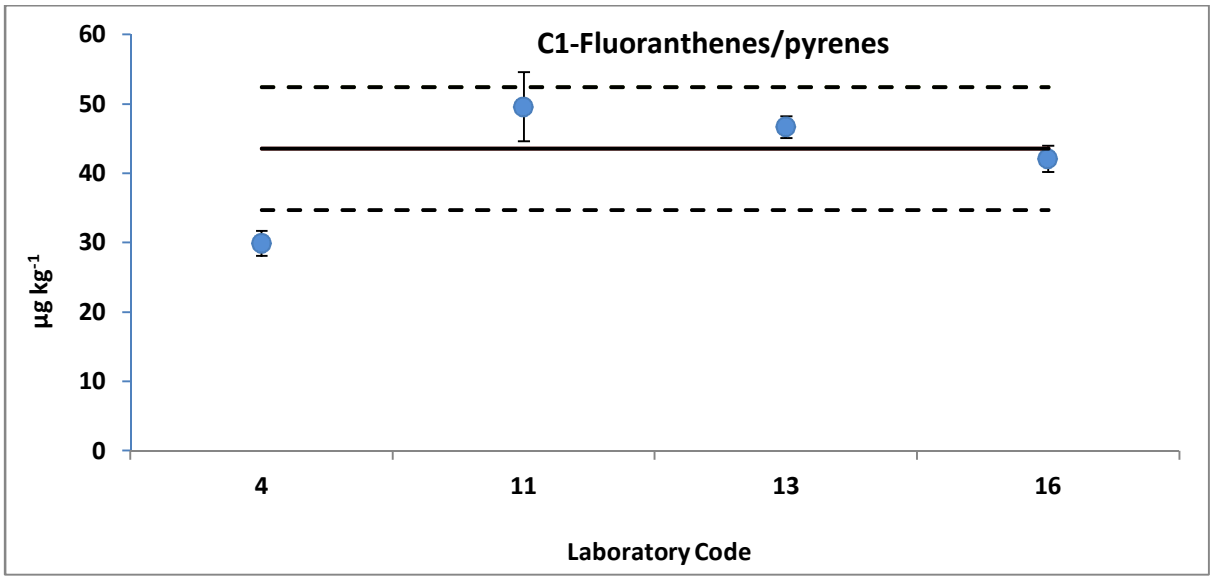


*FIG. 71. Laboratory results for C1-Fluoranthenes/pyrenes in IAEA-459 (*µg kg^{-1}*).*

TABLE 88. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF C2-FLUORANTHENES/PYRENES REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
4	33.99	1.88	2.00	GC-MS	SRM1941b
11	48.98	4.53	0.50	GC-HRMS	
13	53.89	0.99	0.10	GC-MS	NIST 1944
16	49.57	2.61	0.03	GC-MS	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

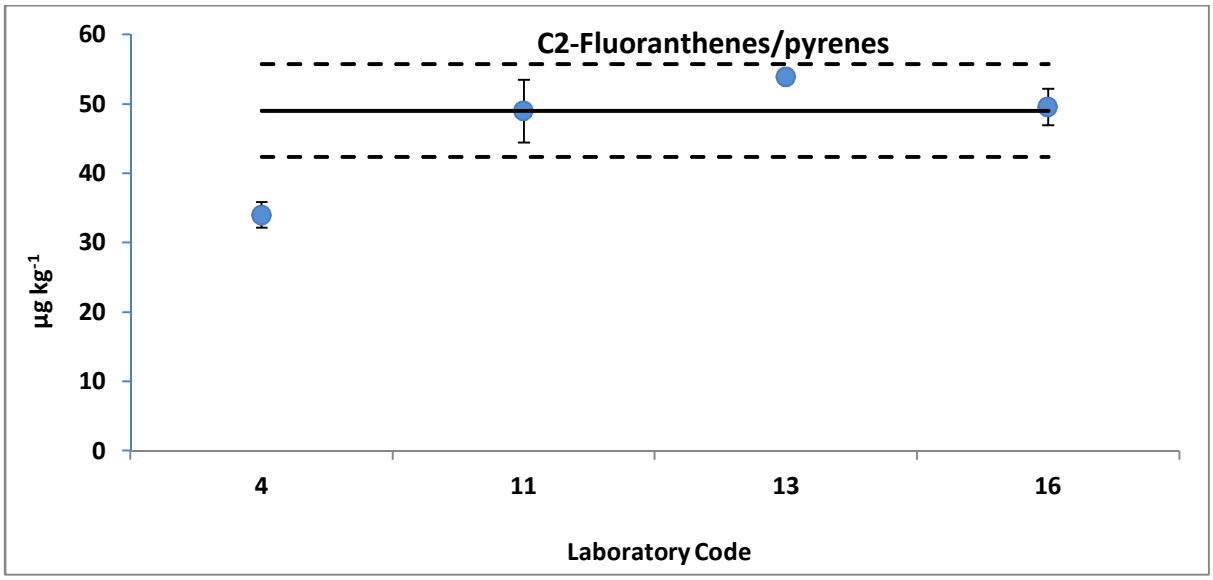


*FIG. 72. Laboratory results for C2-Fuloranthenes/pyrenes in IAEA-459 (*µg kg^{-1}*).*

TABLE 89. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF C3-FLUORANTHENES/PYRENES REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
4	33.42	2.16	4.00	GC-MS	SRM1941b
11	38.56	2.21	0.60	GC-HRMS	

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

TABLE 90. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF CHRYSENE REPORTED BY PARTICIPANTS (µg kg^{-1}

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
4	16.53	0.58	1.00	GC-MS	SRM1941b
7	20.53	0.87	0.50	HPLC-FLD	Quasimeme QPH087
10	17.46		0.50	GC-MS	IMR LRM
13	19.14	0.99	0.10	GC-MS	NIST 1944

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

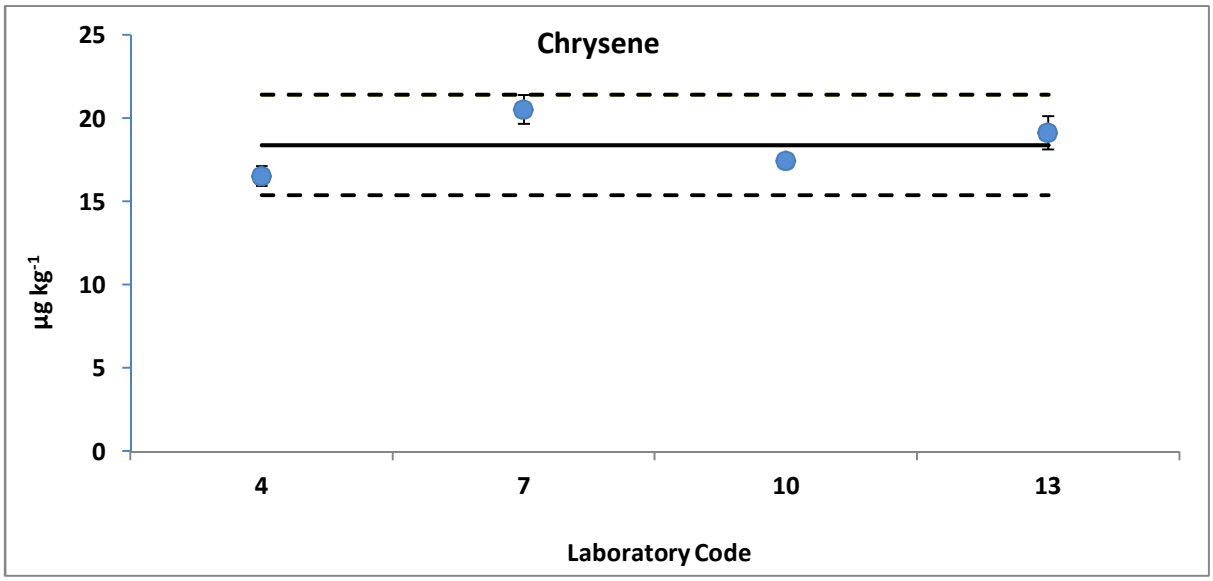


*FIG. 73. Laboratory results for Chrysene in IAEA-459 (*µg kg^{-1}*).*

TABLE 91. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF TRIPHENYLENE REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
4	7.98	0.23	1.00	GC-MS	SRM1941b

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

TABLE 92. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF C1-CHRYSENES REPORTED BY PARTICIPANTS (ng g^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
4	33.33	1.64	2.00	GC-MS	SRM1941b
11	41.34	2.12	0.50	GC-HRMS	
13	33.94	1.19	0.10	GC-MS	NIST 1944
16	35.45	2.32	0.01	GC-MS	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

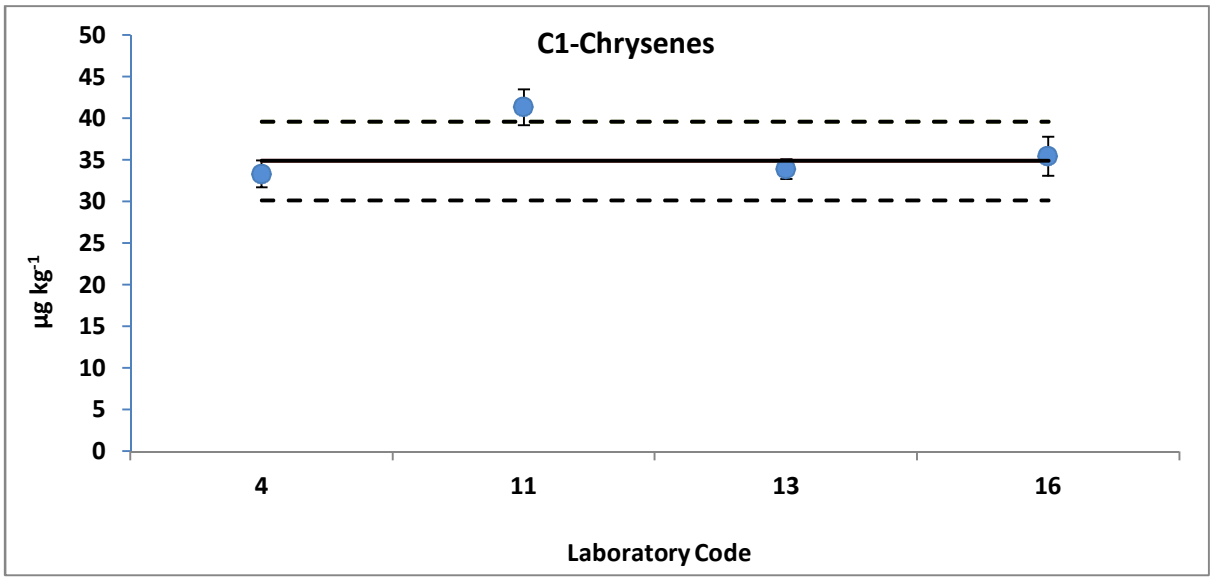


*FIG. 74. Laboratory results for C1-Chrysenes in IAEA-459 (*µg kg^{-1}*).*

TABLE 93. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF C2-CHRYSENES REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
4	44.54	2.40	2.00	GC-MS	SRM1941b
11	50.86	1.03	0.50	GC-HRMS	
16	54.46	1.65	0.01	GC-MS	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

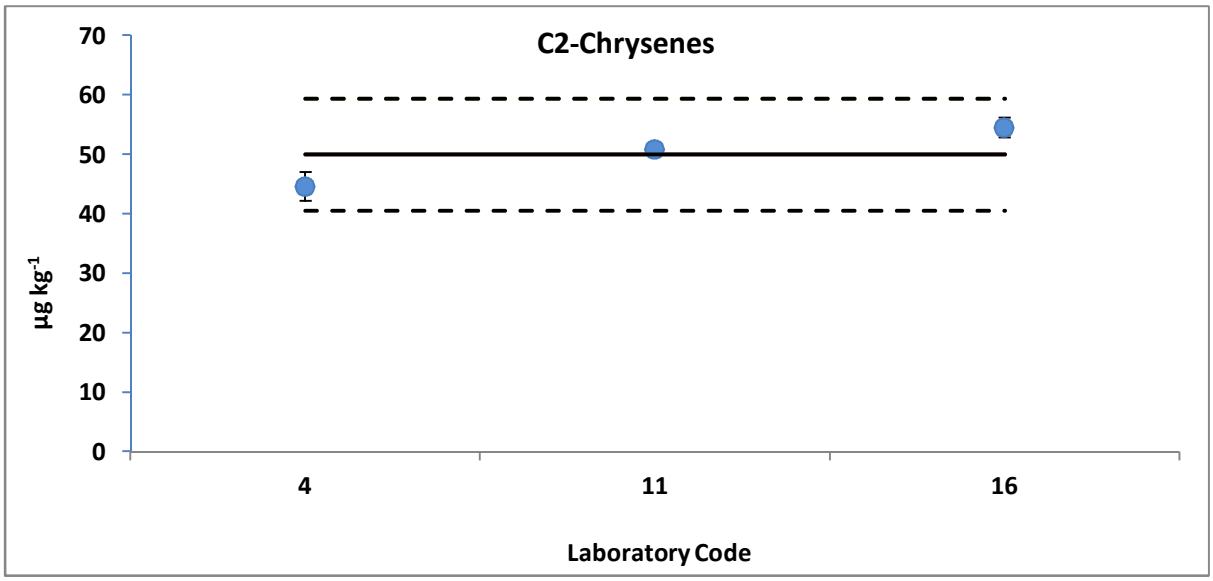


*FIG. 75. Laboratory results for C2-Chrysenes in IAEA-459 (*µg kg^{-1}*).*

TABLE 94. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF C3-CHRYSENES REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
4	36.72	1.68	2.00	GC-MS	SRM1941b
11	40.29	2.70	0.70	GC-HRMS	
16	41.66	1.15	0.01	GC-MS	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

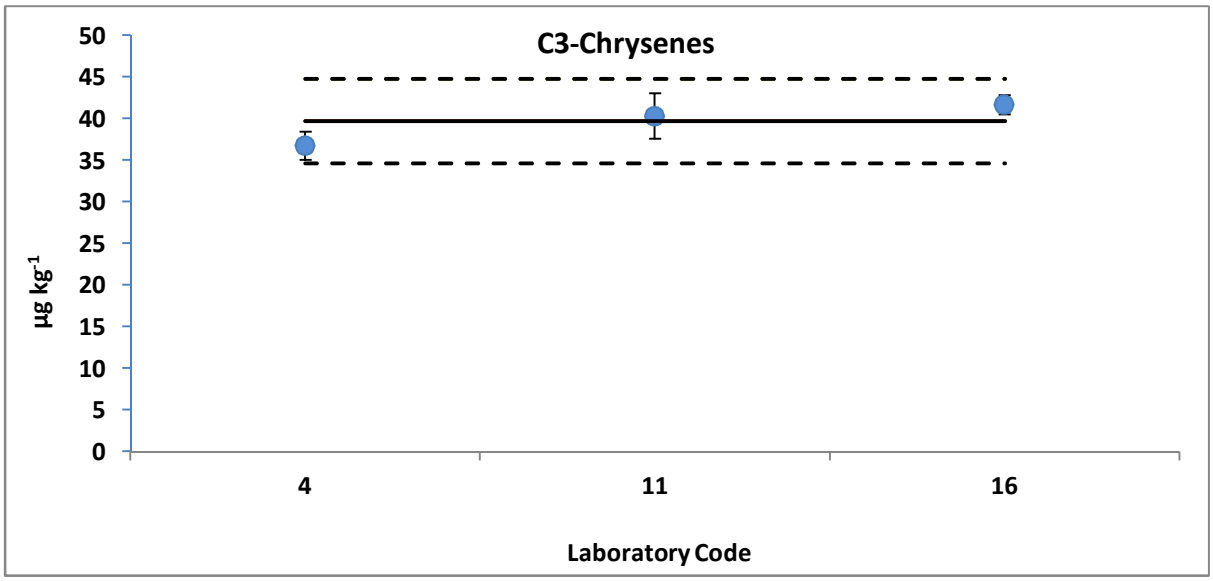


*FIG. 76. Laboratory results for C3-Chrysenes in IAEA-459 (*µg kg^{-1}*).*

TABLE 95. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF BENZO(*j*)FLUORANTHENE REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
4	15.04	0.40	2.00	GC-MS	SRM1941b
10	18.82		0.50	GC-MS	IMR LRM
11	29.34	2.10	0.40	GC-HRMS	

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

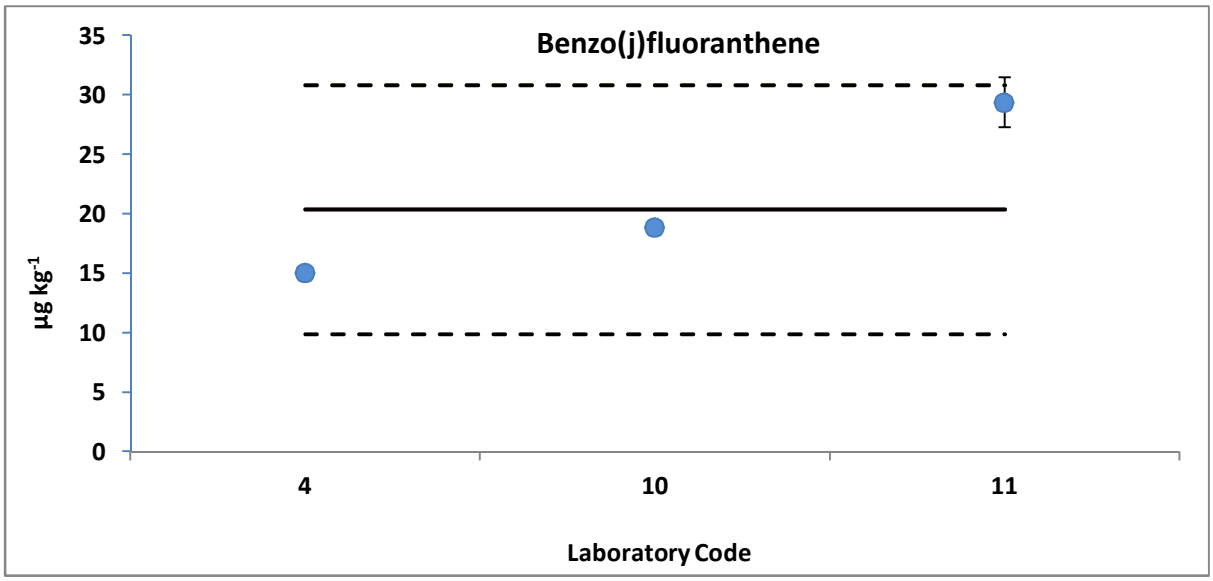


*FIG. 77. Laboratory results for Benzo(j)fluoranthene in IAEA-459 (*µg kg^{-1}*).*

TABLE 96. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF BENZO(*a*)FLUORANTHENE REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
4	4.12	0.18	2.00	GC-MS	SRM1941b
11	6.77	0.79	0.40	GC-HRMS	
16	10.13	0.57	0.03	GC-MS	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

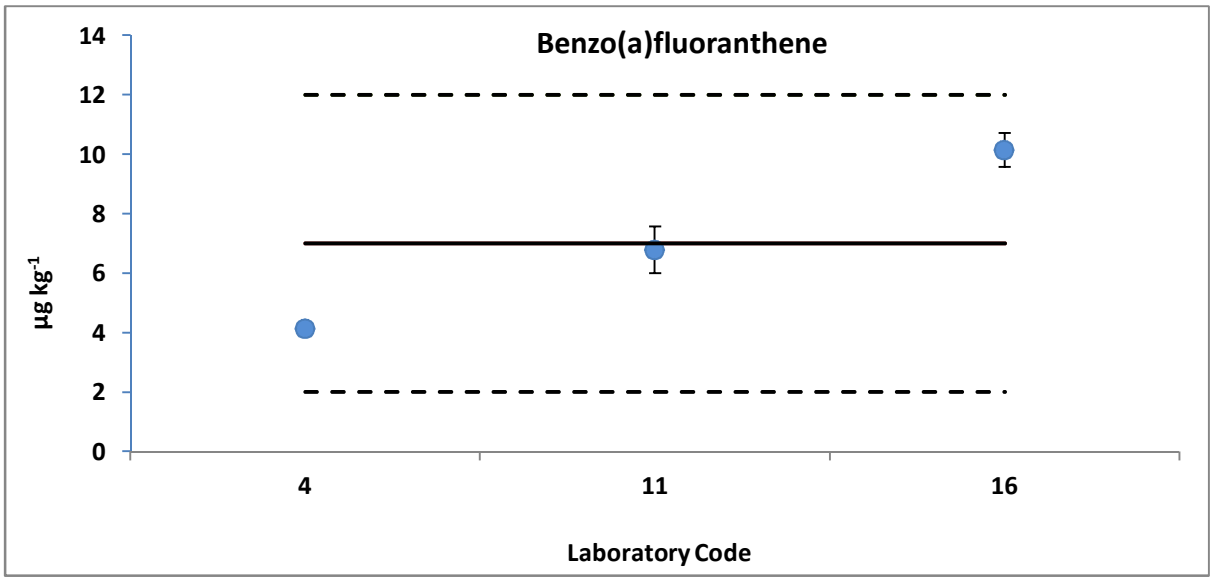


*FIG. 78. Laboratory results for Benzo(a)fluoranthene in IAEA-459 (*µg kg^{-1}*).*

TABLE 97. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF DIBENZ(*a,h*)ANTHRACENE REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
3	2.50		0.10	GC-MS	
4	3.35	0.16	2.00	GC-MS	SRM1941b
6	10.53		0.29	GC-MS/MS	IAEA-408
7	4.20	0.27	0.50	HPLC-FLD	Quasimeme QPH087
8	5.53	0.24	0.88	GC-MS	NIST 1944
9	5.56	0.17	1.00	GC-MS	RM IAEA-159
10	5.77		0.50	GC-MS	IMR LRM
11	44.87	3.15	0.40	GC-HRMS	
13	7.64	0.17	0.10	GC-MS	NIST 1944
16	20.89	0.37	0.07	GC-MS	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

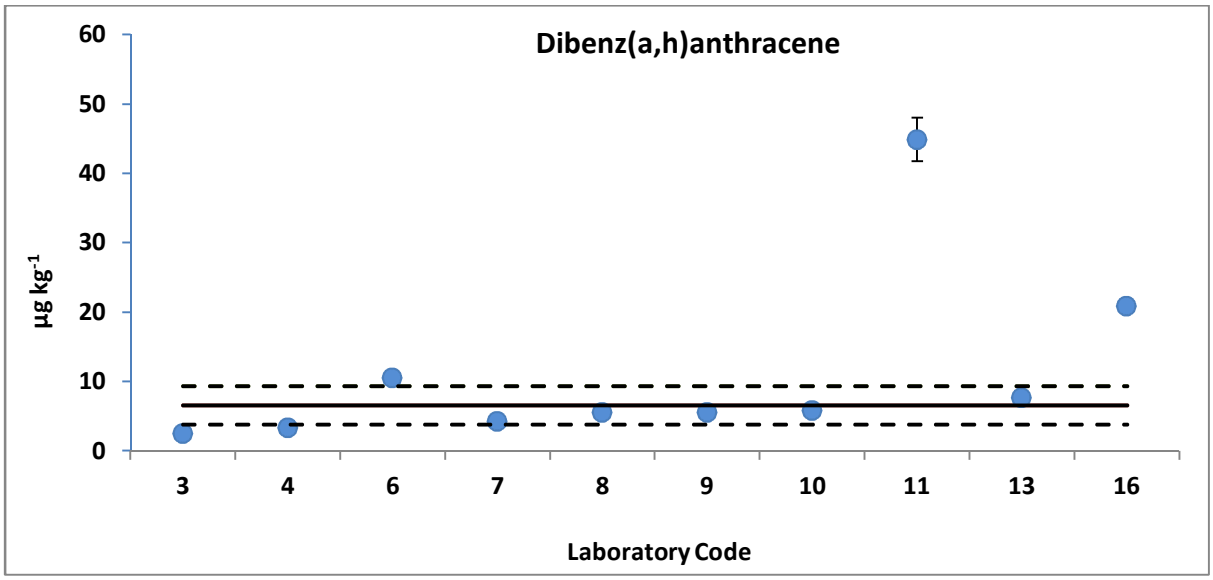


*FIG. 79. Laboratory results for Dibenz(a,h)anthracene in IAEA-459 (*µg kg^{-1}*).*

TABLE 98. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF PERYLENE REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
4	15.94	0.25	1.00	GC-MS	SRM1941b
8	13.70	1.11	0.36	GC-MS	NIST 1944
10	39.42		0.50	GC-MS	IMR LRM
11	36.02	3.09	0.40	GC-HRMS	
13	53.04	2.13	0.10	GC-MS	NIST 1944
16	31.42	1.21	0.01	GC-MS	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

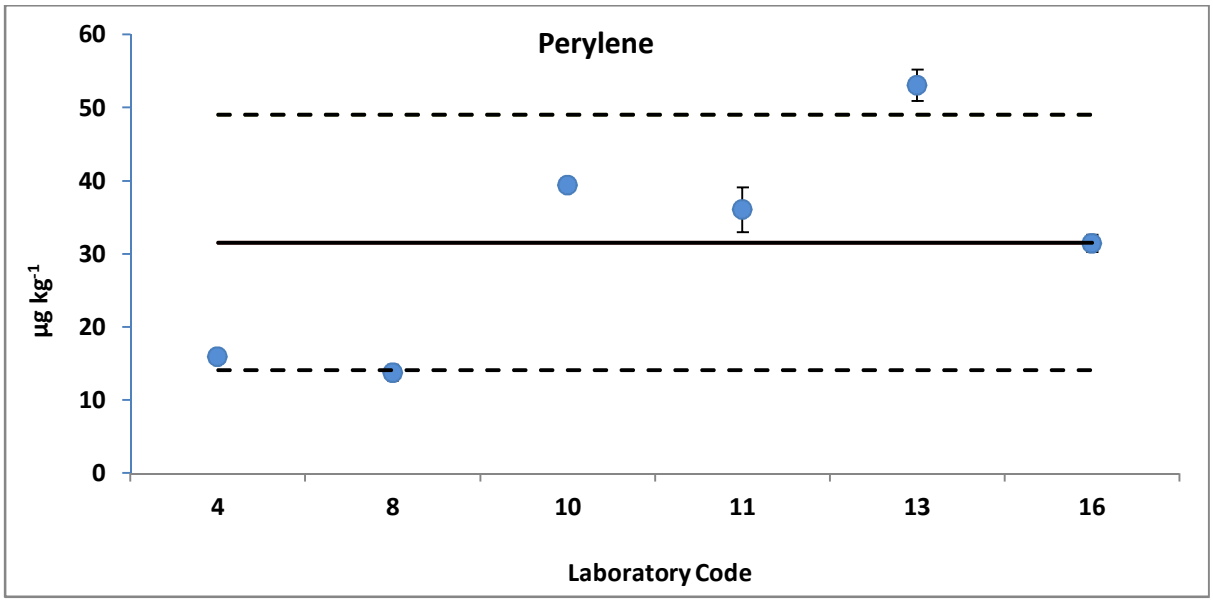


*FIG. 80. Laboratory results for Perylene in IAEA-459 (*µg kg^{-1}*).*

APPENDIX VI

RESULTS FOR THE INFORMATION MEASUREMENTS OF POLYCHLORINATED BIPHENYLS (PCBs)

TABLE 99. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF PCB 8 REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type	(C)RM use for QC
2**	0.406	0.231	5.000	GCxGC-ECD	
4	0.438	0.046	0.100	GC-ECD	SRM1941b
11	0.346	0.016	0.080	GC-HRMS	
16	0.987	0.061	0.092	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported by participants.

**Not taken into account for the calculation of the Information values

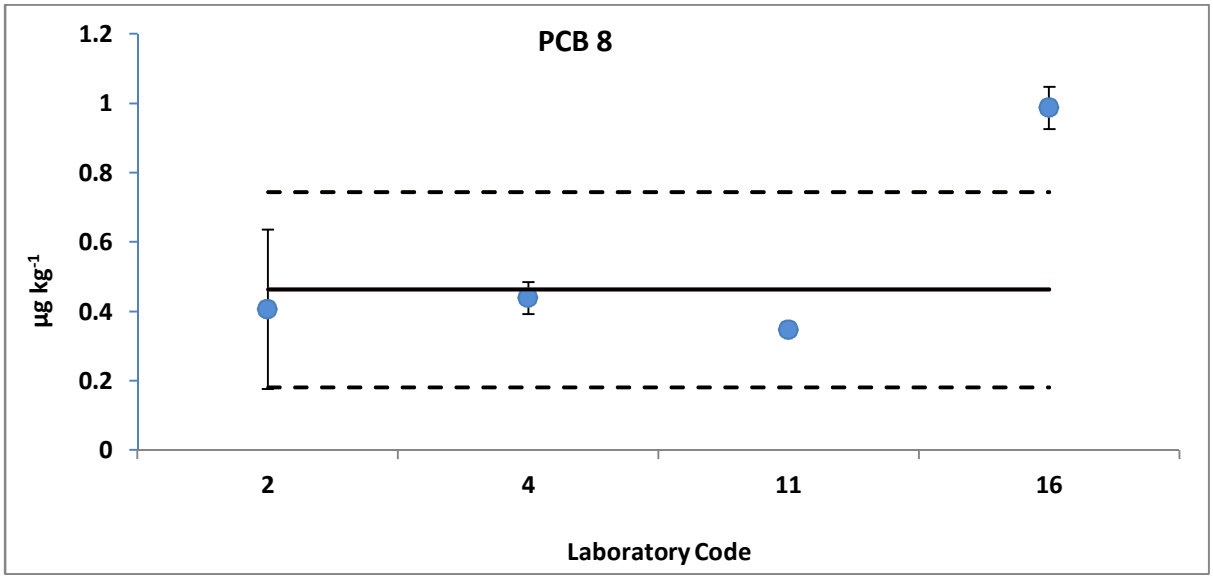


*FIG. 81. Laboratory results for PCB 8 in IAEA-459 (*µg kg^{-1}*).*

TABLE 100. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF PCB 18 REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
2	1.55	0.27	2.000	GCxGC-ECD	
4	0.97	0.01	0.100	GC-ECD	SRM1941b
5	0.59	0.09	0.003	GC-HRMS	NIST 1944
9	1.31	0.03	0.250	GC-MS	RM IAEA-159
11	0.96	0.05	0.040	GC-HRMS	
13	0.97	0.18		GC-MS/MS	NIST1944
16	1.39	0.06	0.100	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

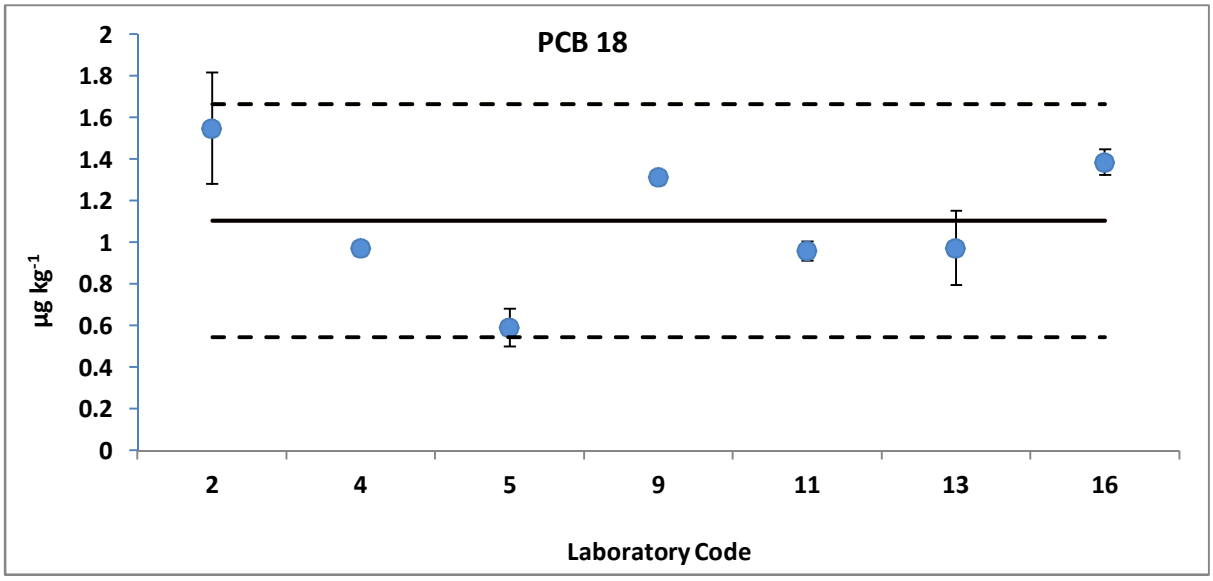


*FIG. 82. Laboratory results for PCB 18 in IAEA-459 (*µg kg^{-1}*).*

TABLE 101. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF PCB 95 REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
2	2.30	0.26	1.00	GCxGC-ECD	
11	2.55	0.26	0.04	GC-HRMS	

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

TABLE 102. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF PCB 97 REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
2	1.64	0.36	1.00	GCxGC-ECD	
11	1.47	0.05	0.02	GC-HRMS	
16	1.16	0.04	0.02	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

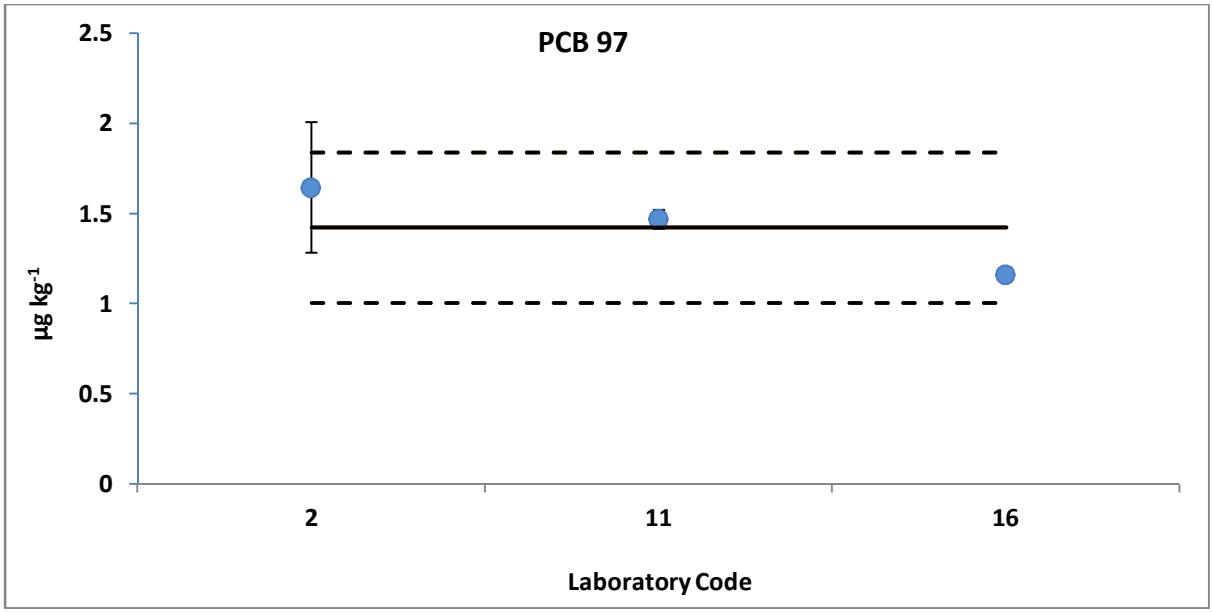


*FIG. 83. Laboratory results for PCB 97 in IAEA-459 (*µg kg^{-1}*).*

TABLE 103. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF PCB 99 REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
2	2.58	0.46	1.000	GCxGC-ECD	
11	2.68	0.17	0.040	GC-HRMS	
16	1.79	0.04	0.020	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

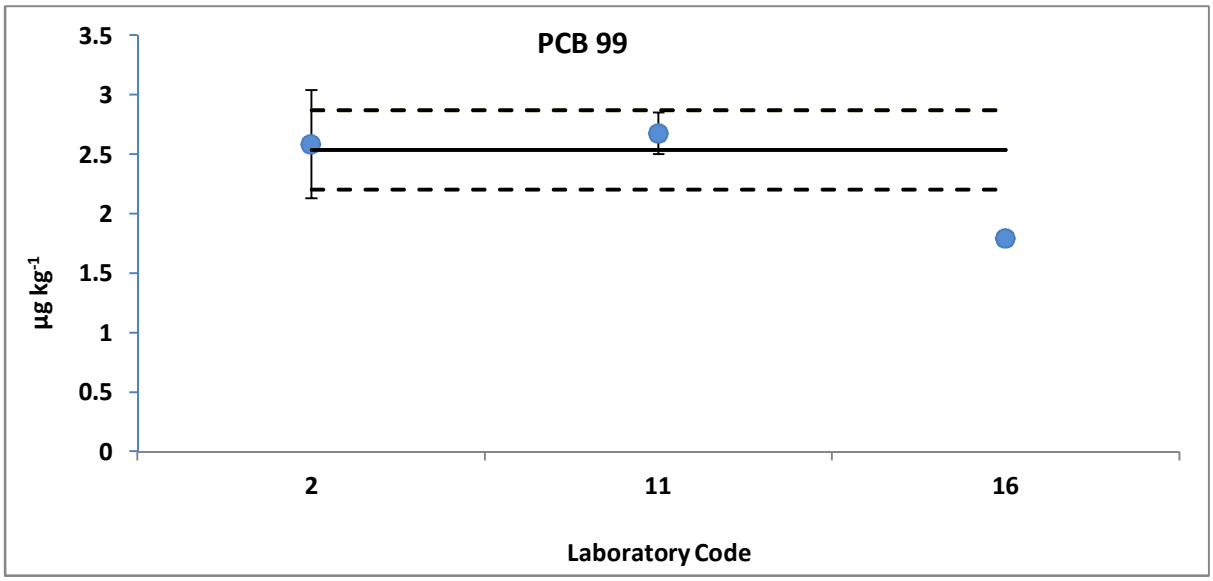


*FIG. 84. Laboratory results for PCB 99 in IAEA-459 (*µg kg^{-1}*).*

TABLE 104. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF PCB 174 REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
2	1.34	0.15	1.000	GCxGC-ECD	
5	0.86	0.04	0.003	GC-HRMS	NIST 1944
11	0.93	0.02	0.005	GC-HRMS	
16	0.86	0.02	0.007	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

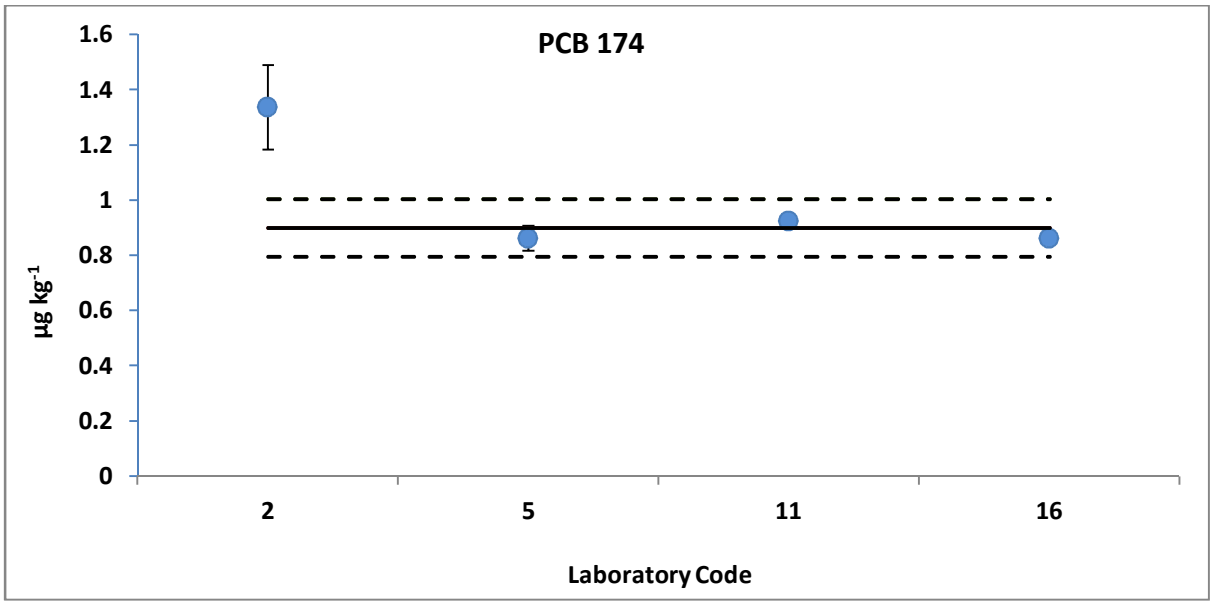


*FIG. 85. Laboratory results for PCB 174 in IAEA-459 (*µg kg^{-1}*).*

TABLE 105. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF PCB 177 REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
2**	0.52	0.12	1.000	GCxGC-ECD	
11	0.48	0.02	0.005	GC-HRMS	
16	0.52	0.02	0.019	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

**Not taken into account for the calculation of the Information values

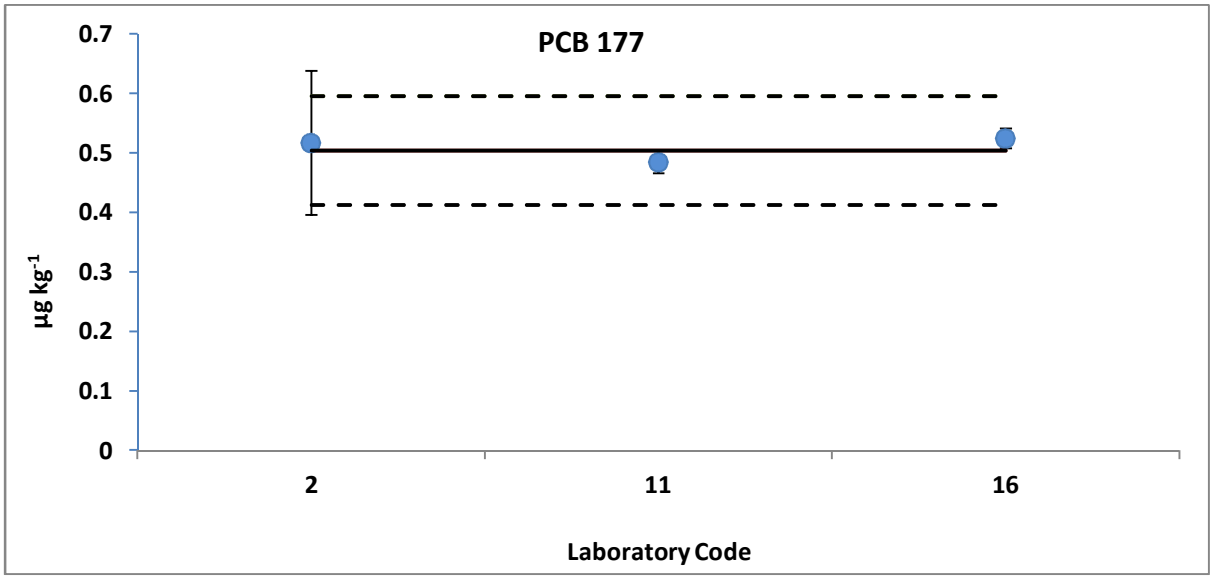


*FIG. 86. Laboratory results for PCB 177 in IAEA-459 (*µg kg^{-1}*).*

TABLE 106. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF PCB 194 REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
2**	0.80	0.12	1.000	GCxGC-ECD	
5	0.38	0.02	0.003	GC-HRMS	NIST 1944
11	0.11	0.01	0.010	GC-HRMS	
13	0.66	0.14		GC-MS/MS	NIST1944
16	0.61	0.02	0.005	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

**Not taken into account for the calculation of the Information values

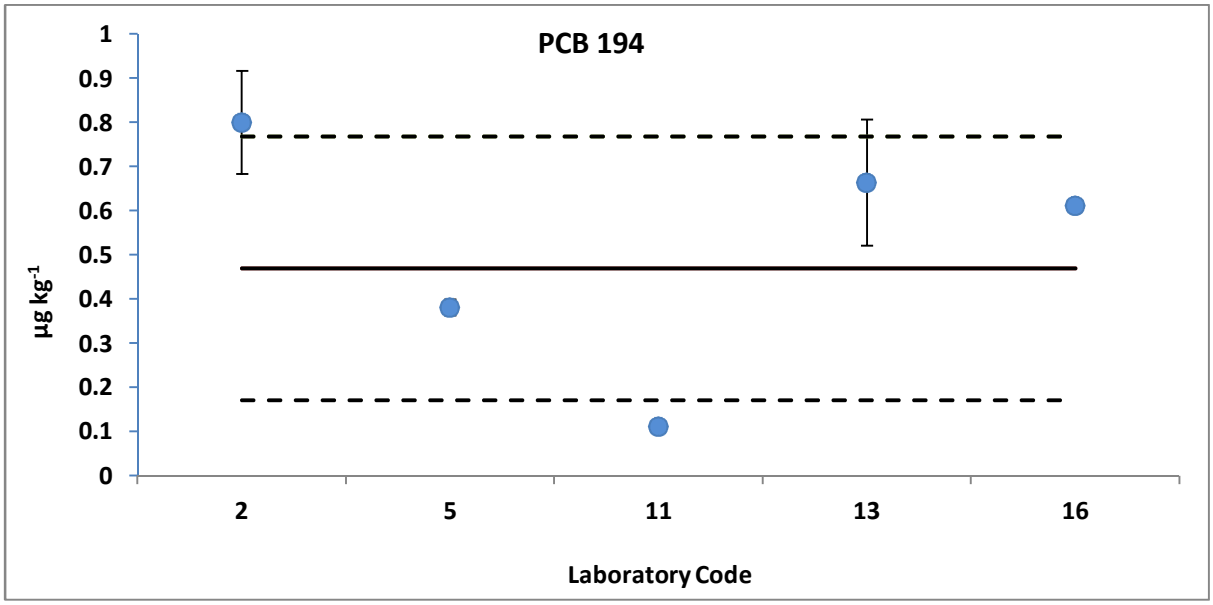


*FIG. 87. Laboratory results for PCB 194 in IAEA-459 (*µg kg^{-1}*).*

TABLE 107. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF PCB 195 REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
4	0.10	0.02	0.100	GC-ECD	SRM1941b
11	0.033	0.003	0.010	GC-HRMS	
16	0.18	0.01	0.002	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

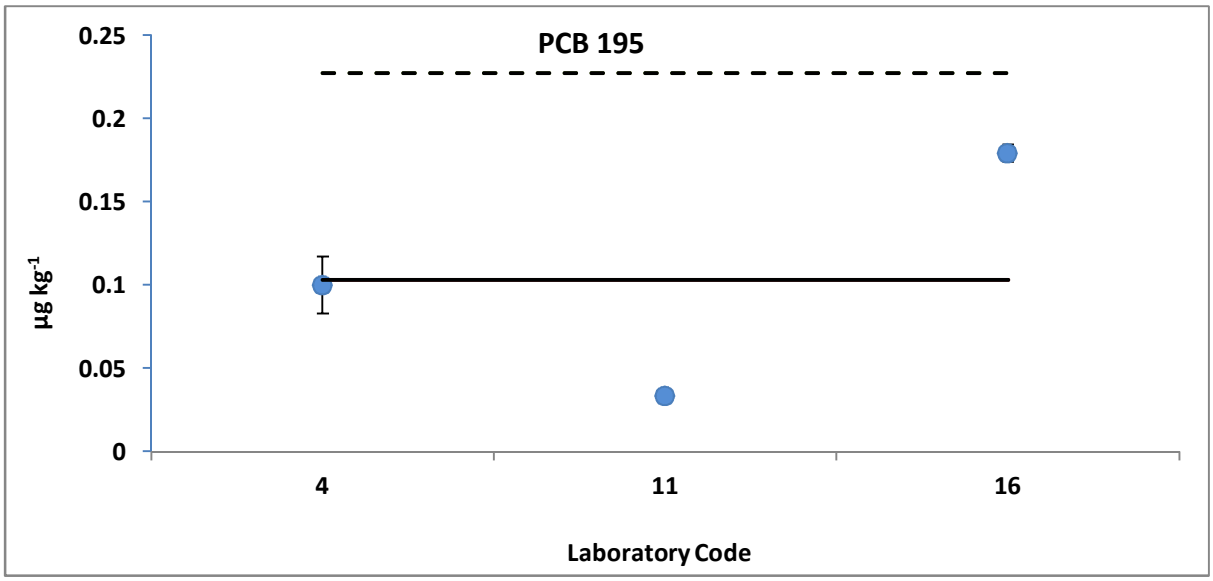


*FIG. 88. Laboratory results for PCB 195 in IAEA-459 (*µg kg^{-1}*).*

TABLE 108. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF PCB 201 REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
2**	0.233	0.139	1.000	GCxGC-ECD	
4	0.185	0.009	0.100	GC-ECD	SRM1941b
11	0.035	0.003	0.010	GC-HRMS	
16	0.186	0.013	0.008	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

**Not taken into account for the calculation of the Information values

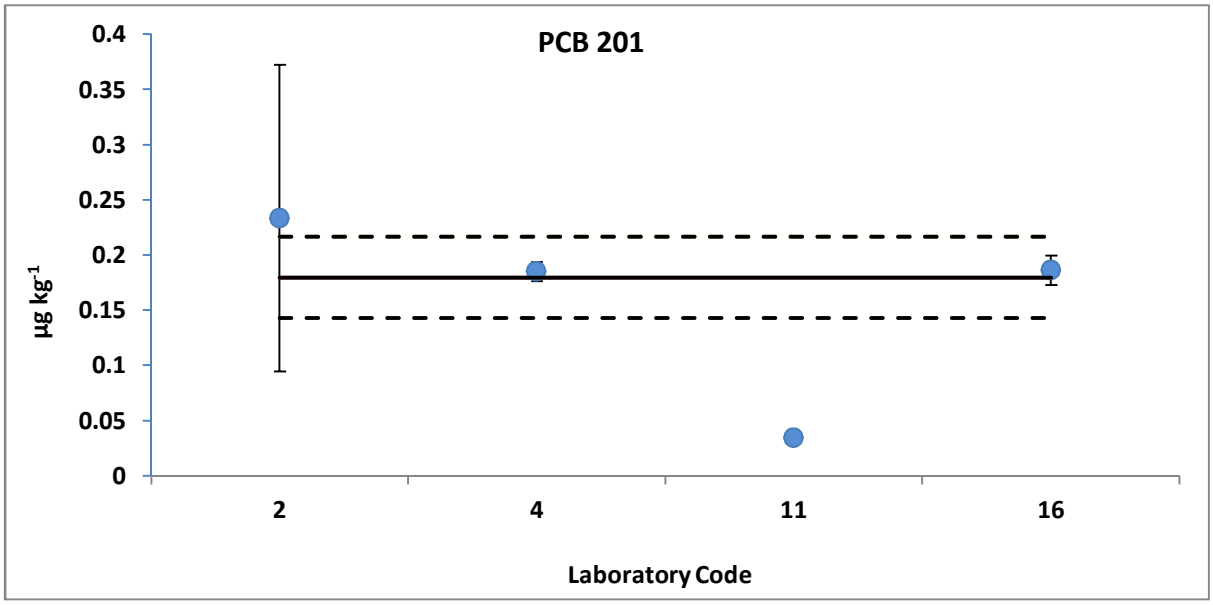


*FIG. 89. Laboratory results for PCB 201 in IAEA-459 (*µg kg^{-1}*).*

TABLE 109. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF PCB 206 REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
2**	0.572	0.144	1.000	GCxGC-ECD	
4	0.200	0.012	0.100	GC-ECD	SRM1941b
11	0.203	0.003	0.010	GC-HRMS	
16	0.258	0.008	0.004	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

**Not taken into account for the calculation of the Information values

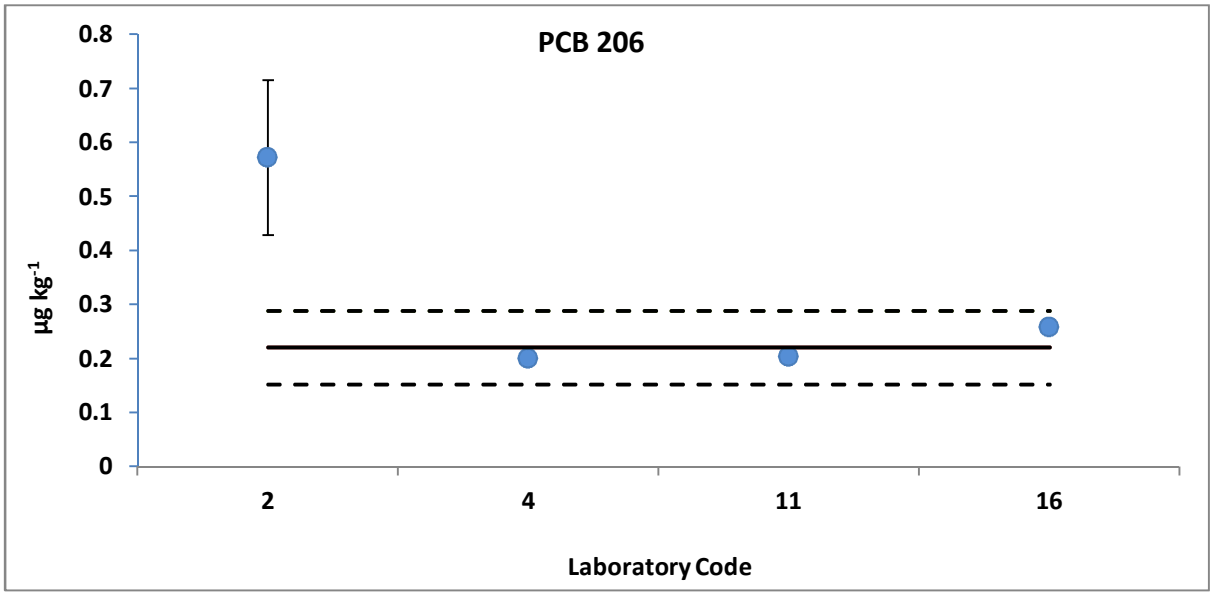


*FIG. 90. Laboratory results for PCB 206 in IAEA-459 (*µg kg^{-1}*).*

APPENDIX VII

RESULTS FOR THE INFORMATION MEASUREMENTS OF ORGANOCHLORINATED PESTICIDES

TABLE 110. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF HCB REPORTED BY PARTICIPANTS (μg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
2**	0.413	0.031	1.000	GCxGC-ECD	
3	0.160		0.010	GC-MS/MS	
4	0.152	0.006	0.100	GC-ECD	SRM1941b
5	0.194	0.010	0.010	GC-HRMS	NIST 1944
6	0.097	0.008	0.002	GC-MS/MS	IAEA-408
7	0.097	0.001	0.035	GC-MS/MS	Quasi QOR123MS
9	0.145	0.004	0.100	GC-ECD	
10	0.303	0.020	0.020	GC-ECD	
11	0.167	0.007	0.040	GC-HRMS	
13	0.153	0.026		GC-MS/MS	NIST1944
15	<0.10		0.100	GC-ECD	Quasi MS-2
16	0.147	0.008	0.085	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.
**Not taken into account for the calculation of the Information values

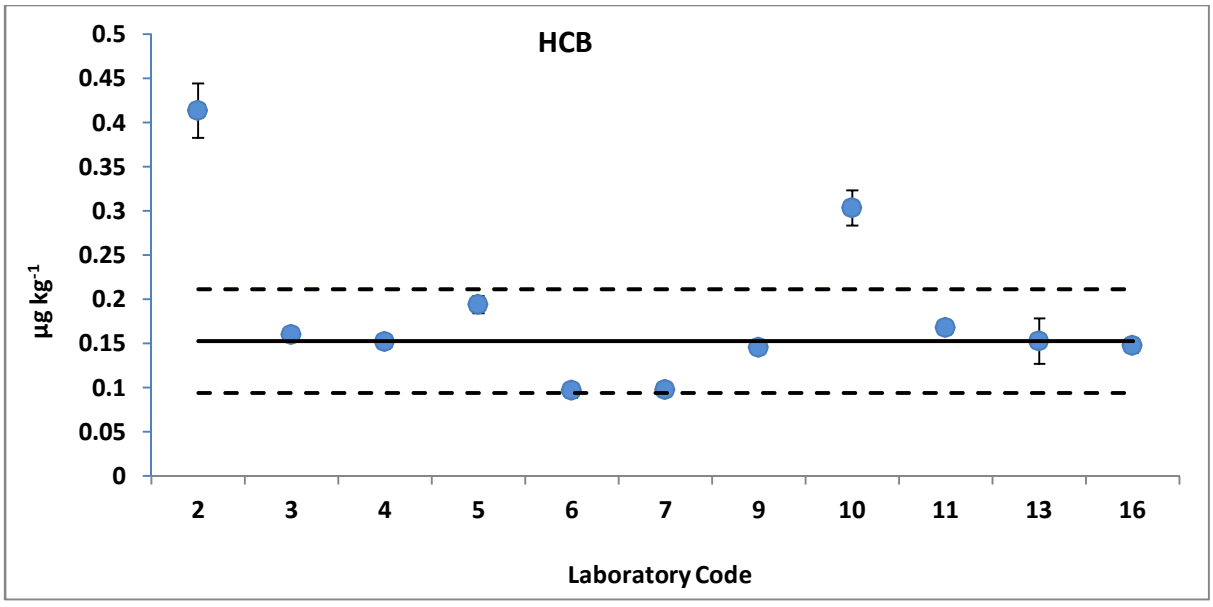


*FIG. 91. Laboratory results for HCB in IAEA-459 (*μg kg^{-1}*).*

TABLE 111. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF α-HCH REPORTED BY PARTICIPANTS (μg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
2**	0.592	0.052	1.000	GCxGC-ECD	
3	0.070		0.010	GC-MS/MS	
7	0.113	0.010	0.035	GC-MS/MS	Quasi QOR123MS
9	0.240	0.016	0.100	GC-ECD	
10	0.156	0.017	0.020	GC-ECD	
11	0.191	0.010	0.080	GC-HRMS	
13	0.138	0.031		GC-MS/MS	NIST1944
15	<0.10		0.100	GC-ECD	Quasi MS-2
16	0.136	0.015	0.004	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{S}{\sqrt{n}}$ were S is the standard deviation and n is the number of measurements reported by participants.
**Not taken into account for the calculation of the Information values

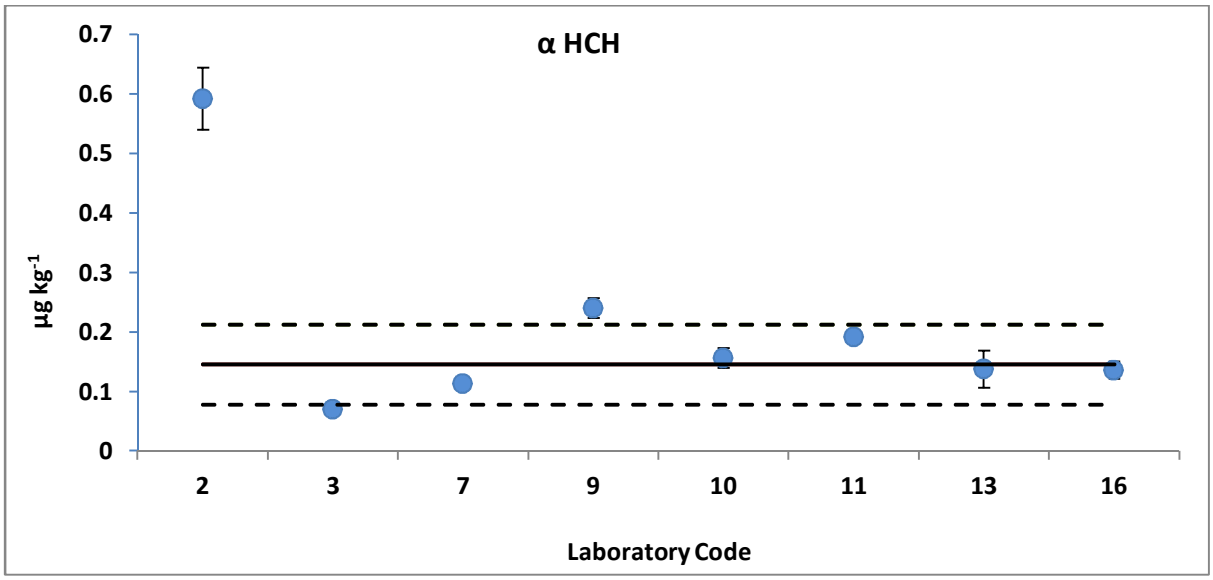


*FIG. 92. Laboratory results for α- HCH in IAEA-459 (*μg kg^{-1}*).*

TABLE 112. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF β-HCH REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
2	0.575	0.045	1.000	GCxGC-ECD	
3	0.020		0.010	GC-MS/MS	
6	0.095	0.011	0.005	GC-MS/MS	IAEA-408
7	0.080	0.003	0.035	GC-MS/MS	Quasi QOR123MS
10	0.193	0.029	0.020	GC-ECD	
11	0.141	0.005	0.020	GC-HRMS	
13	0.203	0.082		GC-MS/MS	NIST1944
15	<0.10		0.100	GC-ECD	Quasi MS-2
16	0.217	0.015	0.037	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

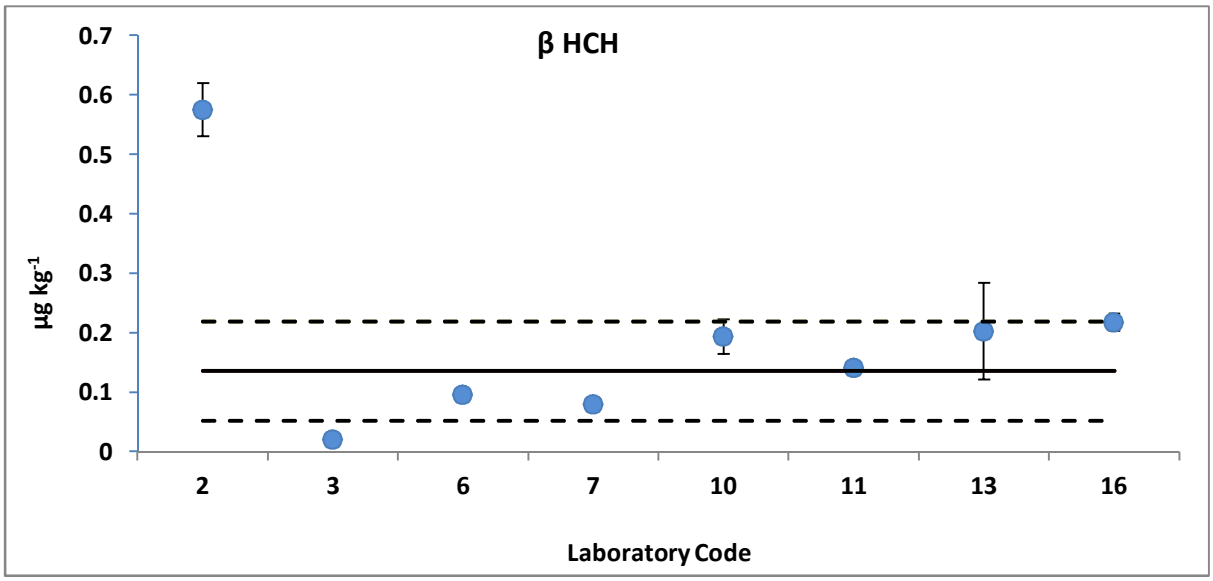


*FIG. 93. Laboratory results for β-HCH in IAEA-459 (*µg kg^{-1}*).*

TABLE 113. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF γ-HCH (LINDANE) REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
3	0.110		0.010	GC-MS/MS	
6	0.328	0.051	0.004	GC-MS/MS	IAEA-408
7	0.147	0.015	0.035	GC-MS/MS	Quasi QOR123MS
10	0.145	0.002	0.020	GC-ECD	
11	0.193	0.021	0.060	GC-HRMS	
13	0.208	0.032		GC-MS/MS	NIST1944
15	<0.10		0.100	GC-ECD	Quasi MS-2
16	0.196	0.021	0.074	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

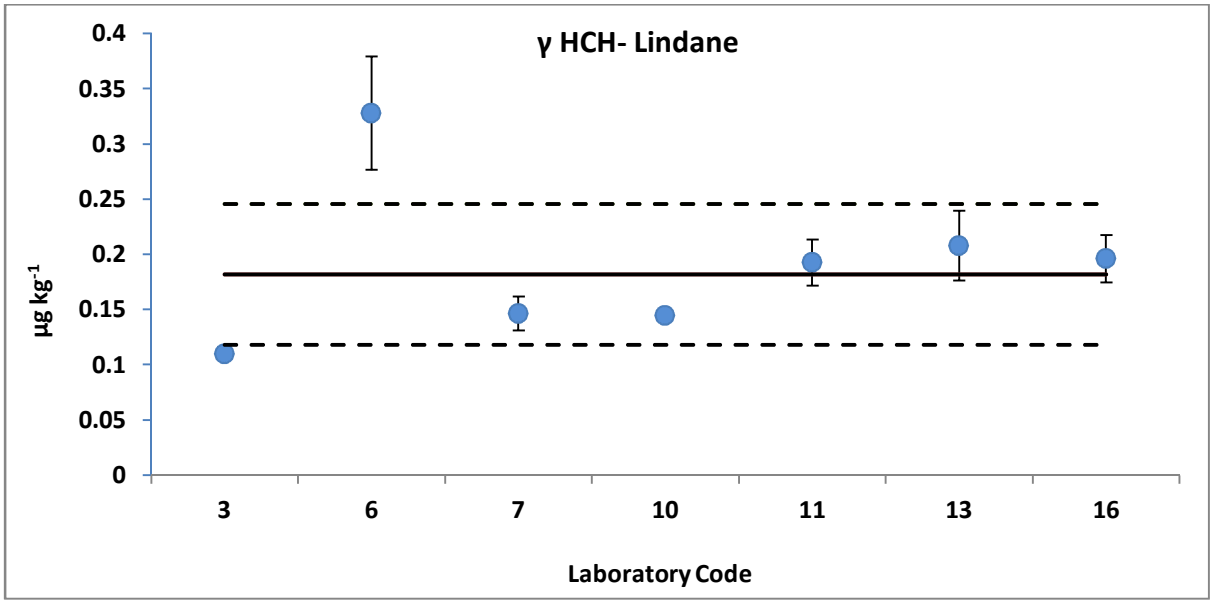


*FIG. 94. Laboratory results for γ HCH (Lindane) in IAEA-459 (*µg kg^{-1}*).*

TABLE 114. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF δ-HCH REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type	(C)RM use for QC
11	0.027	0.002	0.010	GC-HRMS	
15	<0.10		0.100	GC-ECD	Quasi MS-2
16	<0.02		0.019	GC-ECD	NIST1941B

TABLE 115. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF HEPTACHLOR REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
2**	0.360		1.000	GCxGC-ECD	
9	0.190	0.023	0.100	GC-ECD	RM IAEA-159
11	<0.03		0.030	GC-HRMS	
13	0.108	0.058		GC-MS/MS	NIST1944
16	<0.162		0.162	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

**Not taken into account for the calculation of the Information values

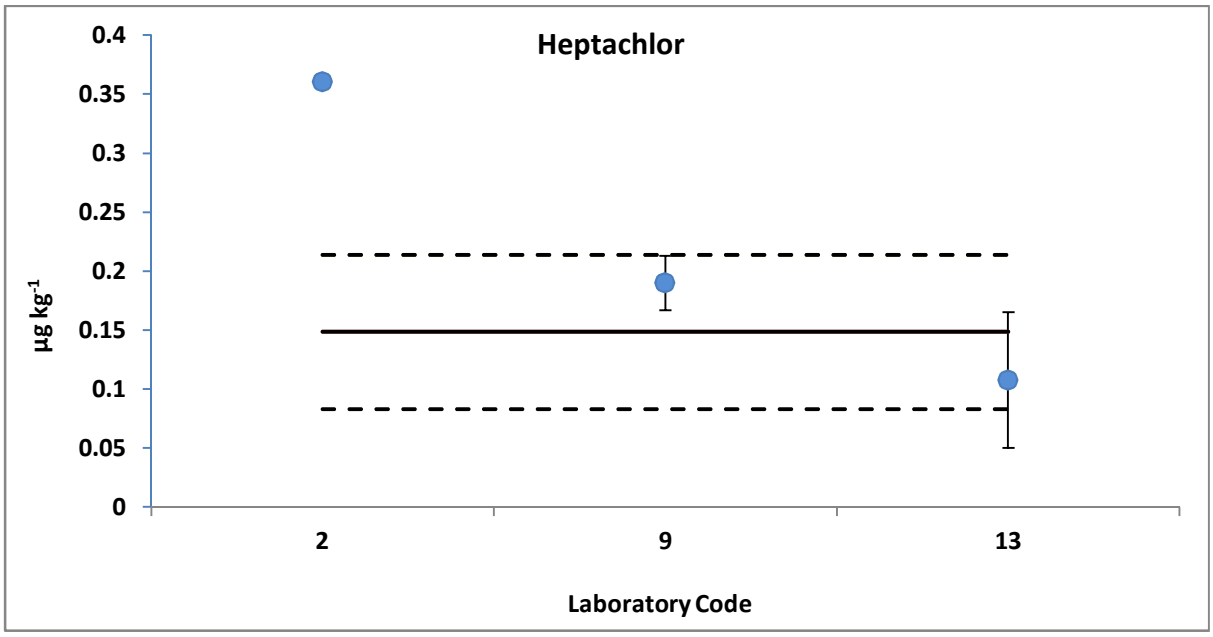


*FIG. 95. Laboratory results for Heptachlor in IAEA-459 (*µg kg^{-1}*).*

TABLE 116. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF ALDRIN REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type	(C)RM use for QC
2	<1.00		1.00	GCxGC-ECD	
15	<0.100		0.100	GC-ECD	
16	<0.049		0.049	GC-ECD	NIST1941B

TABLE 117. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF DIELDRIN REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
2**	0.250		1.000	GCxGC-ECD	
10	0.102	0.001	0.020	GC-ECD	
11	0.063	0.006	0.010	GC-HRMS	
13	<0.500		0.500	GC-MS/MS	NIST1944
15	<0.100		0.100	GC-ECD	Quasi MS-2
16	0.149	0.015	0.009	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

**Not taken into account for the calculation of the Information values

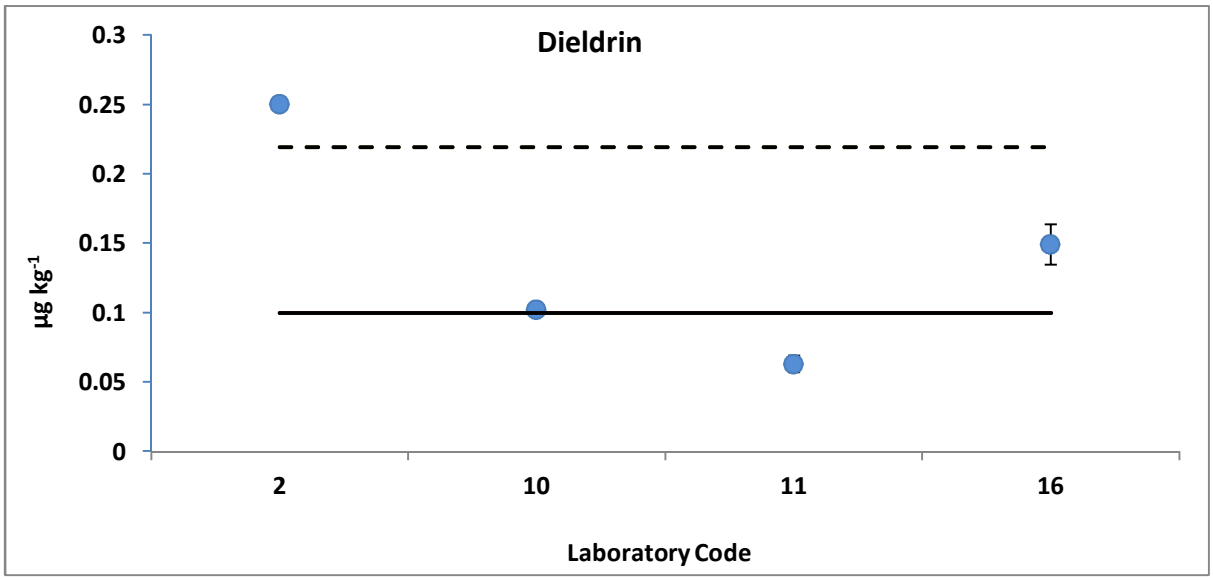


*FIG. 96. Laboratory results for Dieldrin in IAEA-459 (*µg kg^{-1}*).*

TABLE 118. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF ENDRIN REPORTED BY PARTICIPANTS (µg kg^{-1}

LAB Code	Mean	Uncertainty*	Detection limit	Detector type	(C)RM use for QC
2	<1.000		1.000	GCxGC-ECD	
11	<0.020		0.020	GC-HRMS	
16	<0.053		0.053	GC-ECD	NIST1941B

TABLE 119. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF cis-CHLORDANE REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
7	0.044	0.010	0.035	GC-MS/MS	
9	1.440	0.027	0.100	GC-ECD	
11	0.019	0.001	0.010	GC-HRMS	
16	0.048	0.016	0.008	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

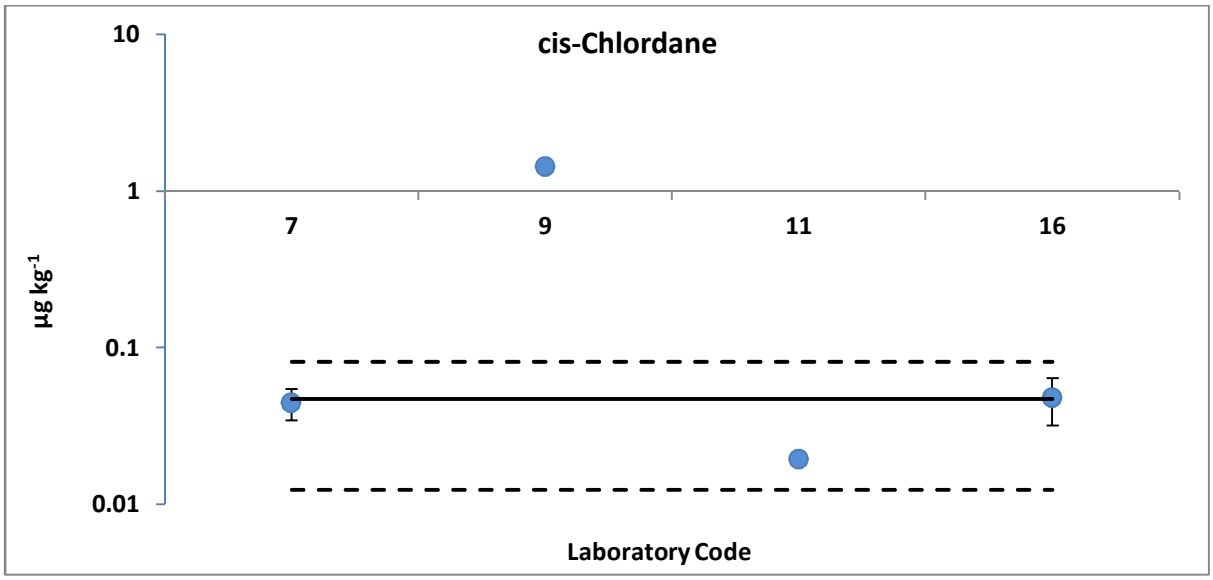


*FIG. 97. Laboratory results for cis-Chlordane in IAEA-459 (*µg kg^{-1}*).*

TABLE 120. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF trans-CHLORDANE REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
2	0.347	0.052	1.000	GCxGC-ECD	
3	0.040		0.010	GC-MS/MS	
7	0.093	0.014	0.035	GC-MS/MS	
11	0.084	0.007	0.010	GC-HRMS	
16	0.046	0.004	0.010	GC-ECD	NIST1941B

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

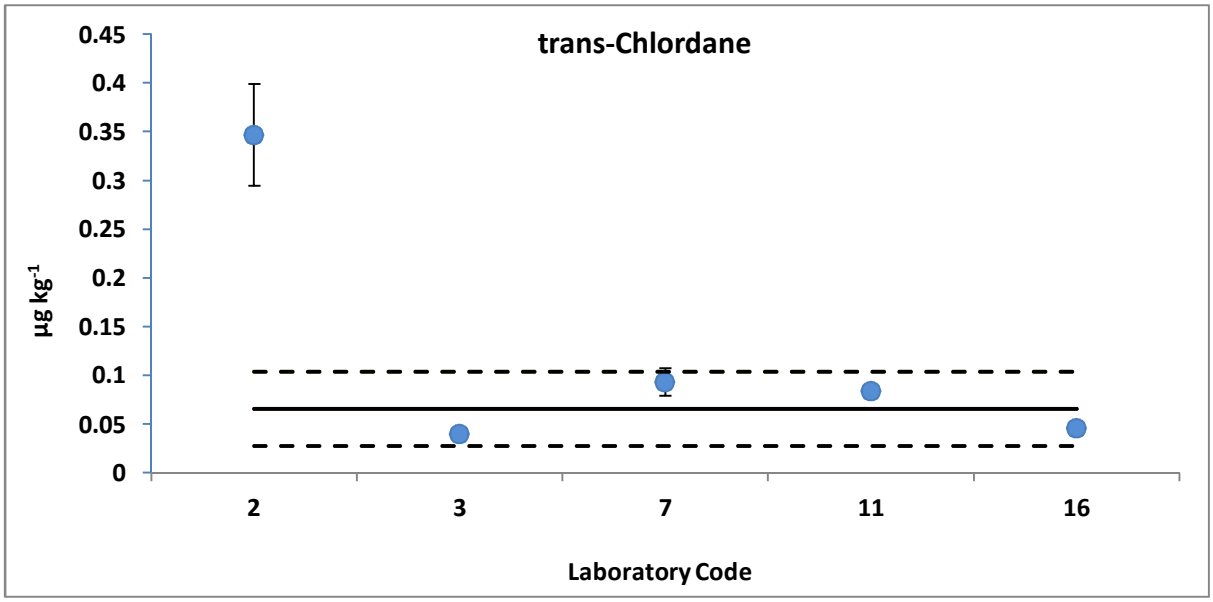


*FIG. 98. Laboratory results for trans-Chlordane in IAEA-459 (*µg kg^{-1}*).*

TABLE 121. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF cis-NONACHLOR REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type	(C)RM use for QC
2	<1.00		1.000	GCxGC-ECD	
11	0.021	0.002	0.010	GC-HRMS	
16	0.100		0.007	GC-ECD	NIST1941B

TABLE 122. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF TRANS-NONACHLOR REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type	(C)RM use for QC
2	<1.00		1.000	GCxGC-ECD	
6	0.018	0.003	0.006	GC-MS/MS	IAEA-408
11	0.012	0.001	0.010	GC-HRMS	
16	0.011	0.002	0.009	GC-ECD	NIST1941B

TABLE 123. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF α-ENDOSULFAN REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type	(C)RM use for QC
2	<1.0		1.000	GCxGC-ECD	
11	<0.01		0.010	GC-HRMS	
13	<0.2			GC-MS/MS	NIST1944
16	0.057	0.008	0.011	GC-ECD	NIST1941B

TABLE 124. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF β-ENDOSULFAN REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type	(C)RM use for QC
2	<1.0		1.000	GCxGC-ECD	
11	<0.01		0.010	GC-HRMS	
13	<0.2			GC-MS/MS	NIST1944
16	0.05		0.010	GC-ECD	NIST1941B

TABLE 125. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF ENDOSULFAN SULFATE REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type	(C)RM use for QC
2	<1.00		1.000	GCxGC-ECD	
11	<0.05		0.050	GC-HRMS	
13	<0.2			GC-MS/MS	NIST1944
16	0.05		0.011	GC-ECD	NIST1941B

APPENDIX VIII

RESULTS FOR THE INFORMATION MEASUREMENTS OF POLYBROMINATED DIPHENYL ETHERS (PBDEs)

TABLE 126. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF BDE 28 REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
2	0.0265	0.0031	0.005	GC-HRMS	In House Spike Solution
3	0.0165	0.0010	0.010	GC-MS-EI	
4	0.0217	0.0033	0.020	GC-MS-NICI	
5	0.0142	0.0009	0.002	GC-HRMS	
10	0.0572		0.020	MS-NICI	
11	0.0125	0.0004	0.002	GC-HRMS	
16	0.0245	0.0045	0.009	GC-ECD	

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

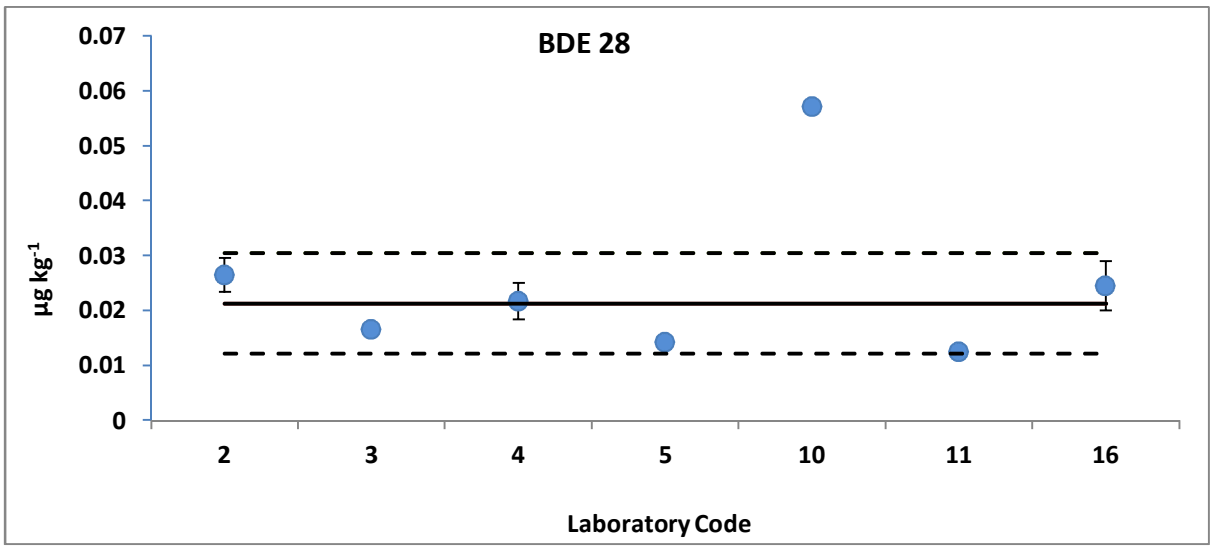


*FIG. 99. Laboratory results for BDE 28in IAEA-459 (*µg kg^{-1}*).*

TABLE 127. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF BDE 66 REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
2	0.024	0.006	0.011	GC-HRMS	In House Spike Solution
4	0.012	0.003	0.020	GC-MS-NICI	
5	0.009	0.002	0.002	GC-HRMS	NIST 1944
10	<0.020		0.020	MS-NICI	
11	0.006	0.001	0.005	GC-HRMS	
16	0.009		0.004	GC-ECD	

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

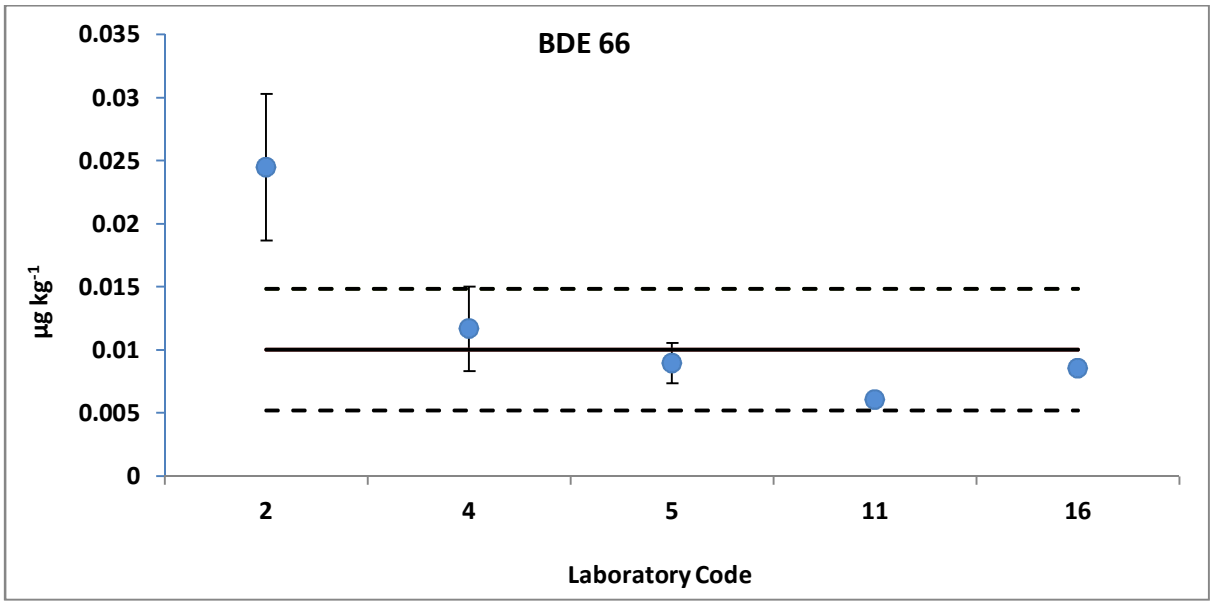


*FIG. 100. Laboratory results for BDE 66in IAEA-459 (*µg kg^{-1}*).*

TABLE 128. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF BDE 85 REPORTED BY PARTICIPANTS (μg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
2	0.0238	0.0122	0.015	GC-HRMS	In House Spike Solution
4	<0.02			GC-MS-NICI	
5	0.0068	0.0008	0.002	GC-HRMS	NIST 1944
11	0.0043	0.0003	0.006	GC-HRMS	
16	0.0072	0.0012	0.005	GC-ECD	

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

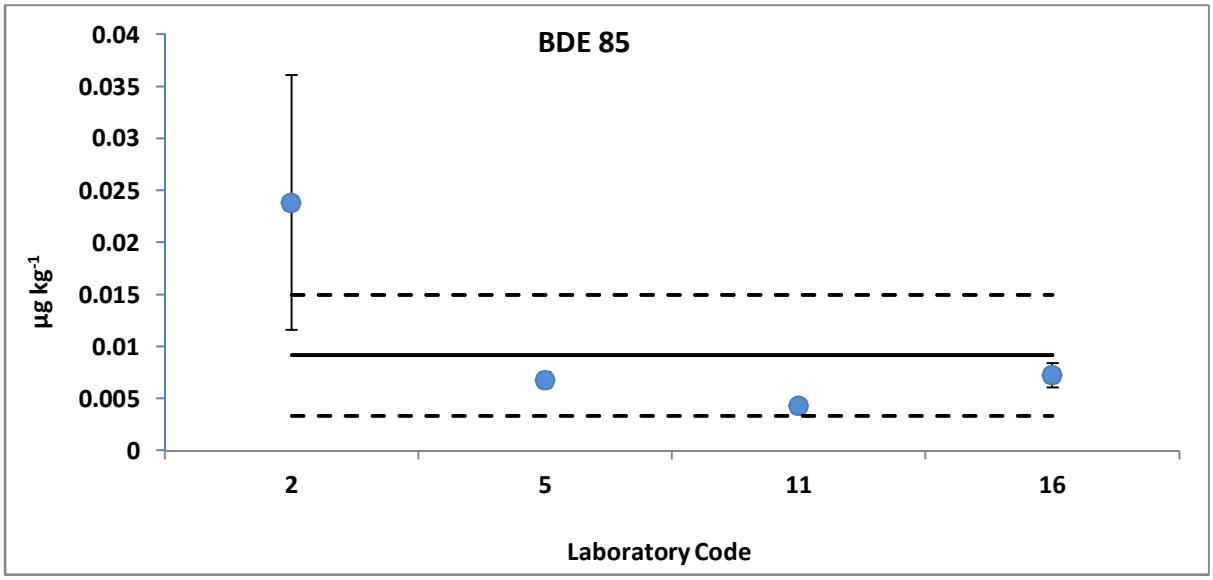


*FIG. 101. Laboratory results for BDE 85in IAEA-459 (*μg kg^{-1}*).*

TABLE 129. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF BDE 100 REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
2	0.115	0.050	0.078	GC-HRMS	In House Spike Solution
3	0.025	0.006	0.020	GC-MS-EI	
4	0.032	0.003	0.020	GC-MS-NICI	
5	0.028	0.003	0.001	GC-HRMS	NIST 1944
10	0.023		0.020	MS-NICI	
11	0.027	0.001	0.003	GC-HRMS	
16	0.035	0.006	0.013	GC-ECD	

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ were s is the standard deviation and n is the number of measurements reported by participants.

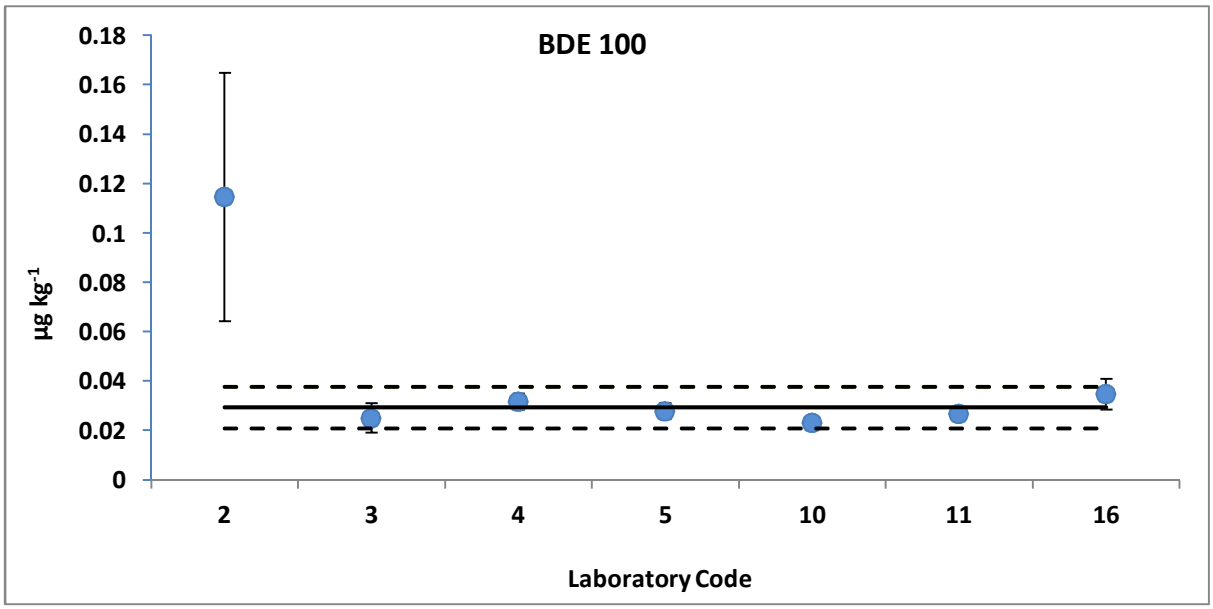


*FIG. 102. Laboratory results for BDE 100 in IAEA-459 (*µg kg^{-1}*).*

TABLE 130. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF BDE 154 REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Detector type:	(C)RM use for QC
2	0.054	0.019	0.020	GC-HRMS	In House Spike Solution
3	0.016	0.002	0.010	GC-MS-EI	
4	0.032	0.003	0.020	GC-MS-NICI	
5	0.023	0.002	0.002	GC-HRMS	NIST 1944
10	0.032		0.020	MS-NICI	
11	0.021	0.002	0.003	GC-HRMS	
16	0.011	0.009	0.006	GC-ECD	

*Calculated as: 2 x $\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported by participants.

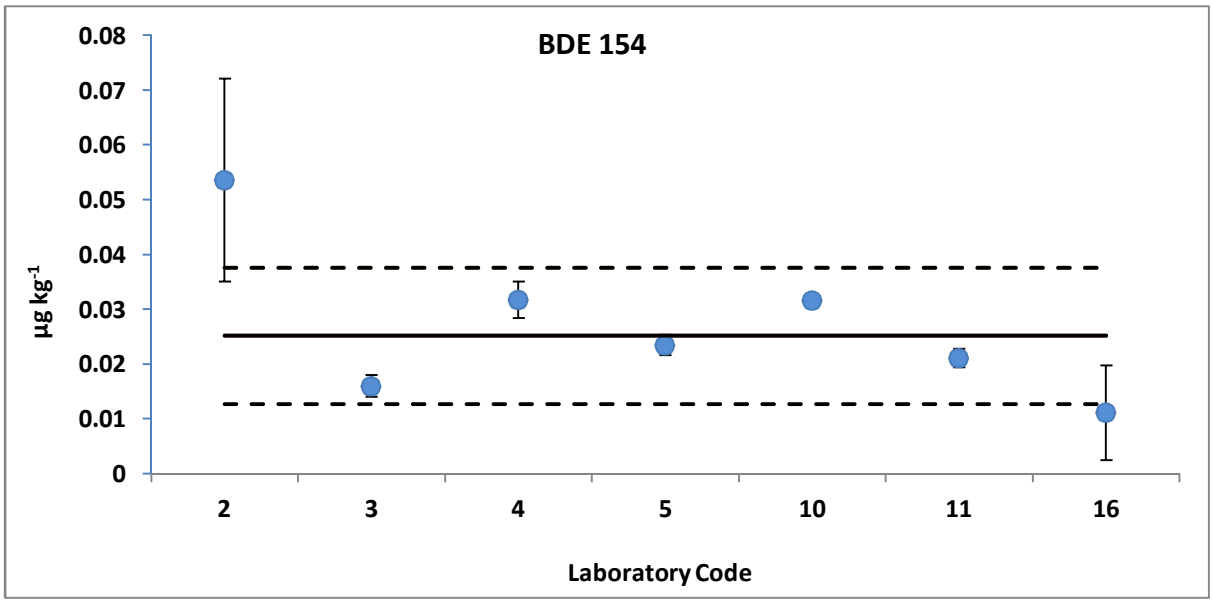


*FIG. 103. Laboratory results for BDE 154 in IAEA-459 (*µg kg^{-1}*).*

APPENDIX IX

RESULTS FOR THE INFORMATION MEASUREMENTS OF ALIPHATIC HYDROCARBONS

TABLE 131. RESULTS USED FOR THE CALCULATION OF INFORMATION MASS FRACTIONS OF ALIPHATIC HYDROCARBONS REPORTED BY PARTICIPANTS

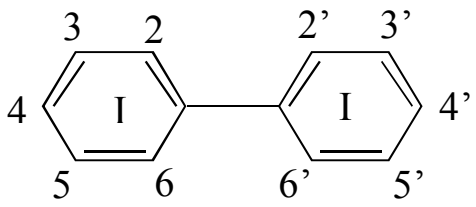
Laboratory Code Number		9	11	16
Equipment		GC-MS	GC-HRMS	GC-FID
UCM Aliphatics	mg kg^{-1}		450	220
n-C12	µg kg^{-1}	98		39
n-C13	µg kg^{-1}	93		71
n-C14	µg kg^{-1}	430	111	375
n-C15	µg kg^{-1}	110	78	86
n-C16	µg kg^{-1}	689	587	620
n-C17	µg kg^{-1}	97	116	111
n-C18	µg kg^{-1}	664	868	1007
n-C19	µg kg^{-1}	100	111	167
n-C20	µg kg^{-1}	724	587	1144
n-C21	µg kg^{-1}	71	116	234
n-C22	µg kg^{-1}	786	868	1395
n-C23	µg kg^{-1}	147	97	346
n-C24	µg kg^{-1}	823	1023	1451
n-C25	µg kg^{-1}	234	58	448
n-C26	µg kg^{-1}	779	875	1566
n-C27	µg kg^{-1}	304	96	762
n-C28	µg kg^{-1}		453	1334
n-C29	µg kg^{-1}		309	1018
n-C30	µg kg^{-1}		215	957
n-C31	µg kg^{-1}		233	965
n-C32	µg kg^{-1}		107	474
n-C33	µg kg^{-1}		127	320
n-C34	µg kg^{-1}		29	496
n-C35	µg kg^{-1}			482
n-C36	µg kg^{-1}			225
Pristane	µg kg^{-1}		62	102
Phytane	µg kg^{-1}		91	168

APPENDIX X

SYSTEMATIC NUMBERING OF PCB CONGENERS

IUPAC No

	Dichlorobiphenyl
5	2,3
8	2,4’
	Trichlorobiphenyl
18	2,2’,5
20	2,3,3’
28	2,4,4’
30	2,4,6
31	2,4’,5
	Tetrachlorobiphenyl
44	2,2’,3,5’
47	2,2’,4,4’
49	2,2’,4,5’
52	2,2’,5,5’
60	2,3,4,4’
66	2,3’,4,4’
70	2,3’,4’,5
74	2,4,4’,5
77	3,3’,4,4’
	Pentachlorobiphenyl
84	2,2’,3,3’,6
87	2,2’,3,4,5’
92	2,2’,3,5,5’
95	2,2’,3,5’,6
97	2,2’,3’,4,5
99	2,2’,4,4’,5
101	2,3,4,4’,5
105	2,3,3’,4,4’
110	2,3,3’,4’,6
118	2,3’,4,4’,5
119	2,3’,4,4’,6
123	2’,3,4,4’,5
	Hexachlorobiphenyl
128	2,2’,3,3’,4,4’
132	2,2’,3,3’,4,6’
135	2,2’,3,3’,5,6’
136	2,2’,3,3’,6,6’
138	2,2’,3,4,4’,5
141	2,2’,3,4,5,5’
147	2,2’,3,4’,5,6
149	2,2’,3,4’,5’,6
151	2,2’,3,5,5’,6
153	2,2’,4,4’,5,5’
156	2,3,3’,4,4’,5
158	2,3,3’,4,4’,6
167	2,3’,4,4’,5,5’
	Heptachlorobiphenyl
170	2,2’,3,3’,4,4’,5
174	2,2’,3,3’,4,5,6
177	2,2’,3,3’,4’,5,6
180	2,2’,3,4,4’,5,5’
183	2,2’,3,4,4’,5’,6
185	2,2’,3,4,5,5’,6
187	2,2’,3,4’,5,5’,6
	Octachlorobiphenyl
194	2,2’,3,3’,4,4’,5,5’
195	2,2’,3,3’,4,4’,5,6
196	2,2’,3,3’,4,4’,5,6’
201	2,2’,3,3’,4,5’,6,6
	Nonachlorobiphenyl
206	2,2’,3,3’,4,4’,5,5’,6
	Decachlorobiphenyl
209	2,2’,3,3’,4,4’,5,5’,6,6’



APPENDIX XI

SYSTEMATIC NUMBERING OF PBDES CONGENERS

IUPAC No

Tribromodiphenyl

17 2,2',4
28 2,4,4'

Tetrabromodiphenyl

47 2,2',4,4'
49 2,2',4,5'
66 2,3',4,4'
71 2,3',4',6
77 3,3',4,4'

Pentabromodiphenyl

85 2,2',3,4,4'
99 2,2',4,4',5
100 2,2',4,4',6
119 2,3',4,4',6

Hexabromodiphenyl

138 2,2',3,4,4',5'
139 2,2',3,4,4',6
140 2,2',3,4,4',6'
153 2,2',4,4',5,5'
154 2,2',4,4',5,6'

Heptabromodiphenyl

180 2,2',3,4,4',5,5'
183 2,2',3,4,4',5',6
184 2,2',3,4,4',6,6'

Octabromodiphenyl

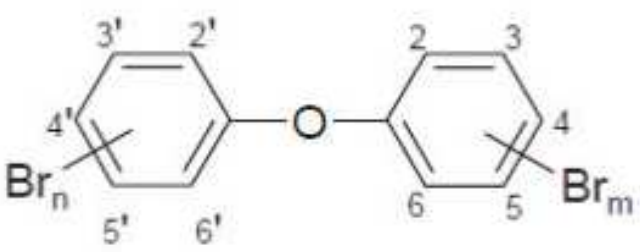
196 2,2',3,3',4,4',5,6'
197 2,2',3,3',4,4',6,6'
201 2,2',3,3',5,5',6,6'
203 2,2',3,4,4',5,5',6
204 2,2',3,4,4',5,6,6'

Nonabromodiphenyl

206 2,2',3,3',4,4',5,5',6
207 2,2',3,3',4,4',5,6,6'
208 2,2',3,3',4,5,5',6,6'

Decabromobiphenyl

209 2,2',3,3',4,4',5,5',6,6'



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