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Certification of Mass Fractions of Organochlorines and Polybrominated Diphenyl Ethers in IAEA-435A Fish Homogenate Sample

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CERTIFICATION OF MASS FRACTIONS OF ORGANOCHLORINES AND POLYBROMINATED DIPHENYL ETHERS IN IAEA-435A FISH HOMOGENATE SAMPLE

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INTERNATIONAL ATOMIC ENERGY AGENCY
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FOREWORD

One of the IAEA's major programmes is to assist Member States in the understanding, monitoring and protection of the marine environment. In order to assess the impact of land and sea based pollution sources on marine coastal environments, it is critical to ensure the quality of the analytical data generated by national and regional pollution monitoring programmes. For this purpose, the IAEA has assisted national laboratories and regional laboratory networks since the 1970s through its Reference Products for Science and Trade programme. This is accomplished through the production of certified reference materials, training in quality assurance and the evaluation of measurement performance by organizing worldwide and regional interlaboratory comparison exercises and proficiency tests.

This publication describes the production of the certified reference material IAEA-435A which is based on a new characterization study of the existing reference material IAEA-435 produced by the IAEA in 2006.

IAEA-435A was produced following the requirements of international standards ISO 17034:2016 and ISO Guide 35:2017. This certified reference material is a fish homogenate sample with certified mass fractions of organochlorines and polybrominated diphenyl ethers. The assigned values and their associated uncertainties were derived from the results provided by selected laboratories with demonstrated technical and quality competencies, following the guidance given in international standards for the production of reference materials.

The material is intended to be used for quality control and the assessment of method performance for a number of organic analytes listed in the Stockholm Convention of Persistent Organic Pollutants and many environmental monitoring programmes.

The IAEA is grateful to the Government of Monaco for the support provided to its Marine Environment Laboratories and to all laboratories participating in the characterization study of this reference material. The IAEA officers responsible for this publication were I. Tolosa, R. Cassi and D. Huertas of the Division of Marine Environment Laboratories.

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1. INTRODUCTION

1.1. BACKGROUND

The Marine Environmental Studies Laboratory (MESL) of the IAEA-Nuclear Applications Marine Environment Laboratories (IAEA-NAML) provides assistance to Member States' laboratories to enhance the quality of analytical measurement results for trace elements and organic contaminants in marine environmental samples. This is achieved through the production of certified reference materials, the 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 NAML's subprogramme "Production of IAEA Certified Reference Materials and Proficiency Tests for the quality assurance of contaminant analysis in marine samples". 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 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) has become effective in 2004, a great number of different analytical methodologies have been extensively developed and there is a great need for CRMs to underpin the measurement of these contaminants in the marine environment. While there are several CRMs certified for organic contaminants, there is still a noticeable lack of matrix CRMs, in particular for POPs in fish homogenate samples, where the concentrations levels are in the low range of $\mu g\ kg^{-1}$.

1.2. OBJECTIVE

In order to strengthen data quality assurance in the analysis of POPs and other priority substances in marine samples, MESL has produced a fish homogenate CRM for the determination of a great number of organic analytes listed as Persistent Organic Pollutants (POPs) by the Stockholm Convention and included in many environment monitoring programmes.This CRM is intended for the assessment of method performance and quality control in laboratories analysing persistent organic pollutants in the marine environment.

1.3. SCOPE

The scope of this publication is to describe and provide information on the sample preparation methodology and on the assignment of property values with their associated uncertainties for a number of persistent organic contaminants in a fish homogenate sample. Certification of the mass fractions of major POPs was accomplished, including polychlorinated biphenyl congeners (PCBs), chlorinated pesticides and polybrominated diphenyl ethers (PBDEs). Results on other minor POPs are also provided as information values. The basic principles for evaluation of measurement uncertainty were followed according to ISO Guide 35 [1] and the Guide to the Expression of Uncertainty in Measurement (GUM) [2]. The uncertainty components from the characterization, heterogeneity and instability were combined.

1.4. STRUCTURE

This publication is structured in five sections, the first being the Introduction. The second section reports the methodology used for the preparation of the reference material, including the selection of laboratories for the characterization campaign, and all procedures for the homogeneity, stability and characterization of the material. This provides a foundation for the subsequent section 3 which covers

the results and discussion on the determination of assigned values and uncertainties. Then, section 4 provides the information on the metrological traceability and commutability of the current CRM. Final section 5 addresses the conclusions.

2. METHODOLOGY

2.1. COLLECTION AND PREPARATION OF THE MATERIAL

A 150 kg sample of Tuna (*Thunnus thynnus*) was collected from the Mediterranean Sea in 2004. This sample was freeze-dried, ground, and sieved at 250 µm using a stainless-steel sieve. This powder was homogenized by mixing it in a stainless-steel rotating drum for two weeks. Then, aliquots of about 30 g were packed into glass bottles with aluminum screw caps, labeled IAEA-435, and sealed with Teflon tape. This material was previously used for a world-wide ILC in 2004-2005, from which the reported values were used to produce the IAEA 435 reference material. This reference material provides information values for organochlorine compounds and petroleum hydrocarbons [3].

In 2019, the IAEA-435 bottles were labelled as ILC-IAEA-MESL-2019-01-OC and used in parallel for the characterization exercise as well as for the worldwide Interlaboratory Comparison (ILC) on the Determination of Organochlorine Compounds, Polybrominated Diphenyl Ethers and Polycyclic aromatic Hydrocarbons in a Fish sample. In the ILC, 51 laboratories from 28 countries reported data on organochlorine compounds, polybrominated diphenyl ethers and polycyclic aromatic hydrocarbons [4].

After the characterization and certification exercise the remaining bottles were labelled: IAEA-435A.

2.2. SELECTION OF LABORATORIES FOR THE CHARACTERIZATION STUDY

The selection of participating laboratories was based on the performance demonstrated in previous certification campaigns and on the results provided during a previous ILC for the same compounds in fish. 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. As the candidate reference material was also used in parallel for the worldwide ILC [4], some few additional participants, exhibiting reliable data and quality control in the ILC, were also invited to increase the data set of some compounds. Once these new laboratories agreed to be included in the characterization campaign, additional information such as calibrants and method validation documents were collected to evaluate the technical integrity of the reported data.

Each participating laboratory received the test sample (two bottles for the characterization test and one bottle for the ILC exercise), accompanied by one information sheet and instruction for the use of the IAEA on-line results reporting system. Participants were requested to use the analytical procedures established in their laboratories to determine the mass fractions of as many compounds as possible from the provided list of PCBs, organochlorine pesticides, PBDEs and PAHs (see Annex). Only PCB, organochlorine pesticide and PBDE data were processed for the characterization campaign. PAHs were present at trace levels not fulfilling the criteria for certification. Information values for PAH compounds can be found on the worldwide interlaboratory comparison publication [4].

Participants were requested to make at least three independent determinations, and to report the results together with their uncertainties. Also requested were the results of the CRM used and a short description of the method applied. Participants were required to provide proof of traceability of obtained results to the International System of Units (SI), via standard calibration solutions and CRM applied as a part of their analytical procedures.

It was additionally requested that concentrations have to be calculated on a dry mass basis and given as ng g^{-1}, leaving as many significant figures as justified by the precision of the method used.

Participants were advised that if a compound was not detected by the method used, the corresponding limit of detection should be given rather than the statement “not detected”.

As described in the Annex, for each class of compounds, participants were required also to report information about:

- Water content (in %);
- Total lipids (mg/g);
- Extractable lipids (mg/g);
- Extraction technique;
- Quantity extracted;
- Solvent used;
- Sample clean up;
- Sample fractionation;
- Detection method;
- Injector;
- Gas chromatography (GC) or high performance liquid chromatography (HPLC) column;
- Results quantification (internal or external calibration);
- Statement on traceability of the obtained measurement results (standards and reference materials used);
- QA/QC;
- Surrogates spiked before extraction;
- Internal standards spiked before injection;
- Use of CRMs;
- Use of validated methods;
- Accreditation.

Participants were requested to report their data and information using an on-line reporting form. The laboratories participating in the characterization study are listed on page 89.

2.3. HOMOGENEITY ASSESSMENT

The homogeneity assessment shall ensure that all distributed units of the CRM carry the same property values within the stated uncertainty. 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 are usually required [1].

A homogeneity test was performed in 2019 using the bottles units of IAEA-435 in line with ISO 17034 requirements [5]. ISO Guide 35 [1] was followed for the design and evaluation of the homogeneity study. The between bottle homogeneity of the material was assessed by determining the concentration of selected organochlorine compounds and polybrominated diphenyl ethers in 2 sample aliquots of 3 g taken from 10 randomly selected bottles (about 3% of the total batch). The tested

compounds for each group of analytes (PCBs, organochlorine pesticides and PBDEs) were selected based on higher abundance in the commercial mixtures, as they are considered representative for the other components of the family group, and are also subject to less analytical variability. Homogeneity assessment was based on the analysis of variance (ANOVA) to calculate between-unit variation (s_{bb}) and within bottle heterogeneity (s_{wb}). The measurement repeatability of the method (s_{method}) was estimated as the relative standard deviation of 6 independent measurements of the target compounds, performed on the same bottle and within the same batch. All measurements were performed in MESL, IAEA Organic Chemistry Laboratory using previously validated methods. For organochlorine and PBDE compounds, subsamples of 3 g were extracted with a microwave oven, purified with sulphuric acid, fractionated by Florisil solid phase extraction (SPE) column, and determined by Gas Chromatography – electron capture detector (GC-ECD) and peak confirmation with gas chromatography with tandem mass spectrometry (GC/MSMS).

2.4. STABILITY STUDY

Stability information is important to determine the presence of any potential degradation of the analytes or matrix 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.

For short-term stability study, the candidate CRM is usually exposed to elevated/reduced temperature to elucidate whether any degradation can be expected during transport. The study is usually of short duration, typically not longer than 2-4 weeks and the measurements performed under repeatable conditions. As this is a recertification exercise, no stability test was performed (see. Section 3.2).

2.5. CHARACTERIZATION

Characterization refers to the process of determining 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 characterization was based on the results provided by selected laboratories with demonstrated competence, as described in section 2.2. The laboratories included in the characterization campaign provided most of their results with their method validation data in accordance with ISO 17025 requirements [6]. A formal accreditation was not mandatory for participation.

Figure 1, 2 and 3 exhibit graphic representations of the types of instruments used by participants to analyze the test sample for each group of compounds: PCBs, organochlorine pesticides and PBDEs. As expected, a clear predominance of the use of gas chromatography-mass spectrometry techniques for the measurement of all different groups of compounds was observed.

Table 1 reports abbreviations used in Figs 1, 2 and 3 and along this publication.

TABLE 1. INSTRUMENTAL TECHNIQUES

Method code	Instrumental technique
GC/ECD	Gas Chromatography – Electron Capture Detector
GC/ECD & GC/MSMS	Gas Chromatography – ECD and peak confirmation with GC/MSMS
GC/MS EI	Gas Chromatography–Mass Spectrometry, Electron Impact
GC/MSMS	Gas Chromatography - Tandem Mass Spectrometry
GC/HRMS	Gas Chromatography - High Resolution Mass Spectrometry

The characterization of the PCBs and organochlorine pesticides was based on the application of five different analytical techniques: gas chromatography/electron capture detector and peak confirmation with GC/MSMS (GC/ECD & GC/MSMS), gas chromatography coupled to tandem mass spectrometry (GC/MSMS), GC/HRMS, GC/ECD and GC/MS EI, as summarized in Figs 1 and 2.

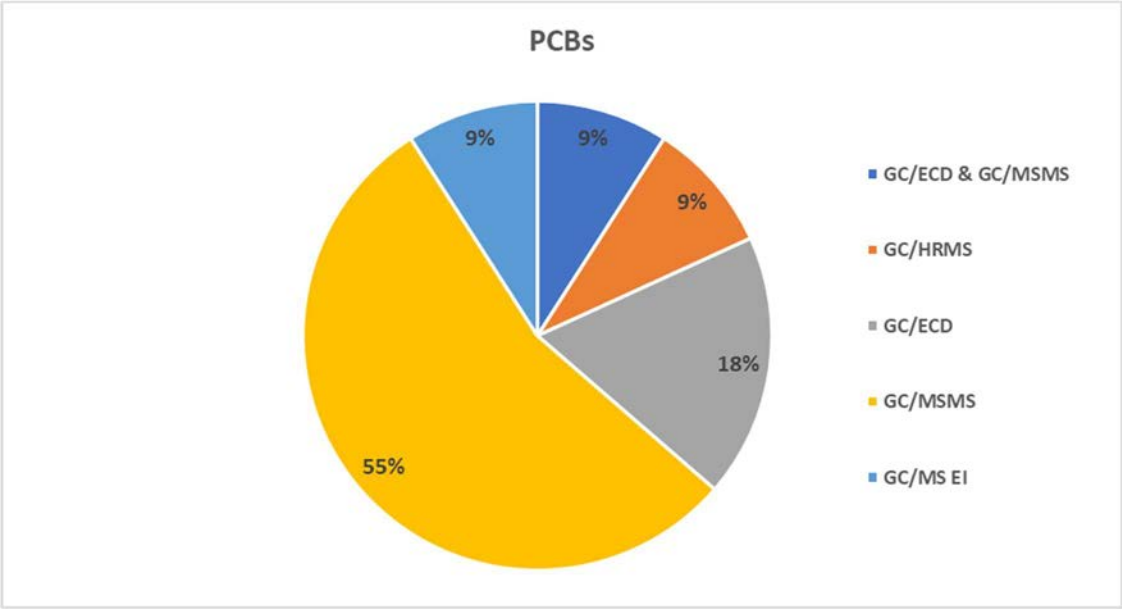

FIG. 1. Analytical methods used for the characterization of PCBs in the IAEA-435A Fish sample.

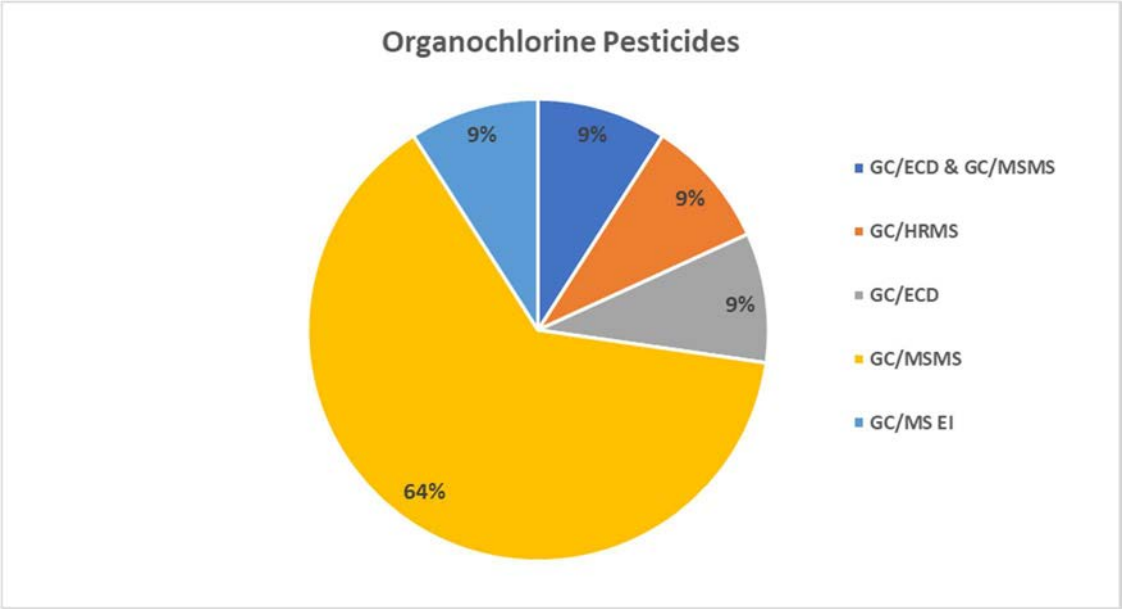

FIG. 2. Analytical methods used for the characterization of organochlorine pesticides in the IAEA-435A fish sample.

The characterization of the PBDEs was based on the application of three different analytical techniques: gas chromatography/electron capture detector and peak confirmation with GC/MS (GC/ECD & GC/MSMS), GC/HRMS and gas chromatography coupled to tandem mass spectrometry (GC/MSMS), as summarized in Fig 3.

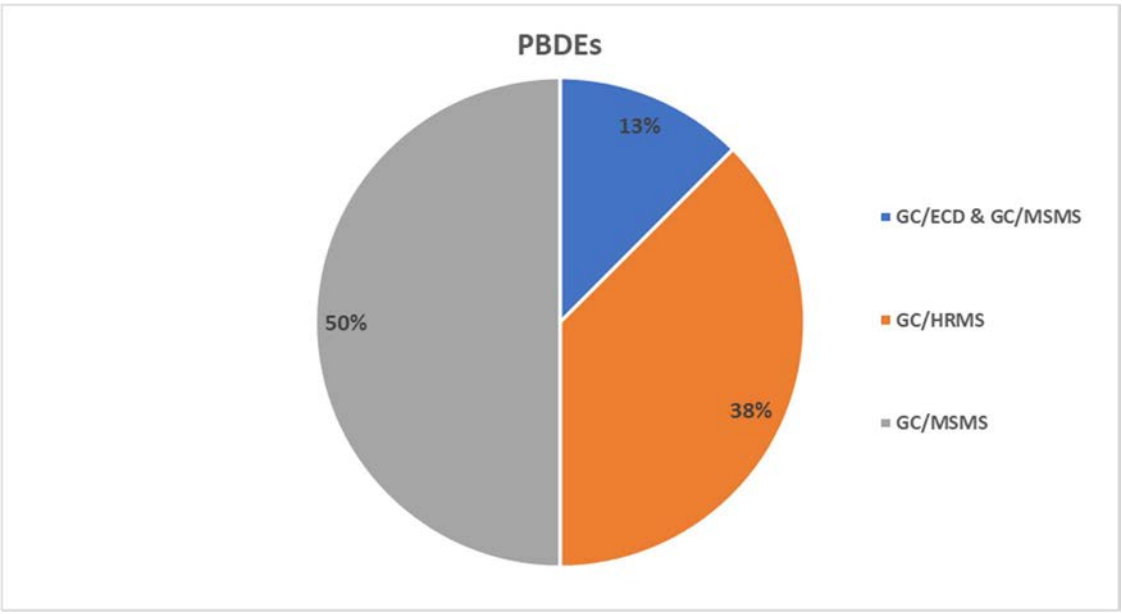

FIG. 3. Analytical methods used for the characterization of PBDEs in the IAEA-435A fish sample.

The number of independent datasets obtained was 11 for both PCBs and organochlorinated pesticides, and was 8 for PBDEs. For the assignment of the property values, the unweighted mean of the means was considered the most reliable estimate of the property values of the target compounds [1]. The basic principles for evaluation of measurement uncertainty were followed according to the ISO Guide

35 [1] and the Guide to the Expression of Uncertainty in Measurement (GUM) [2]. The uncertainty components from the characterization, heterogeneity and instability were combined.

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 5.0 ± 0.7% at the time of the preparation of this sample in 2004 [3] and 7.0 ± 0.1% in 2019. As the moisture content may change with the ambient humidity and temperature, it was recommended that participants determine the water content of the reference sample from a separate sub-sample (not the one taken for analysis of contaminants), by drying to a constant weight for at least 8 hours at 105 °C at the time of analysis in the laboratory.

3. RESULTS AND DISCUSSION

3.1 HOMOGENEITY TEST

The homogeneity (between and within bottles) of the material was assessed for organochlorine compounds and polybrominated diphenyl ethers by determining the concentration of these compounds in 10 bottles randomly selected from the IAEA-435 units. Each bottle unit was extracted and analyzed in duplicate, resulting in 2 independent data values by bottle unit. A one-way variance analysis of the results indicated that the material can be considered homogeneous.

Grubbs-tests at 95% and 99% confidence levels were performed to identify potentially outlying individual results as well as outlying bottle means. Few unit means were detected as outliers at 95% confidence levels for PCB 170, PCB 201, BDE154 and BDE153, but they were retained as they followed a normal distribution, and no technical reasons were identified for excluding those particular results.

Quantification of between-unit homogeneity was estimated according to ISO Guide 35 [1] by analysis of variance (ANOVA) which can separate the between-unit variation (s_{bb}) from the within-unit variation (s_{wb}). The latter is equivalent to the method repeatability if the individual aliquots are representative for the whole unit. ANOVA allows the calculation of within-unit standard deviation s_{wb} as well as between-units standard deviation s_{bb}:

$$s_{wb} = u_{wb} = \sqrt{MS_{wb}} \quad (1)$$

$$s_{bb} = u_{bb} = \sqrt{\frac{MS_{bb} - MS_{wb}}{n}} \quad (2)$$

where s_{bb} and s_{wb} are estimates of the true standard deviations and are therefore subject to random fluctuations. In some cases, the mean square between groups (MS_{bb}) can be smaller than the mean squares within groups (MS_{wb}), resulting in negative arguments under the square root used for the estimation of the between-unit variation. In this case, u^*_{bb}, the maximum between-unit variability that could be masked by method repeatability, was calculated as described by Linsinger et al. [7]:

$$u^*_{bb} = \frac{s_{wb}}{\sqrt{n}} \sqrt[4]{\frac{2}{\nu_{MSwb}}} \quad (3)$$

where: n is the number of replicate sub-samples per bottle; and ν_{MSwb} is the degrees of freedom of MS_{wb}.

As presented in Table 2, the between-unit variations (s_{bb} and u_{bb}*) for most of the selected compounds were between 1.2 and 4.1%, small enough to ensure the homogeneity of the material at 3 g sample size. The within-unit variation (s_{wb}) derived from the ANOVA calculation ranged between 3.3 and 8.7%, which is similar as the variation of the measurements (s_{meas}: 1.8-5.6%) defined as the intrinsic variability of the method (s_{method}) divided by the square root of the number of replicates from the same bottle (n=6) analyzed in the same batch [7].

As the within-unit variation was relatively high due to the variation of the measurements, the uncertainty u_{hom} associated with inhomogeneity of the material was estimated according to the ISO Guide 35 [1] by Eq. 4:

$$u_{hom} = \sqrt{u_{wb}^2 + u_{bb}^2} \quad (4)$$

where the u_{wb} was derived from ANOVA and the u_{bb} was taken as the maximum values of the between-unit variations (s_{bb} and u_{bb}*). As presented in Table 2, the uncertainty contribution related to inhomogeneity was estimated to range from 3.9 to 9.6 %. The scattered homogeneity uncertainty values obtained for the different congeners of the same group of analytes (e.g. PCBs, PBDEs) confirms that the variability is more of an artefact derived from the variability of the measurement for each individual congener, rather than inhomogeneity of the material. Thus, as a conservative approach, we set the uncertainty associated with inhomogeneity at 8% for all analytes. This value is certainly overestimated as it combines both (u_{wb} and u_{bb}) to allow for the deficient precision of the measurement procedure used for the homogeneity study.

TABLE 2. THE ESTIMATE OF INHOMOGENEITY CONTRIBUTIONS TO THE TOTAL UNCERTAINTY FOR THE SELECTED PCBs, ORGANOCHLORINE PESTICIDES AND PBDEs COMPOUNDS AND VARIATION OF THE MEASUREMENTS.

Compound	$s_{wb\ rel}$	$s_{bb\ rel}$	$u_{bb\ rel}^{*}$	$s_{meas\ rel}$	$u_{hom,\ rel}$
	%	%	%	%	%
PCB 28	4.7	[1)]	2.3	2.6	5.3
PCB 31	6.1	[1)]	3.0	3.5	6.8
PCB 52	3.3	2.1	1.6	1.8	3.9
PCB 99	4.9	2.5	2.4	3.4	5.5
PCB 101	4.0	1.2	1.9	3.4	4.4
PCB 118	5.9	4.3	2.9	3.0	7.3
PCB 138	6.9	[1)]	3.4	3.0	7.7
PCB 149	5.0	[1)]	2.4	3.4	5.5
PCB 151	6.9	[1)]	3.4	2.0	7.7
PCB 153	6.6	[1)]	3.2	2.9	7.3
PCB 170	5.0	3.7	2.4	3.4	6.2
PCB 174	3.8	1.7	1.9	3.4	4.2
PCB 180	5.0	2.8	2.4	3.5	5.7
PCB 183	4.6	[1)]	2.3	2.3	5.2
PCB 187	4.5	[1)]	2.2	3.2	5.0
PCB 194	5.7	1.9	2.8	3.0	6.4
PCB 195	6.9	[1)]	3.3	3.3	7.6
PCB 201	5.3	3.8	2.6	4.0	6.5
PCB 206	4.2	2.0	2.0	3.6	4.6
PCB 209	6.1	[1)]	3.0	3.2	6.8
pp'DDT	6.6	5.5	3.2	4.3	8.6
BDE 154	7.8	3.2	3.7	5.6	8.6
BDE 153	8.7	2.2	4.1	3.4	9.6

[1)] Not defined due to negative argument under the square root

3.2. RESULTS FOR STABILITY STUDY

3.2.1. Short-term stability study

No short-term stability test was performed in this freeze-dried material. Based on previous short-term stability results on CRMs of persistent organic analytes, the target compounds in the freeze-dried matrix do not show any significant trend of degradation over the timeframe at different temperatures (+20ºC and 40ºC) [8, 9]. These outcomes are supported by the chemical nature of the persistent organic pollutants, which exhibit high chemical stability and persistence in the natural environmental matrices. Therefore, no additional uncertainty with respect to instability due to transport is taken into account, and the uncertainty associated with short-term stability under transport conditions is set 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. This assessment relates to CRMs for persistent organic analytes at their natural concentration level and stored under recommended storage conditions to obtain information about the stability during the period of validity. Based on prior evidence with this type of matrices, and the high chemical stability and persistence of the targeted persistent organic pollutants, the freeze-dried CRMs have proven to be stable for more than 10 years, provided that the matrix material is stored in the dark at temperature below 30 ºC [10, 11, 12, 13]. Further evidence for the long-term stability of this material is evidenced by the relatively close assigned values obtained in this certification exercise compared to those assigned values obtained in 2006 for the major components [3]. The material stored in glass bottles with airtight lid is expected to be stable and the uncertainty associated with stability (u_{stab}) has been set to zero.

3.3. DETERMINATION OF ASSIGNED VALUES AND UNCERTAINTIES

The data provided by the participant laboratories were first checked for their validity based on the fully documented method and performance in the previous and current interlaboratory comparisons. Criteria considered during the technical evaluation of the dataset includes their performance in the ILC-IAEA-MESL-2019-01-OC fish sample and other external PTs, the quality of the method validation and analytical method applied, and the coherence between method repeatability extracted from the measurement dataset and reported uncertainties. The use of CRM/RMs and reporting uncertainties were not considered discriminatory as CRM/RMs for all the targeting analytes in the same biological matrix are not readily available. Based on those criteria, the measurement result of trans-chlordane from laboratory 78 was rejected as only one value was reported. Exceptionally, the lower repeatability for PCB 28 was considered acceptable due to the close coelution with the PCB 31 congener.

The characterization datasets resulted from 1 to 11 measurement results for each target compound. As each participant used different extraction techniques/solvents followed by their own fractionation procedures and GC separation, the data set is expected to provide a certain scatter. Values given as below limit of detection or below limit of quantification were excluded for the statistical evaluation. All accepted sets of results were submitted to the following statistical tests: Grubbs and Dixon's test to detect outliers with respect to the mean and Kolmogorov-SmirNov's test for the normality test.

For PCBs, outliers at 99% were found for PCB 110 (lab 25 and 40) and PCB 146 (lab 25), and then all data sets were normally distributed.

For organochlorine pesticides, one outlier each at 99% were found for *op*'-DDE (lab 79), *trans*-Chlordane (lab 42) and *cis*-Nonachlor (lab 72). The data sets were normally distributed after rejecting the outliers.

For PBDEs, all data sets were normally distributed and not outliers at 99% were found.

The median, unweighted mean of the means and robust mean from the ISO standard 13528 [14] were calculated for each individual compound and compared (Tables 3, 4 and 5). For the unweighted mean, all data were retained, except the outliers at 99% and the median and robust mean were calculated for datasets of $n \geq 7$. No significant differences were observed for the major compounds and the reference values obtained with the unweighted mean of the means were considered the most reliable estimates of the property values of the target compounds [1]. For compounds with concentration levels very close to the detection limit (HCHs, heptachlor, aldrin, endrin, endosulfans, and BDEs 17, 85, 183,

209), an informative value lower than detection limit was attributed based on expert judgment from the values provided by reference laboratories using HRMS and MS/MS.

The evaluation of uncertainties associated with the assigned property values was conducted according to ISO Guide 35 [1]. The combined uncertainty of the assigned property value of the CRM was calculated combining the individual standard uncertainties associated with the characterization (u_{char}), homogeneity (u_{hom}), short term stability (u_{short}) and long-term stability (u_{stab}) using the law of propagation of uncertainty. The uncertainty components derived from the short term and long term stability was insignificant and assumed to be zero. The final expanded uncertainty was calculated by multiplying the combined uncertainty by a coverage factor. It was calculated as shown in Eq. 9:

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

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

u_{hom} was calculated as described in section 3.1.

u_{char} for all target analytes was calculated as in ISO Guide 35 [1] using Eq. 10:

$$u_{char} = \frac{s}{\sqrt{p}} \quad (10)$$

The final assigned values are shown in Tables 6, 7 and 8 together with their individual characterization uncertainty and final expanded uncertainty.

TABLE 3. COMPARISON OF DIFFERENT MEANS FOR PCBs

Compound	No. Results accepted	Mean of the means µg kg^{-1}	Median µg kg^{-1}	Robust mean µg kg^{-1}	Outliers 95%	Outliers 99%
PCB 8	2	<0.30*	-	-	0	0
PCB 18	2	0.52	-	-	0	0
PCB 28	10	1.32	1.01	1.25	0	0
PCB 31	5	0.61	-	-	0	0
PCB 44	3	1.73	-	-	0	0
PCB 49	2	2.15	-	-	0	0
PCB 52	11	3.97	3.99	3.89	1	0
PCB 66	2	8.59	-	-	0	0
PCB 70	1	5.70	-	-	0	0
PCB 74	1	4.35	-	-	0	0
PCB 87	2	6.57	-	-	0	0
PCB 95	3	7.02	-	-	0	0
PCB 97	2	5.34	-	-	0	0
PCB 99	4	18.83	-	-	0	0
PCB 101	11	23.35	24.88	23.45	0	0
PCB 105	8	8.28	8.54	8.57	1	0
PCB 110	2	15.12	-	-	0	2
PCB 118	11	28.00	26.50	27.69	0	0
PCB 128	3	10.51	-	-	1	0
PCB 138	10	72.16	64.89	68.31	1	0
PCB 146	2	19.03	-	-	0	1
PCB 149	7	27.67	26.93	27.57	0	0
PCB 151	4	9.37	-	-	0	0
PCB 153	10	94.26	89.65	92.81	0	0
PCB 156	8	3.85	3.94	3.94	1	0
PCB 170	6	11.75	-	-	0	0
PCB 174	2	7.67	-	-	0	0
PCB 177	4	8.94	-	-	0	0
PCB 180	11	34.19	36.03	34.54	0	0
PCB 183	4	10.13	-	-	2	0
PCB 187	5	32.33	-	-	0	0
PCB 194	5	3.84	-	-	0	0
PCB 195	2	1.32	-	-	0	0
PCB 201	2	3.59	-	-	0	0
PCB 206	2	3.22	-	-	0	0
PCB 209	3	1.93	-	-	0	0

*For compounds "< ", no assigned value was calculated as most participating laboratories using mass spectrometry provided values lower than their detection limit

TABLE 4. COMPARISON OF DIFFERENT MEANS FOR ORGANOCHLORINE PESTICIDES

Compound	No. Results accepted	Mean of the means µg kg^{-1}	Median µg kg^{-1}	Robust mean µg kg^{-1}	Outliers 95%	Outliers 99%
HCB	10	2.65	2.80	2.68	0	0
α-HCH	9	<0.15*	-	-		
β-HCH	9	<0.25*	-	-		
γ-HCH- Lindane	10	<0.15*	-	-		
δ-HCH	5	<0.15*	-	-		
pp'-DDD	10	13.59	12.08	12.46	0	0
pp'-DDE	10	134.40	140.61	134.40	0	0
pp'-DDT	11	18.32	18.17	18.32	0	0
op'-DDE	6	2.09	-	-	0	1
op'-DDD	7	2.67	2.77	2.68	0	0
op'-DDT	9	7.15	7.05	7.16	0	0
Heptachlor	8	<0.16*	-	-		
Aldrin	8	<0.10*	-	-		
Dieldrin	5	5.46	-	-	0	0
Endrin	6	<0.15*	-	-		
cis-Chlordane	7	7.06	7.46	7.06	0	0
trans-Chlordane	3	0.38	-	-	0	1
cis-Nonachlor	3	11.89			0	1
trans-Nonachlor	5	20.21			0	0
α-Endosulfan	6	<0.30*	-	-		
β-Endosulfan	5	<0.10*	-	-		
Endosulfan sulfate	3	<0.30*	-	-		
Heptachlor epoxide	4	0.73	-	-	0	0

*For compounds "< ", no assigned value was calculated as most participating laboratories using mass spectrometry provided values lower than their detection limit

TABLE 5. COMPARISON OF DIFFERENT MEANS FOR PBDEs

Compound	No. Results accepted	Mean of the means µg kg^{-1}	Median µg kg^{-1}	Robust mean µg kg^{-1}	Outliers 95%	Outliers 99%
BDE 17	3	<0.10	-	-		
BDE 28	8	1.26	1.31	1.30	1	0
BDE 47	8	22.93	22.96	22.93	0	0
BDE 49	3	2.19	-	-	0	0
BDE 66	5	2.48	-	-	0	0
BDE 85	4	<0.10*	-	-		
BDE 99	7	1.44	1.37	1.40	1	0
BDE 100	8	7.29	7.40	7.39	0	0
BDE 153	7	0.70	0.68	0.68	2	0
BDE 154	8	5.04	5.06	5.01	0	0
BDE 183	4	<0.10*	-	-		
BDE 209	4	<1.00*	-	-		

*For compounds "< ", no assigned value was calculated as most participating laboratories using mass spectrometry provided values lower than their detection limit

TABLE 6. MEAN OF THE MEANS, CHARACTERIZATION UNCERTAINTY AND RELATIVE EXPANDED UNCERTAINTY FOR PCBs

Compound	No. Results	Mean of the means µg kg^{-1}	u_{char_rel} %	U_{rel} (k=2) %
PCB 8	2	<0.30*	-	-
PCB 18	2	0.52	31	64
PCB 28	10	1.32	15	34
PCB 31	5	0.61	9	25
PCB 44	3	1.73	10	26
PCB 49	2	2.15	2	16
PCB 52	11	3.97	7	21
PCB 66	2	8.59	22	47
PCB 70	1	5.70	-	-
PCB 74	1	4.35	-	-
PCB 87	2	6.57	2	17
PCB 95	3	7.02	18	39
PCB 97	2	5.34	7	22
PCB 99	4	18.8	15	35
PCB 101	11	23.4	6	20
PCB 105	8	8.28	7	22
PCB 110	2	15.1	2	16
PCB 118	11	28.0	5	19
PCB 128	3	10.5	16	35
PCB 138	10	72.2	6	20
PCB 146	2	19.0	0	16
PCB 149	7	27.7	6	20
PCB 151	4	9.37	7	21
PCB 153	10	94.3	3	17
PCB 156	8	3.85	5	19
PCB 170	6	11.8	5	18
PCB 174	2	7.67	4	18
PCB 177	4	8.94	10	26
PCB 180	11	34.2	5	19
PCB 183	4	10.1	15	34
PCB 187	5	32.3	11	27
PCB 194	5	3.84	6	20
PCB 195	2	1.32	14	33
PCB 201	2	3.59	12	30
PCB 206	2	3.22	2	16
PCB 209	3	1.93	7	21

*For compounds "< ", no assigned value was calculated as most participating laboratories using mass spectrometry provided values lower than their detection limit

TABLE 7. MEAN OF THE MEANS, CHARACTERIZATION UNCERTAINTY AND RELATIVE EXPANDED UNCERTAINTY FOR ORGANOCHLORINE PESTICIDES

Compound	No. Results	Mean of the means µg kg^{-1}	u_{char_rel} %	U_{rel} (k=2) %
HCB	10	2.65	10	25
α-HCH	9	<0.15*	-	-
β-HCH	9	<0.25*	-	-
γ-HCH- Lindane	10	<0.15*	-	-
δ-HCH	5	<0.15*	-	-
pp'-DDD	10	13.6	10	25
pp'-DDE	10	134	6	19
pp'-DDT	11	18.3	7	22
op'-DDE	6	2.09	11	27
op'-DDD	7	2.67	10	26
op'-DDT	9	7.15	10	25
Heptachlor	8	<0.16*	-	-
Aldrin	8	<0.10*	-	-
Dieldrin	5	5.46	13	30
Endrin	6	<0.15	-	-
cis-Chlordane	7	7.06	12	29
trans-Chlordane	3	0.380	7	21
cis-Nonachlor	3	11.3	0.3	16
trans-Nonachlor	5	20.2	8	23
α-Endosulfan	6	<0.30*	-	-
β-Endosulfan	5	<0.10*	-	-
Endosulfan sulfate	3	<0.30*	-	-
Heptachlor epoxide	4	0.73	15	34

*For compounds "< ", no assigned value was calculated as most participating laboratories using mass spectrometry provided values lower than their detection limit.
** Robust mean of the reported values

TABLE 8. MEAN OF THE MEANS, CHARACTERIZATION UNCERTAINTY AND RELATIVE EXPANDED UNCERTAINTY FOR PBDEs

Compound	No. Results	Mean of the means µg kg^{-1}	u_{char_rel} %	U_{rel} (k=2) %
BDE 17	3	<0.10	-	-
BDE 28	8	1.26	6	20
BDE 47	8	22.93	3	17
BDE 49	3	2.19	6	20
BDE 66	5	2.48	6	20
BDE 85	4	<0.10*	-	-
BDE 99	7	1.44	6	20
BDE 100	8	7.29	4	18
BDE 153	7	0.70	8	23
BDE 154	8	5.04	7	22
BDE 183	4	<0.10*	-	-
BDE 209	4	<1.00*	-	-

*For compounds "< ", no assigned value was calculated as most participating laboratories using mass spectrometry provided values lower than their detection limit

The results for the mass fractions of PCB congeners, organochlorine pesticides and PBDEs in IAEA-435A fish homogenate, as reported by the participants in this characterization, are presented in Appendix I and II. The laboratory means are plotted together with the assigned value denoted by a bold line, while the dashed lines represent assigned value ± expanded uncertainty (k=2) in all figures (as calculated with Eq. 9). The error bars represent the expanded uncertainty reported by participants. If a participant didn't report their uncertainties, they were calculated as 2 x $\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported by the participant. Tables are provided in place of graphical plots for compounds with concentration levels close to the detection limit.

As shown previously in Table 1, different independent analytical techniques (GC/ECD, GC/ECD & GC/MSMS, GC/MS EI, GC/MSMS and GC/HRMS) were used for the characterization of the material. A good agreement was observed for results obtained with different methods, which confirms the absence of any significant method bias and demonstrates the identity of the analyte.

The mean of the means of the laboratory were assigned as certified values, for those compounds where the assigned value was derived from at least 5 datasets, 3 analytical techniques, and its relative expanded uncertainties was less than 35 % of the assigned value.

These criteria were fulfilled for:

- 14 PCBs (PCB 28, PCB 31, PCB 52, PCB 101, PCB 105, PCB 118, PCB 138, PCB 149, PCB 153, PCB 156, PCB 170, PCB 180, PCB 187, PCB 194) where all relative expanded uncertainties were lower than 25% excepting PCB 28 with an expanded uncertainty of 34%;
- 10 organochlorine pesticides (HCB, *pp'*-DDD, *pp'*-DDE, *pp'*-DDT, *op'*-DDE, *op'*-DDD, *op'*-DDT, Dieldrin, *cis*-Chlordane, *trans*-Nonachlor) where the relative expanded uncertainties were lower than 30%; and
- 7 PBDEs (BDE 28, BDE 47, BDE 66, BDE 99, BDE 100, BDE 153, BDE 154) with relative expanded uncertainties lower than 25%.

The certified values together with their expanded uncertainty are presented in Tables 9, 10 and 11 for PCBs, organochlorine pesticides and PBDEs respectively.

Compounds that did not fulfill the criteria of certification are considered information values. They include PCB 8, PCB 18, PCB 44, PCB 49, PCB 66, PCB 70, PCB 74, PCB 87, PCB 95, PCB 97, PCB 99, PCB 110, PCB 128, PCB 146, PCB 151, PCB 174, PCB 177, PCB 183, PCB 195, PCB 201, PCB 206, PCB 209, *trans*-Chlordane, *cis*-Nonachlor, Heptachlor epoxide and BDE 49. Additional information values stated as lower than the detection limit were attributed to α-HCH, β-HCH, γ-HCH-Lindane, δ-HCH, Heptachlor, Aldrin, Endrin, α-Endosulfan, β-Endosulfan, Endosulfan sulfate, BDE 17, BDE 85, BDE 183, BDE 209, as most participant laboratories using mass spectrometry provided values lower than their detection limits. Tables 12, 13 and 14 show the information values for PCBs, organochlorine pesticides and PBDEs together with their associated expanded uncertainties when these could be calculated.

TABLE 9. CERTIFED VALUES FOR PCBs MASS FRACTIONS AND THEIR EXPANDED UNCERTAINTY (*k=2*) IN THE IAEA-435A FISH SAMPLE

Compound	Unit	Certified value[1]	*U (k=2)* [2]
PCB 28	µg kg^{-1}	1.32	0.45
PCB 31	µg kg^{-1}	0.61	0.15
PCB 52	µg kg^{-1}	3.97	0.83
PCB 101	µg kg^{-1}	23.4	4.8
PCB 105	µg kg^{-1}	8.3	1.8
PCB 118	µg kg^{-1}	28.0	5.3
PCB 138	µg kg^{-1}	72	15
PCB 149	µg kg^{-1}	27.7	5.6
PCB 153	µg kg^{-1}	94	16
PCB 156	µg kg^{-1}	3.85	0.73
PCB 170	µg kg^{-1}	11.8	2.2
PCB 180	µg kg^{-1}	34.2	6.4
PCB 187	µg kg^{-1}	32.3	8.9
PCB 194	µg kg^{-1}	3.84	0.77

[1] The value is the mean of the mean of the 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 evaluated according to ISO Guide 35 [1] 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 [2], corresponding to the level of confidence of about 95%.

TABLE 10. CERTIFED VALUES FOR ORGANOCHLORINE PESTICIDE MASS FRACTIONS AND THEIR EXPANDED UNCERTAINTY (*k=2*) IN THE IAEA-435A FISH SAMPLE

Compound	Unit	Certified value[1]	*U* (*k*=2) [2]
HCB	µg kg^{-1}	2.65	0.66
pp'-DDD	µg kg^{-1}	13.6	3.4
pp'-DDE	µg kg^{-1}	134	26
pp'-DDT	µg kg^{-1}	18.3	4.0
op'-DDE	µg kg^{-1}	2.10	0.56
op'-DDD	µg kg^{-1}	2.67	0.69
op'-DDT	µg kg^{-1}	7.2	1.8
Dieldrin	µg kg^{-1}	5.5	1.6
cis-Chlordane	µg kg^{-1}	7.1	2.1
trans-Nonachlor	µg kg^{-1}	20.2	4.6

[1] The value is the mean of the mean of the 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 evaluated according to ISO Guide 35 [1] 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 [2], corresponding to the level of confidence of about 95%.

TABLE 11. CERTIFED VALUES FOR PBDEs MASS FRACTIONS AND THEIR EXPANDED UNCERTAINTY (*k=2*) IN THE IAEA-435A FISH SAMPLE

Compound	Unit	Certified value[1]	*U* (*k*=2) [2]
BDE 28	µg kg^{-1}	1.26	0.25
BDE 47	µg kg^{-1}	22.9	3.9
BDE 66	µg kg^{-1}	2.48	0.50
BDE 99	µg kg^{-1}	1.44	0.29
BDE 100	µg kg^{-1}	7.3	1.3
BDE 153	µg kg^{-1}	0.70	0.16
BDE 154	µg kg^{-1}	5.0	1.1

[1] The value is the mean of the mean of the 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 evaluated according to ISO Guide 35 [1] 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 [2], corresponding to the level of confidence of about 95%.

TABLE 12. INFORMATION VALUES FOR PCBs MASS FRACTIONS AND THEIR EXPANDED UNCERTAINTY (*k=2*) IN THE IAEA-435A FISH SAMPLE

Compound	Unit	Information value[1]	*U (k=2)* [2]
PCB 8	µg kg^{-1}	<0.3	-
PCB 18	µg kg^{-1}	0.5	0.3
PCB 44	µg kg^{-1}	1.7	0.4
PCB 49	µg kg^{-1}	2.2	0.4
PCB 66	µg kg^{-1}	9	4
PCB 70	µg kg^{-1}	5.7	-
PCB 74	µg kg^{-1}	4.4	-
PCB 87	µg kg^{-1}	7	1
PCB 95	µg kg^{-1}	7	3
PCB 97	µg kg^{-1}	5	1
PCB 99	µg kg^{-1}	19	7
PCB 110	µg kg^{-1}	15	3
PCB 128	µg kg^{-1}	11	4
PCB 146	µg kg^{-1}	19	3
PCB 151	µg kg^{-1}	9	2
PCB 174	µg kg^{-1}	8	1
PCB 177	µg kg^{-1}	9	2
PCB 183	µg kg^{-1}	10	3
PCB 195	µg kg^{-1}	1.3	0.4
PCB 201	µg kg^{-1}	4	1
PCB 206	µg kg^{-1}	3.2	0.5
PCB 209	µg kg^{-1}	1.9	0.4

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

[2] Expanded uncertainty evaluated according to ISO Guide 35 [1] 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 [2], corresponding to the level of confidence of about 95%.

TABLE 13. INFORMATION VALUES FOR ORGANOCHLORINE PESTICIDE MASS FRACTIONS AND THEIR EXPANDED UNCERTAINTY (*k=2*) IN THE IAEA-435A FISH SAMPLE

Compound	Unit	Information value[1]	*U (k=2)* [2]
α-HCH	µg kg^{-1}	<0.2	-
β-HCH	µg kg^{-1}	<0.3	-
γ-HCH- Lindane	µg kg^{-1}	<0.2	-
δ-HCH	µg kg^{-1}	<0.2	-
Heptachlor	µg kg^{-1}	<0.2	-
Aldrin	µg kg^{-1}	<0.1	-
Endrin	µg kg^{-1}	<0.2	-
trans-Chlordane	µg kg^{-1}	0.38	0.08
cis-Nonachlor	µg kg^{-1}	11	2
α-Endosulfan	µg kg^{-1}	<0.3	-
β-Endosulfan	µg kg^{-1}	<0.1	-
Endosulfan sulfate	µg kg^{-1}	<0.3	-
Heptachlor epoxide	µg kg^{-1}	0.7	0.2

[1] The value is the mean of the mean of the accepted sets of data, each set being obtained by different laboratory. The information values are reported on dry mass basis and are traceable to the SI.
[2] Expanded uncertainty evaluated according to ISO Guide 35 [1] 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 [2], corresponding to the level of confidence of about 95%.

TABLE 14. INFORMATION VALUES FOR PBDEs MASS FRACTIONS AND THEIR EXPANDED UNCERTAINTY (*k=2*) IN THE IAEA-435A FISH SAMPLE

Compound	Unit	Information value[1]	*U (k=2)* [2]
BDE 17	µg kg^{-1}	<0.1	-
BDE 49	µg kg^{-1}	2.2	0.4
BDE 85	µg kg^{-1}	<0.1	-
BDE 183	µg kg^{-1}	<0.1	-
BDE 209	µg kg^{-1}	<1	-

[1] The value is the mean of the mean of the accepted sets of data, each set being obtained by different laboratory. The information values are reported on dry mass basis and are traceable to the SI.
[2] Expanded uncertainty evaluated according to ISO Guide 35 [1] 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 [2], 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 [15]. All laboratories participating in the IAEA-435A characterization exercise stated and provided evidence of using analytical standards (both calibrating and internal/surrogates standard solutions) with demonstrated SI traceability. The suppliers of the certificate standards were Dr. Ehrenstorfer, Cambridge Isotope Laboratories, Wellignton Laboratories, CPA Chem, AccuStandard, Ultra Scientific, Chiron, Tech lab, O2SI and A2S (Analytical Standard Solutions). Most of the methods used by the participating laboratories were validated using matrix certified reference materials (CRMs) from NIST (SRM 2974a, SRM 1947), Joint Research Centre (ERM-CE100), IAEA (IAEA-459) and Wellington Laboratories (WMF-02, WMF-03). In the absence of appropriate matrix CRMs, some participant laboratories validated their method using matrix reference materials (IAEA-406, IAEA-432, IAEA-435), spike matrices and proficiency test (PT) materials characterized by FAPAS proficiency tests (0685 Cod liver Oil, dioxins, PCBs and PBDEs). The fact that values reported by participants are based on calibration standard certificates of known purity with documented unbroken chain of calibrations, issued by accredited commercial companies, demonstrates that the assigned values derived from combining the individual results are traceable to International System of Units (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 analyte.

Commutability is a property of a RM, established by the closeness of agreement between A) the relation among the measurement results for a stated quantity in the material, attained according to two specified measurement procedures, and B) the relation among the measurement results for other specified materials [16].

A material is said to be commutable when an analyte in the routine test samples behaves similarly to the CRM with respect to the different measurement procedures. This implies that applying the procedures to the CRM would produce the same quantitative value as normal routine samples containing the same concentration of the analyte. In this respect, IAEA-435A is a natural marine fish sample and its analytical behavior should be the same as any other routine dried fish sample. The agreement between the data acquired with different analytical procedures for the IAEA-435A characterization study endorses the absence of any significant method bias, and shows commutability of the material for all certified organic compounds.

5. CONCLUSIONS

The combination of different data sets from at least three different analytical techniques has allowed the assignment of certified concentrations for 14 PCBs, 10 organochlorinated pesticides and 7 PBDEs following the recommendation of ISO Guide 35. The extensive characterization at relatively low concentration levels and associated uncertainties will make CRM 435A a valuable fish reference material. This CRM can be used to validate analytical methods for the determination of a large number of persistent organic contaminants both listed at the Stockholm Convention and included within environmental monitoring programs.

APPENDIX I: CERTIFIED VALUES

I.1. CERTIFIED VALUES FOR PCB CONGENERS

TABLE 15. RESULTS REPORTED BY PARTICIPANTS FOR PCB 28 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
3	1.01	0.05[1]	0.05	GC/MSMS	NIST 2974a
5	0.93	0.13[1]		GC/MSMS	IAEA-435, IAEA-432
7	1.78	0.22	0.40	GC/ECD	IAEA-406
10	1.75	0.05[1]	0.01	GC/MSMS	
25	0.68	0.04	0.03	GC/MSMS	
27	2.38	0.60[1]	0.02	GC/ECD	
40*	1.00	0.07	0.31	GC/HRMS	FAPAS 0685, IAEA-459
74*	0.73	0.01[1]	0.02	GC/MSMS	
78*	2.07	1.49[1]	0.20	GC/MSMS	
79	0.86	0.17	0.06	GC/ECD[2]	NIST 2974a, WMF-03

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

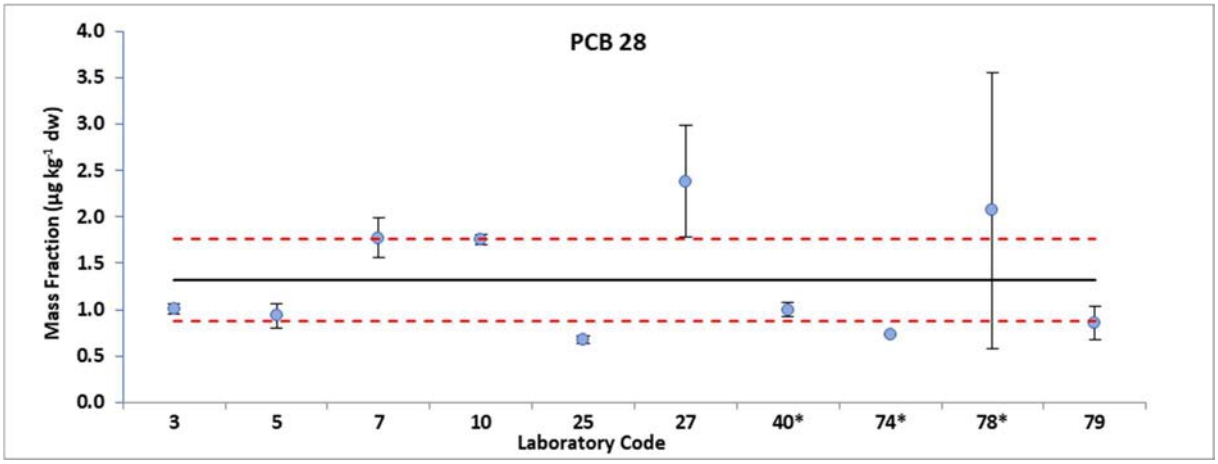

FIG. 4. Laboratory results for PCB 28 in IAEA-435A (µg kg^{-1} dw)

TABLE 16. RESULTS REPORTED BY PARTICIPANTS FOR PCB 31 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
25	0.49	0.03	0.03	GC/MSMS	
27	0.47	0.29[1]	0.02	GC/ECD	
40*	0.69	0.24	0.25	GC/HRMS	FAPAS 0685, IAEA-459
74*	0.61	0.01[1]	0.02	GC/MSMS	
79	0.77	0.15	0.05	GC/ECD[2]	NIST 2974a, WMF-03
Results not used for the assignment of the value					
78*[1]	<1.00			GC/MSMS	

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

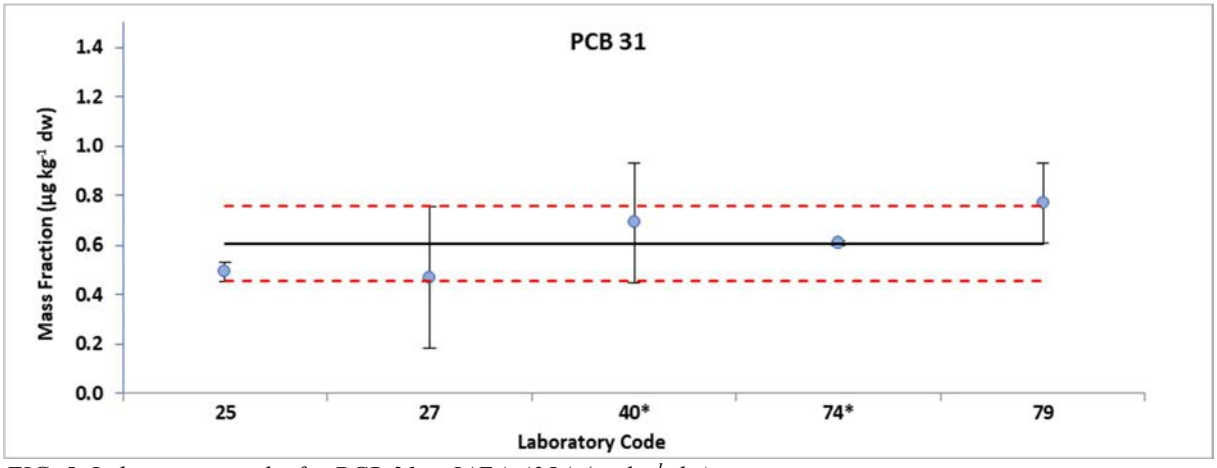

FIG. 5. Laboratory results for PCB 31 in IAEA-435A (µg kg^{-1} dw)

TABLE 17. RESULTS REPORTED BY PARTICIPANTS FOR PCB 52 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
3	2.95	0.62[1]	0.05	GC/MSMS	NIST 2974a
5	4.20	0.43[1]		GC/MSMS	IAEA-435, IAEA-432
7	4.42	0.57	0.40	GC/ECD	IAEA-406
10	4.08	0.11[1]	0.01	GC/MSMS	
25	3.06	0.17	0.03	GC/MSMS	
27	3.00	0.29[1]	0.02	GC/ECD	
40*	3.95	0.25	1.17	GC/HRMS	FAPAS 0685, IAEA-459
49*	3.99	0.41	0.10	GC/MS EI	IAEA-435, IAEA-451, NIST 1946
74*	3.49	0.07[1]	0.02	GC/MSMS	
78*	4.63	0.07[1]	0.30	GC/MSMS	
79	5.94	1.19	0.13	GC/ECD[2]	NIST 2974a, WMF-03

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

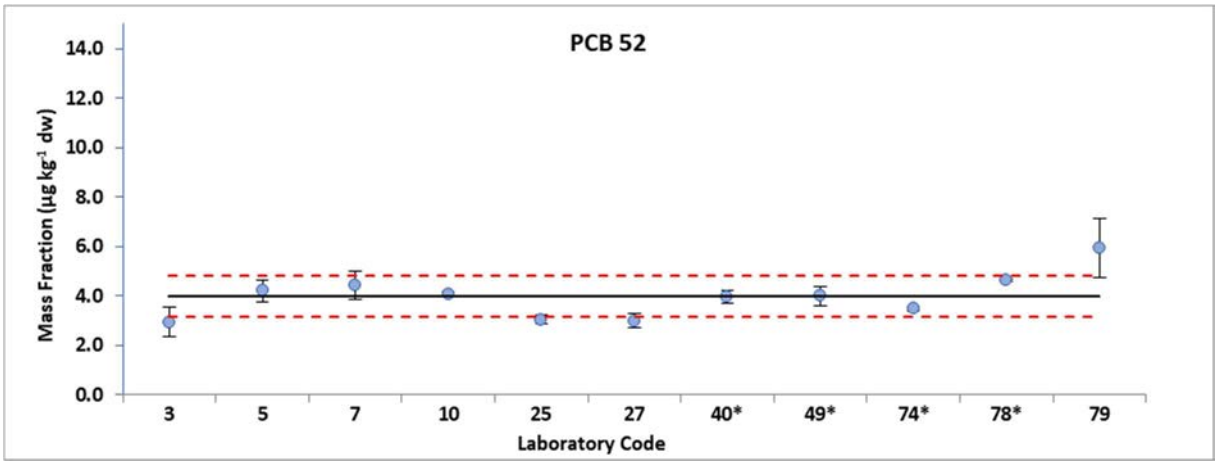

FIG. 6. Laboratory results for PCB 52 in IAEA-435A (µg kg^{-1} dw)

TABLE 18. RESULTS REPORTED BY PARTICIPANTS FOR PCB 101 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
3	20.1	1.1[1]	0.05	GC/MSMS	NIST 2974a
5	23.8	1.8[1]		GC/MSMS	IAEA-435, IAEA-432
7	25.1	3.7	0.40	GC/ECD	IAEA-406
10	31.3	0.4[1]	0.01	GC/MSMS	
25	14.8	0.9	0.03	GC/MSMS	
27	19.0	0.9[1]	0.02	GC/ECD	
40*	29.1	1.8	0.46	GC/HRMS	FAPAS 0685, IAEA-459
49*	18.3	1.9	0.10	GC/MS EI	IAEA-435, IAEA-451, NIST 1946
74*	24.87	0.08[1]	0.02	GC/MSMS	
78*	25.3	1.8[1]	0.30	GC/MSMS	
79	25.1	5.0	0.01	GC/ECD[2]	NIST 2974a, WMF-03

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

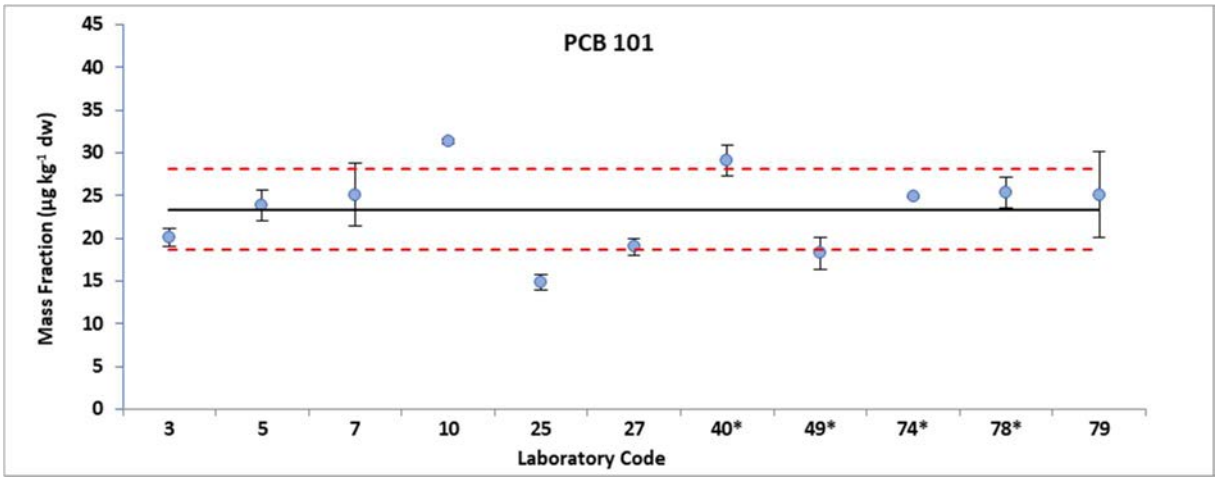

FIG. 7. Laboratory results for PCB 101 in IAEA-435A (µg kg^{-1} dw)

TABLE 19. RESULTS REPORTED BY PARTICIPANTS FOR PCB 105 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
3	4.50	0.31[1]		GC/MSMS	NIST 2974a
10	8.90	0.20[1]	0.01	GC/MSMS	
25	7.65	1.51	0.03	GC/MSMS	
27	8.18	1.07[1]	0.02	GC/ECD	
40*	10.3	0.6	0.09	GC/HRMS	FAPAS 0685, IAEA-459
74*	8.01	0.02[1]	0.01	GC/MSMS	
78*	9.07	0.47[1]	0.30	GC/MSMS	
79	9.63	1.93	0.03	GC/ECD[2]	NIST 2974a, WMF-03

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

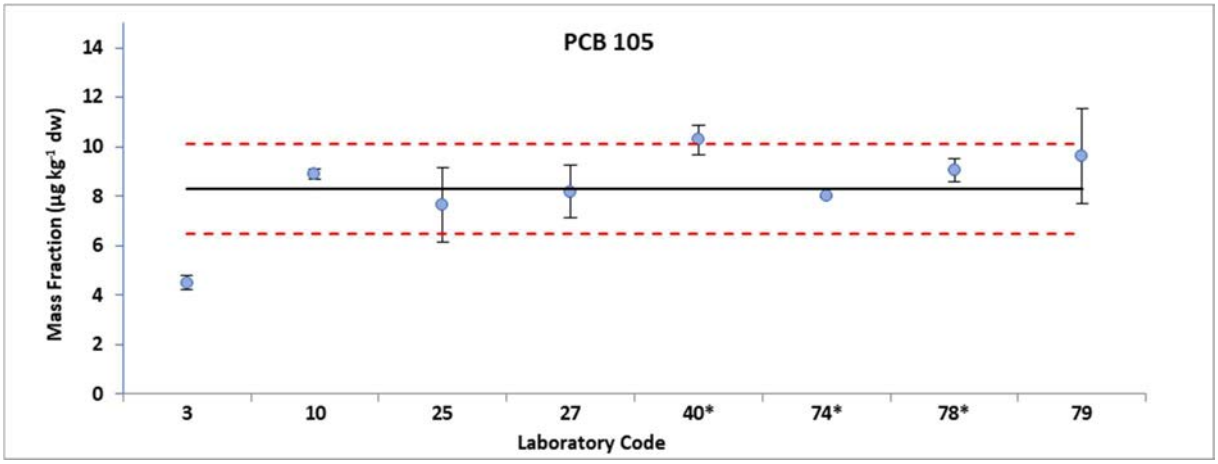

FIG. 8. Laboratory results for PCB 105 in IAEA-435A (µg kg^{-1} dw)

TABLE 20. RESULTS REPORTED BY PARTICIPANTS FOR PCB 118 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
3	25.0	0.7[1]	0.05	GC/MSMS	NIST 2974a
5	26.5	1.9[1]		GC/MSMS	IAEA-435, IAEA-432
7	20.7	2.6	0.40	GC/ECD	IAEA-406
10	34.3	0.6[1]	0.01	GC/MSMS	
25	22.5	2.0	0.03	GC/MSMS	
27	29.9	2.9[1]	0.02	GC/ECD	
40*	36.4	2.2	0.31	GC/HRMS	FAPAS 0685, IAEA-459
49*	26.4	2.9	0.10	GC/MS EI	IAEA-435, IAEA-451, NIST 1946
74*	29.2	0.5[1]	0.01	GC/MSMS	
78*	31.4	2.2[1]	0.30	GC/MSMS	
79	25.7	5.2	0.06	GC/ECD[2]	NIST 2974a, WMF-03

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

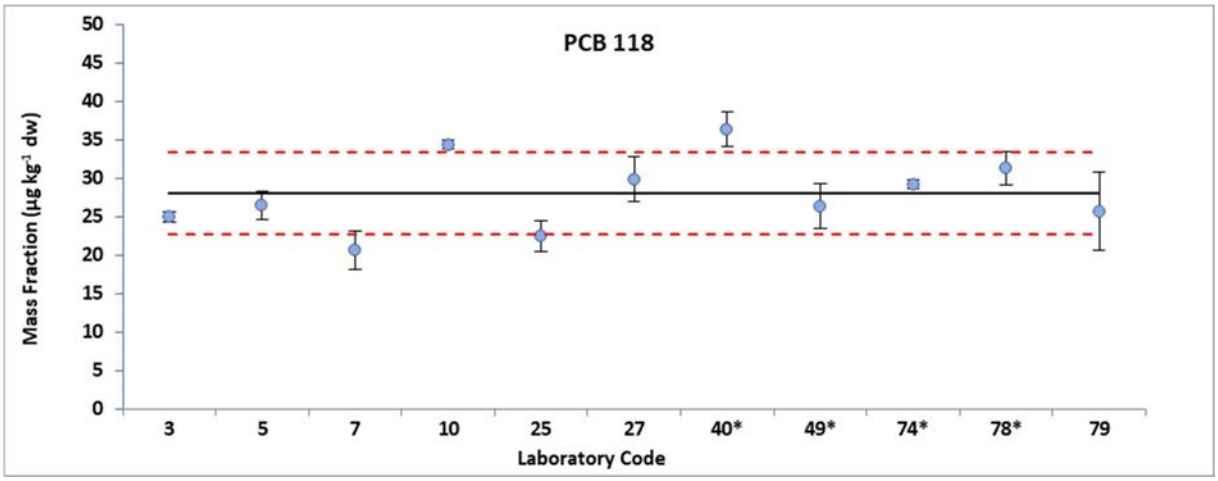

FIG. 9. Laboratory results for PCB 118 in IAEA-435A (µg kg^{-1} dw)

TABLE 21. RESULTS REPORTED BY PARTICIPANTS FOR PCB 138 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
3	65.1	1.9[1]	0.05	GC/MSMS	NIST 2974a
5	85.9	3.0[1]		GC/MSMS	IAEA-435, IAEA-432
10	64.2	2.9[1]	0.01	GC/MSMS	
25	58.8	9.9	0.03	GC/MSMS	
27	64.2	1.5[1]	0.02	GC/ECD	
40*	107	8	0.24	GC/HRMS	FAPAS 0685, IAEA-459
49*	64.7	7.2	0.10	GC/MS EI	IAEA-435, IAEA-451, NIST 1946
74*	63.0	4.3[1]	0.02	GC/MSMS	
78*	74.0	5.9[1]	0.30	GC/MSMS	
79	75.2	15.0	0.07	GC/ECD[2]	NIST 2974a, WMF-03

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

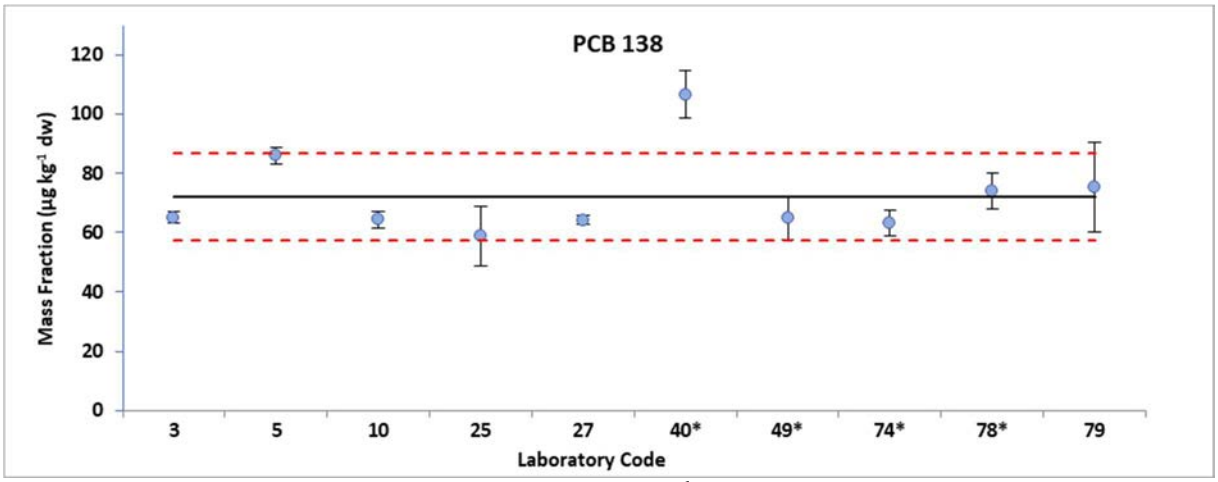

FIG. 10. Laboratory results for PCB 138 in IAEA-435A (µg kg^{-1} dw)

TABLE 22. RESULTS REPORTED BY PARTICIPANTS FOR PCB 149 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
10	26.9	0.7[1]	0.01	GC/MSMS	
25	23.4	0.9	0.03	GC/MSMS	
27	26.1	1.3[1]	0.02	GC/ECD	
40*	35.0	12.2	0.15	GC/HRMS	FAPAS 0685, IAEA-459
49*	22.7	2.5	0.10	GC/MS EI	IAEA-435, IAEA-451, NIST 1946
78*	32.6	2.6[1]	0.30	GC/MSMS	
79	27.0	5.4	0.02	GC/ECD[2]	NIST 2974a, WMF-03

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

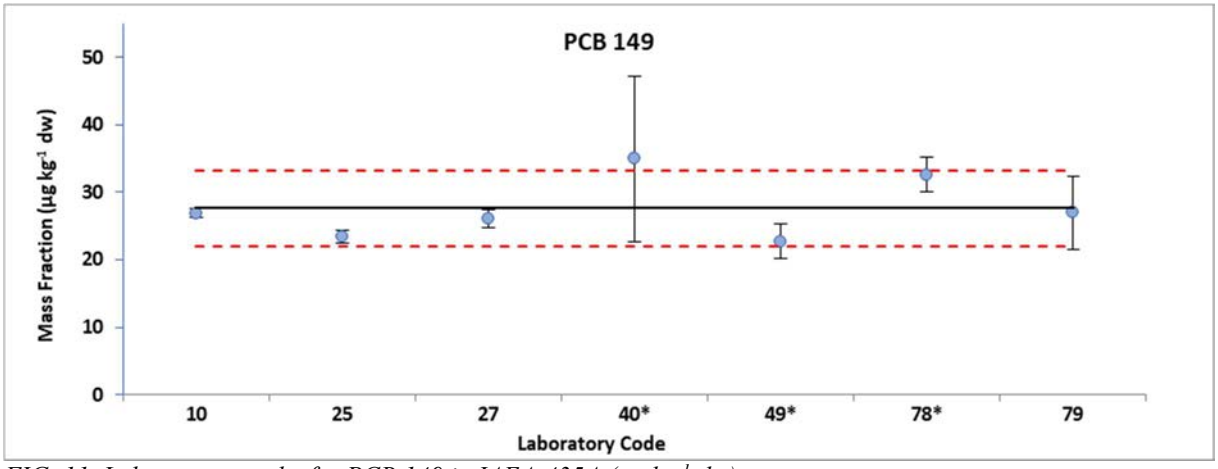

FIG. 11. Laboratory results for PCB 149 in IAEA-435A (µg kg^{-1} dw)

TABLE 23. RESULTS REPORTED BY PARTICIPANTS FOR PCB 153 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
3	85.1	1.8[1]	0.05	GC/MSMS	NIST 2974a
5	90.5	6.2[1]		GC/MSMS	IAEA-435, IAEA-432
7	82.4	12.0	0.40	GC/ECD	IAEA-406
10	107	3[1]	0.01	GC/MSMS	
25	86.7	10.3	0.03	GC/MSMS	
27	86.2	1.5[1]	0.02	GC/ECD	
40*	109	7	0.30	GC/HRMS	FAPAS 0685, IAEA-459
74*	105	1.0[1]	0.02	GC/MSMS	
78*	103	4[1]	0.30	GC/MSMS	
79	88.8	17.8	0.06	GC/ECD[2]	NIST 2974a, WMF-03

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

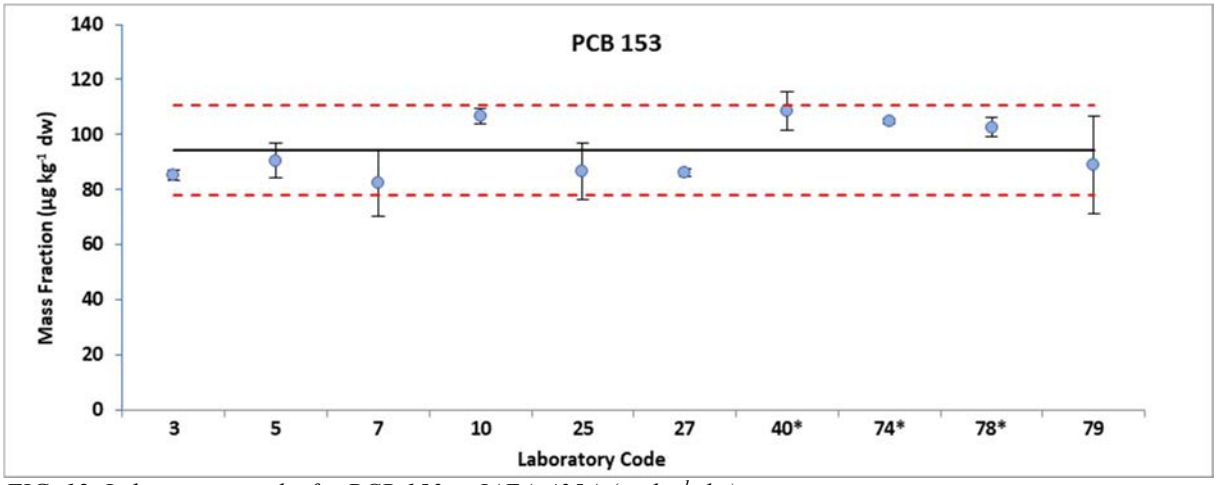

FIG. 12. Laboratory results for PCB 153 in IAEA-435A (µg kg^{-1} dw)

TABLE 24. RESULTS REPORTED BY PARTICIPANTS FOR PCB 156 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
3	2.63	0.24[1]	0.05	GC/MSMS	NIST 2974a
10	3.98	0.03[1]	0.01	GC/MSMS	
25	3.90	1.02	0.03	GC/MSMS	
27	3.54	0.13[1]	0.02	GC/ECD	
40*	4.27	0.30	0.01	GC/HRMS	FAPAS 0685, IAEA-459
74*	3.87	0.02[1]	0.003	GC/MSMS	
78*	4.23	0.47[1]	0.30	GC/MSMS	
79	4.40	0.88	0.08	GC/ECD[2]	NIST 2974a, WMF-03

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

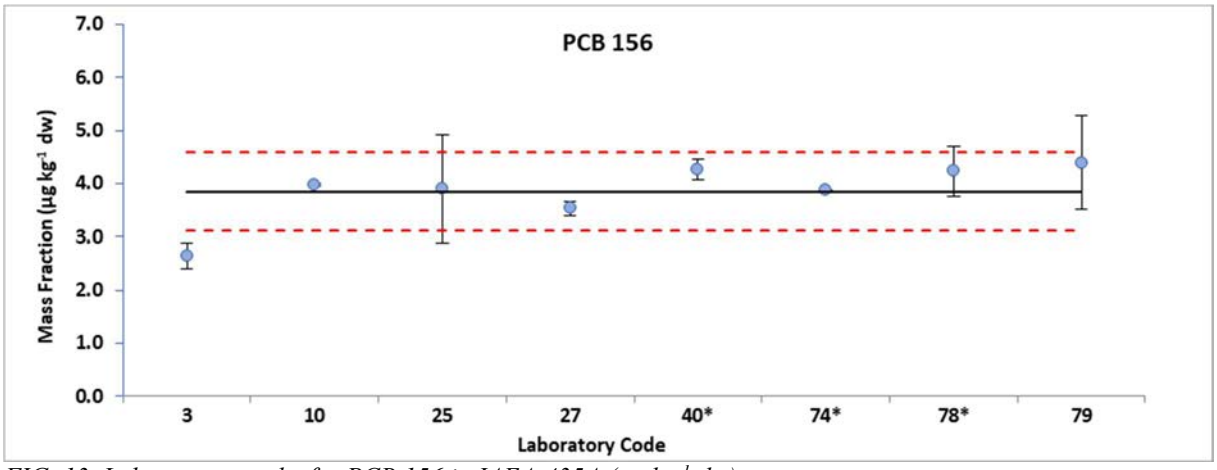

FIG. 13. Laboratory results for PCB 156 in IAEA-435A (µg kg^{-1} dw)

TABLE 25. RESULTS REPORTED BY PARTICIPANTS FOR PCB 170 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
10	12.6	0.1[1]	0.01	GC/MSMS	
25	11.8	2.9	0.03	GC/MSMS	
27	10.5	0.1[1]	0.02	GC/ECD	
40*	9.92	3.50	0.03	GC/HRMS	FAPAS 0685, IAEA-459
78*	12.2	2.2[1]	0.30	GC/MSMS	
79	13.4	2.7	0.01	GC/ECD[2]	NIST 2974a, WMF-03

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

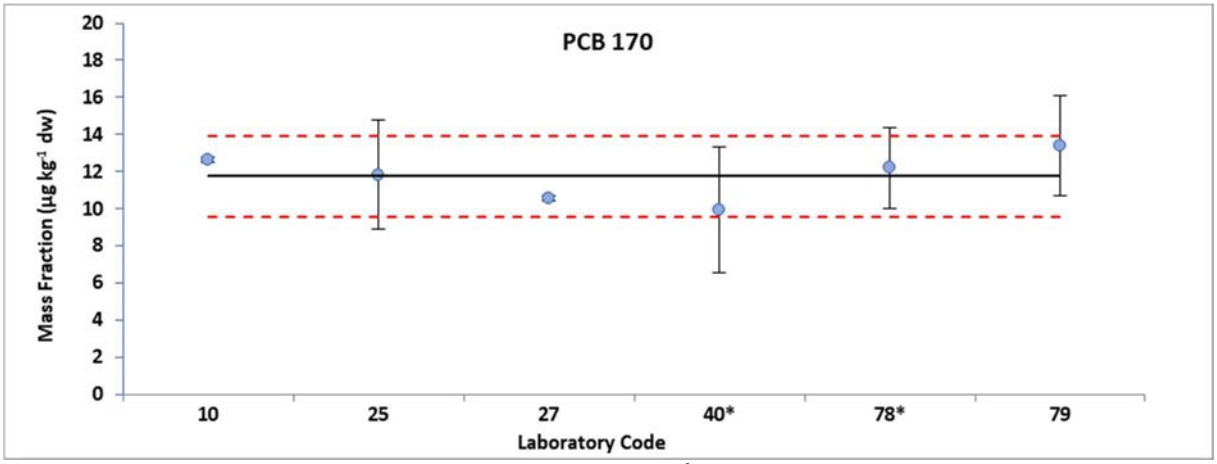

FIG. 14. Laboratory results for PCB 170 in IAEA-435A (µg kg^{-1} dw)

TABLE 26. RESULTS REPORTED BY PARTICIPANTS FOR PCB 180 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
3	23.8	1.4[1]	0.05	GC/MSMS	NIST 2974a
5	36.5	1.4[1]		GC/MSMS	IAEA-435, IAEA-432
7	30.4	4.4	0.40	GC/ECD	IAEA-406
10	41.0	0.9[1]	0.01	GC/MSMS	
25	36.5	4.6	0.03	GC/MSMS	
27	27.1	0.5[1]	0.02	GC/ECD	
40*	38.3	2.4	0.09	GC/HRMS	FAPAS 0685, IAEA-459
49*	31.3	3.2	0.10	GC/MS EI	IAEA-435, IAEA-451, NIST 1946
74*	36.0	0.2[1]	0.01	GC/MSMS	
78*	40.1	4.3[1]	0.30	GC/MSMS	
79	35.0	7.0	0.02	GC/ECD[2]	NIST 2974a, WMF-03

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

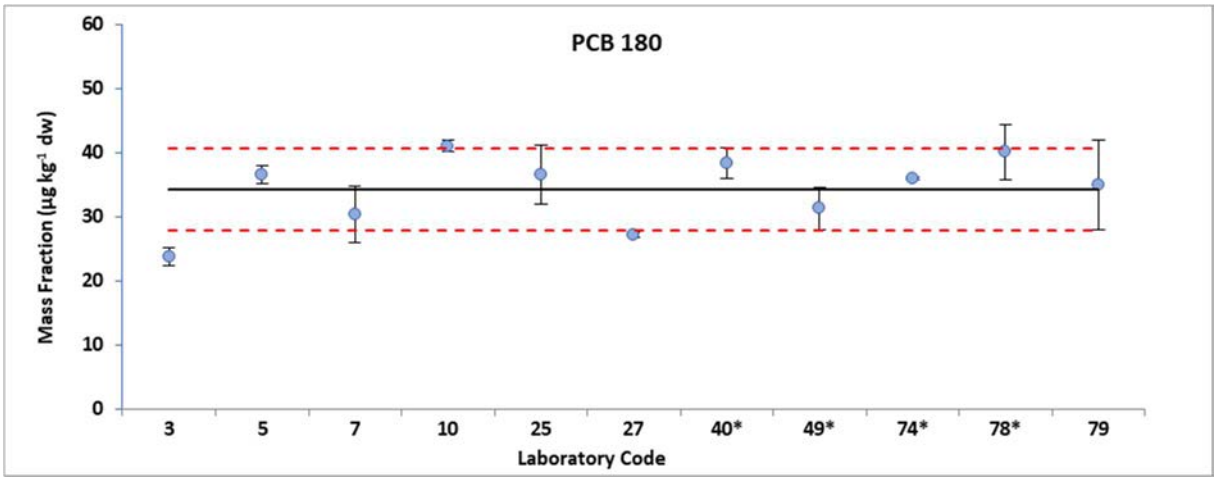

FIG. 15. Laboratory results for PCB 180 in IAEA-435A (µg kg^{-1} dw)

TABLE 27. RESULTS REPORTED BY PARTICIPANTS FOR PCB 187 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
10	31.0	1.0[1]	0.01	GC/MSMS	
25	24.1	1.7	0.03	GC/MSMS	
27	28.7	1.3[1]	0.02	GC/ECD	
40*	45.7	16.0	0.10	GC/HRMS	FAPAS 0685, IAEA-459
79	32.1	6.4	0.02	GC/ECD[2]	NIST 2974a, WMF-03

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

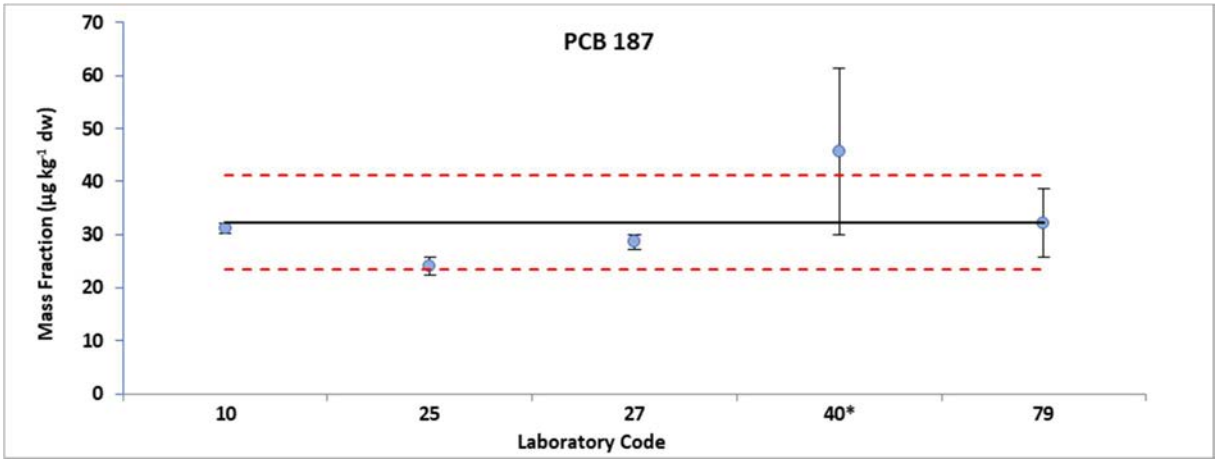

FIG. 16. Laboratory results for PCB 187 in IAEA-435A (µg kg^{-1} dw)

TABLE 28. RESULTS REPORTED BY PARTICIPANTS FOR PCB 194 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
27	3.63	0.69[1]	0.02	GC/ECD	
40*	3.36	1.20	0.03	GC/HRMS	FAPAS 0685, IAEA-459
49*	3.49	0.38	0.10	GC/MS EI	IAEA-435, IAEA-451, NIST 1946
78*	4.37	1.03[1]	0.30	GC/MSMS	
79	4.33	0.87	0.01	GC/ECD[2]	NIST 2974a, WMF-03

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

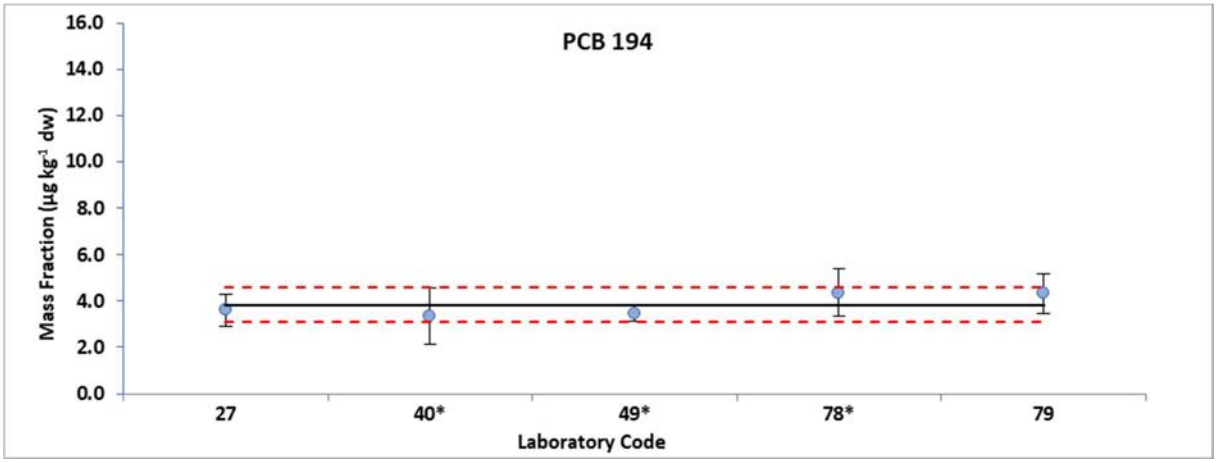

FIG. 17. Laboratory results for PCB 194 in IAEA-435A (µg kg^{-1} dw)

I.2. ASSIGNED VALUES ORGANOCHLORINE PESTICIDES

TABLE 29. RESULTS REPORTED BY PARTICIPANTS FOR HCB (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
3	1.98	0.23[1]	0.05	GC/MSMS	NIST 2974a
10	3.72	0.06[1]		GC/MSMS	ERM-CE100
25	2.59	0.57	0.03	GC/MSMS	
27	2.13	0.22[1]	0.02	GC/ECD	
40*	3.00	0.19	0.32	GC/HRMS	FAPAS 0685, IAEA-459
42	2.08	0.08[1]		GC/MSMS	spiked blank
54	3.48	1.80	1.70	GC/MSMS	
72*	3.30	0.14	0.10	GC/MS EI	NIST1947
78*	3.13	0.35[1]	0.80	GC/MSMS	
79	1.13	0.23	0.49	GC/ECD[2]	NIST 2974a, WMF-03

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

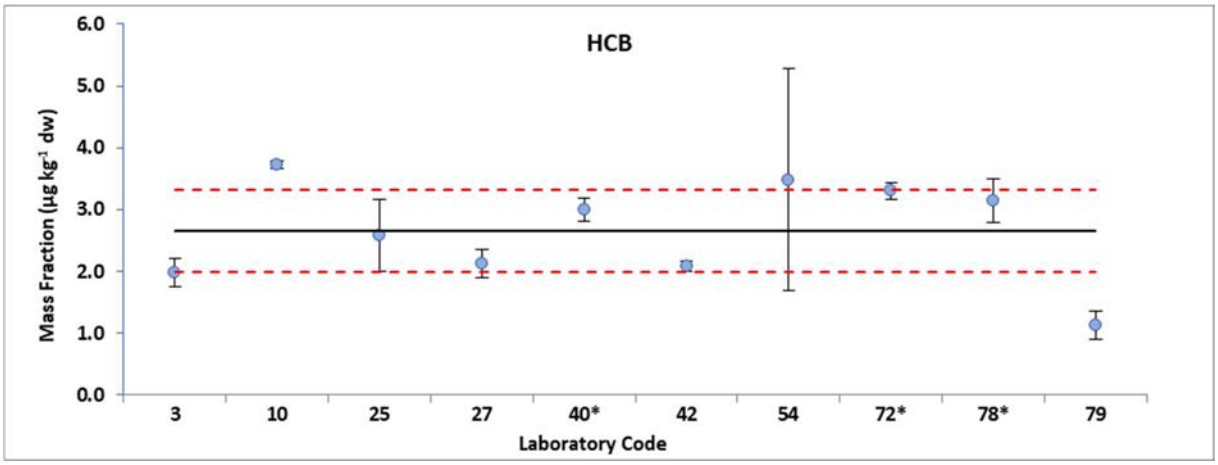

FIG. 18. Laboratory results for HCB in IAEA-435A (µg kg^{-1} dw)

TABLE 30. RESULTS REPORTED BY PARTICIPANTS FOR *pp'*-DDD (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
3	7.03	0.18[1]	0.05	GC/MSMS	NIST 2974a
5	13.4	0.3[1]		GC/MSMS	IAEA-435, IAEA-432
25	17.7	1.6	0.03	GC/MSMS	
27	11.6	0.4[1]	0.02	GC/ECD	
40*	12.2	0.8	0.04	GC/HRMS	FAPAS 0685, IAEA-459
42	11.5	0.5[1]		GC/MSMS	spiked blank
54	21.8	10.9	1.70	GC/MSMS	
72*	12.0	0.5	0.10	GC/MS EI	NIST1947
78*	17.0	1.2[1]	0.30	GC/MSMS	
79	11.6	3.2	0.02	GC/ECD[2]	NIST 2974a, WMF-03

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

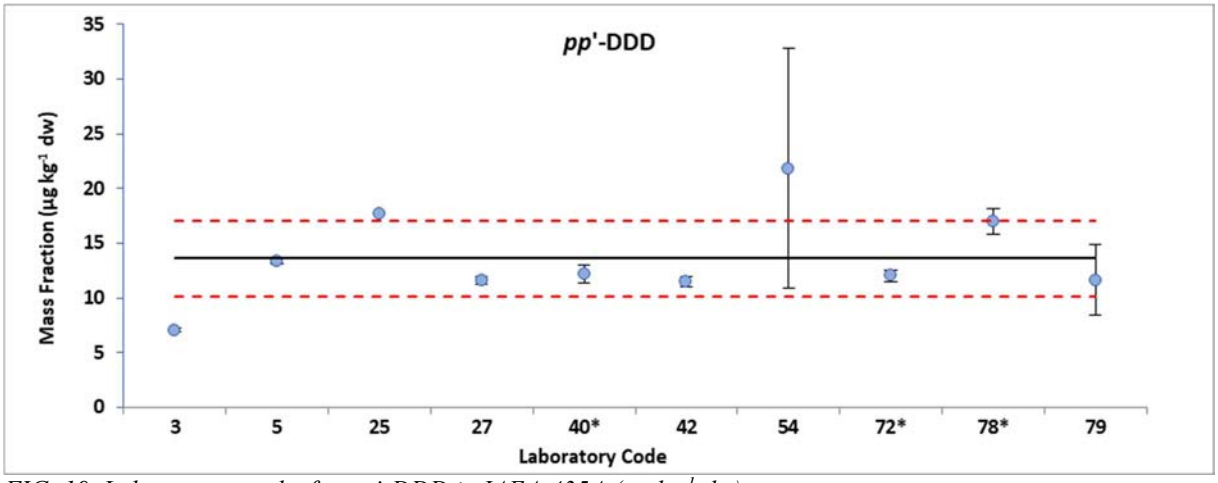

FIG. 19. Laboratory results for pp'-DDD in IAEA-435A (µg kg^{-1} dw)

TABLE 31. RESULTS REPORTED BY PARTICIPANTS FOR *pp'*-DDE (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
3	120	1[1]	0.05	GC/MSMS	NIST 2974a
5	100	4[1]		GC/MSMS	IAEA-435, IAEA-432
25	163	8	0.03	GC/MSMS	
27	101	8[1]	0.02	GC/ECD	
40*	145	9	0.46	GC/HRMS	FAPAS 0685, IAEA-459
42	138	6[1]		GC/MSMS	spiked blank
54	160	80	1.70	GC/MSMS	
72*	158	9	0.10	GC/MS EI	NIST 1947
78*	143	11[1]	0.30	GC/MSMS	
79	117	23	0.05	GC/ECD[2]	NIST 2974a, WMF-03

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

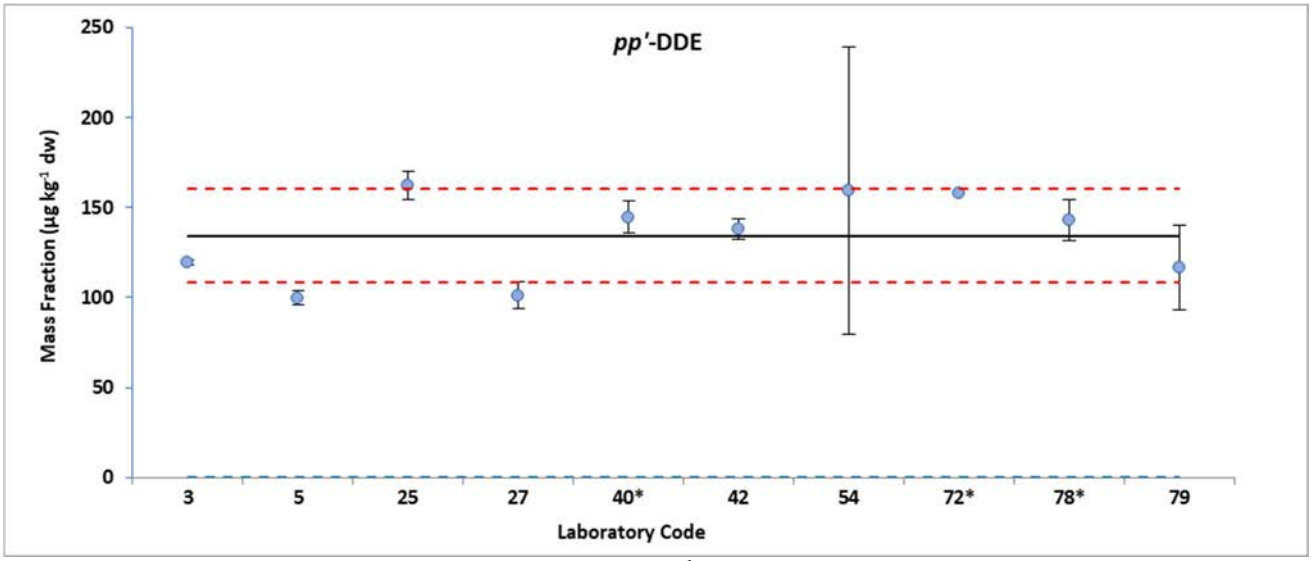

FIG. 20. Laboratory results for pp'-DDE in IAEA-435A (µg kg^{-1} dw)

TABLE 32. RESULTS REPORTED BY PARTICIPANTS FOR *pp'*-DDT (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
3	12.1	1.9[1]	0.05	GC/MSMS	NIST 2974a
5	19.4	0.3[1]		GC/MSMS	IAEA-435, IAEA-432
10	18.2	0.7[1]	0.02	GC/MSMS	
25	23.8	2.9	0.03	GC/MSMS	
27	11.0	1.1[1]	0.02	GC/ECD	
40*	17.5	0.1	0.44	GC/HRMS	FAPAS 0685, IAEA-459
42	24.3	0.9[1]		GC/MSMS	spiked blank
54	21.7	10.9	1.70	GC/MSMS	
72*	17.4	1.1	0.10	GC/MS EI	NIST 1947
78*	21.7	2.9[1]	0.80	GC/MSMS	
79	14.4	2.9	0.05	GC/ECD[2]	NIST 2974a, WMF-03

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

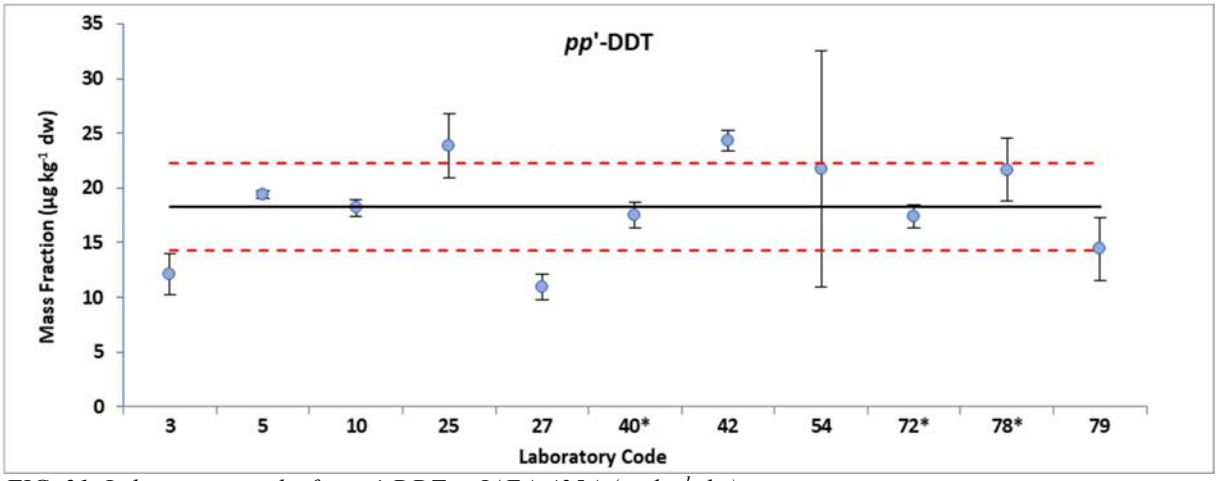

FIG. 21. Laboratory results for pp'-DDT in IAEA-435A (µg kg^{-1} dw)

TABLE 33. RESULTS REPORTED BY PARTICIPANTS FOR *op'* -DDE (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
25	2.19	0.37	0.03	GC/MSMS	
40*	1.96	0.12	0.04	GC/HRMS	FAPAS 0685, IAEA-459
42	1.30	0.07[1]		GC/MSMS	spiked blank
54	2.32	1.20	1.70	GC/MSMS	
72*	2.94	0.11	0.10	GC/MS EI	NIST 1947
78*	1.83	0.24[1]	0.30	GC/MSMS	
Results not used for the assignment of the value					
79	5.76	1.15	0.01	GC/ECD[2]	NIST 2974a, WMF-03

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

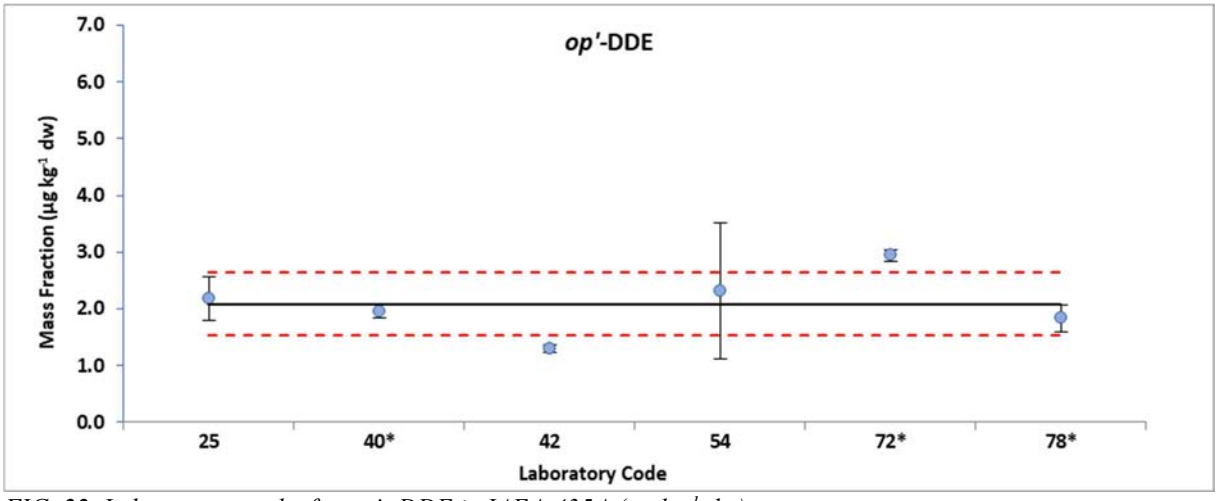

FIG. 22. Laboratory results for op' -DDE in IAEA-435A (µg kg^{-1} dw)

TABLE 34. RESULTS REPORTED BY PARTICIPANTS FOR *op'*-DDD (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
25	3.34	0.67	0.03	GC/MSMS	
40*	2.14	0.80	0.03	GC/HRMS	FAPAS 0685, IAEA-459
42	1.53	0.07[1]		GC/MSMS	spiked blank
54	3.65	1.90	1.70	GC/MSMS	
72*	3.00	0.14	0.10	GC/MS EI	NIST 1947
78*	2.77	0.13[1]	0.30	GC/MSMS	
79	2.29	0.46	0.01	GC/ECD[2]	NIST 2974a, WMF-03

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

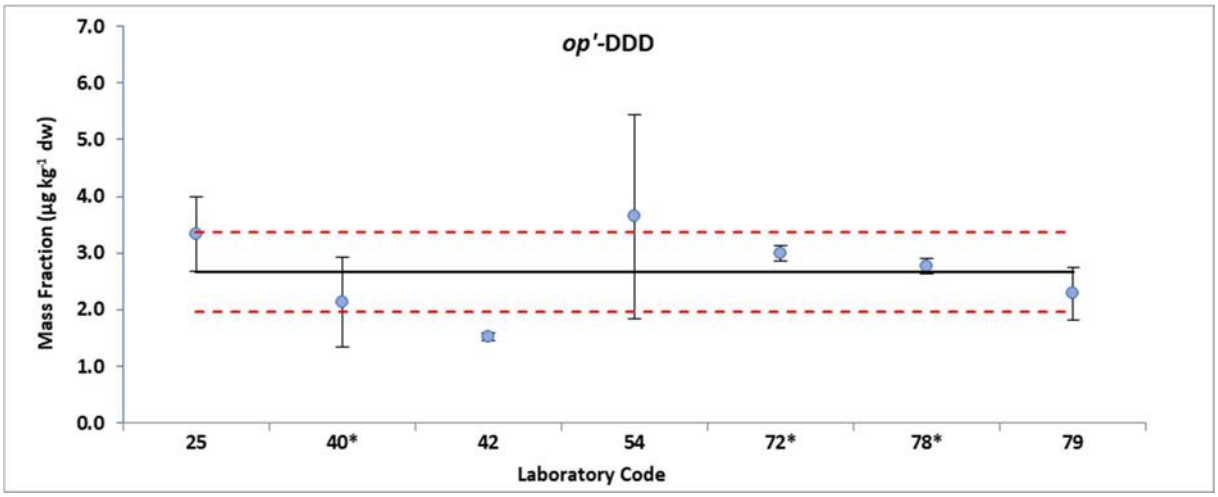

FIG. 23. Laboratory results for op'-DDD in IAEA-435A (µg kg^{-1} dw)

TABLE 35. RESULTS REPORTED BY PARTICIPANTS FOR *op*'-DDT (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
10	6.97	0.24[1]	0.02	GC/MSMS	
25	7.05	0.91	0.03	GC/MSMS	
27	4.02	0.14[1]	0.02	GC/ECD	
40*	6.55	0.40	0.22	GC/HRMS	FAPAS 0685, IAEA-459
42	8.30	0.17[1]		GC/MSMS	spiked blank
54	10.2	5.1	1.70	GC/MSMS	
72*	9.02	0.24	0.10	GC/MS EI	NIST 1947
78*	8.03	0.93[1]	0.80	GC/MSMS	
79	4.23	0.85	0.02	GC/ECD[2]	NIST 2974a, WMF-03

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

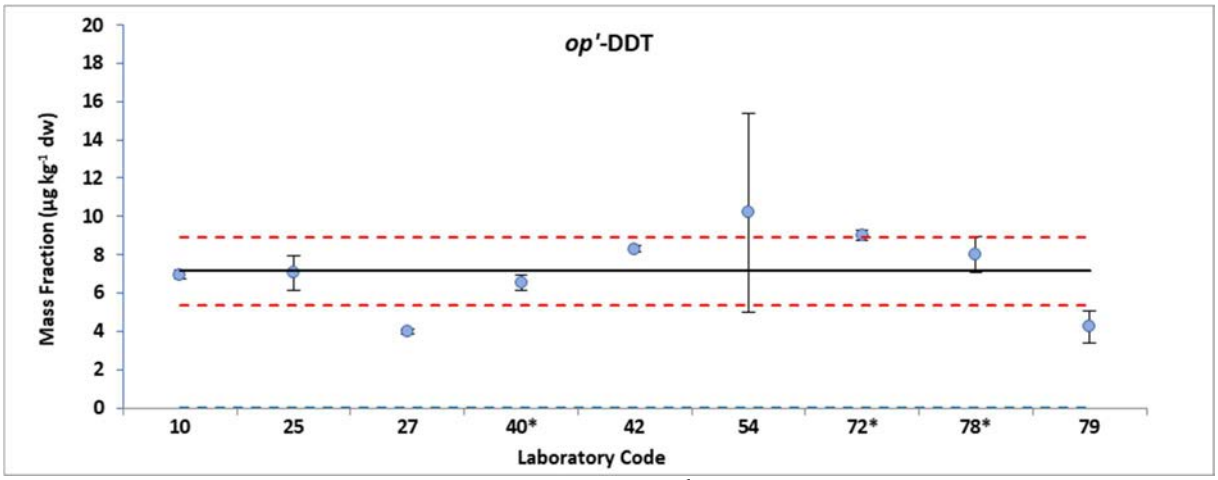

FIG. 24. Laboratory results for op'-DDT in IAEA-435A (µg kg^{-1} dw)

TABLE 36. RESULTS REPORTED BY PARTICIPANTS FOR DIELDRIN (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
3	4.78	0.70[1]	0.05	GC/MSMS	NIST 2974a
25	6.93	1.65	0.03	GC/MSMS	
40*	6.92	0.50	0.09	GC/HRMS	FAPAS 0685, IAEA-459
72*	5.38	0.40	0.10	GC/MS EI	NIST 1947
79	3.28	0.66	0.05	GC/ECD[2]	NIST 2974a, WMF-03
Results not used for the assignment of the value					
54	<3.30		3.30	GC/MSMS	
78*	<12.5		4.0	GC/MSMS	

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a -- $X_a \pm U_a$ (k=2)

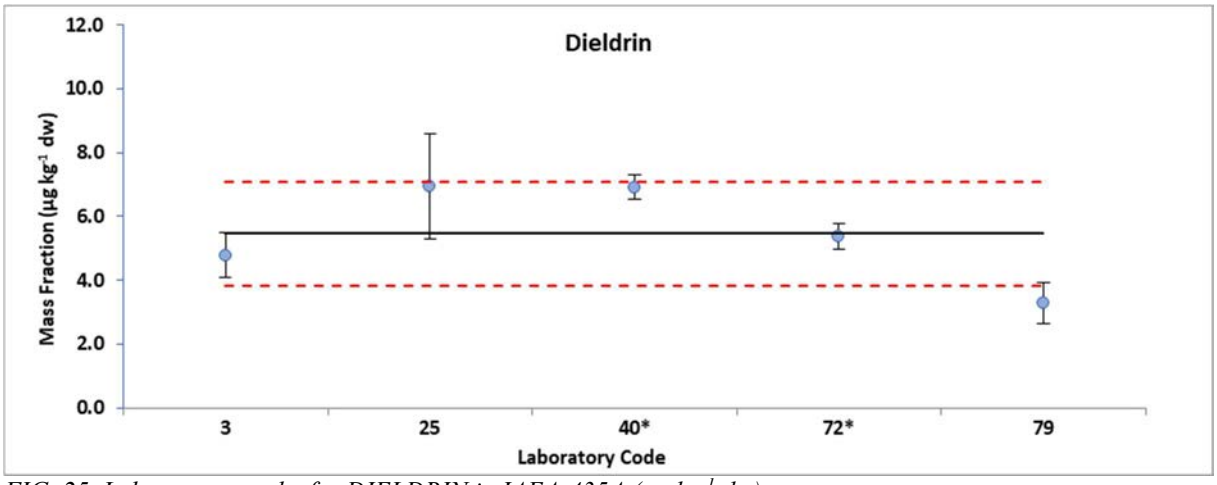

FIG. 25. Laboratory results for DIELDRIN in IAEA-435A (µg kg^{-1} dw)

TABLE 37. RESULTS REPORTED BY PARTICIPANTS FOR *cis*-CHLORDANE (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
27	5.24	0.77[1]	0.02	GC/ECD	
40*	7.46	0.50	0.01	GC/HRMS	FAPAS 0685, IAEA-459
42	3.92	0.38[1]		GC/MSMS	spiked blank
54	10.6	5.3	3.30	GC/MSMS	
72*	8.38	0.42	0.10	GC/MS EI	NIST 1947
78*	8.40	0.40[1]	0.30	GC/MSMS	
79	5.42	1.08	0.02	GC/ECD[2]	NIST 2974a, WMF-03

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

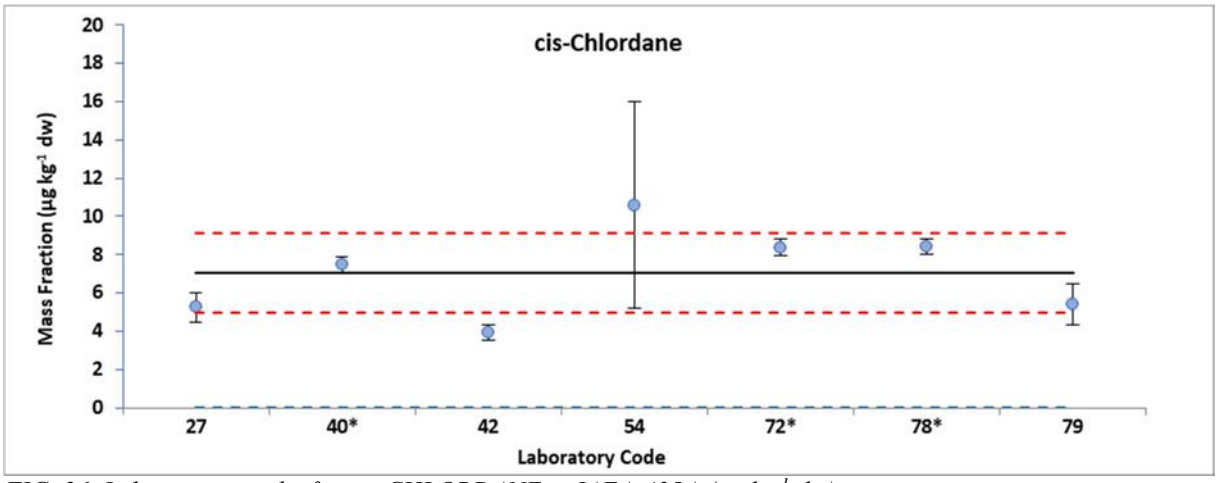

FIG. 26. Laboratory results for cis-CHLORDANE in IAEA-435A (µg kg^{-1} dw)

TABLE 38. RESULTS REPORTED BY PARTICIPANTS FOR *trans*-NONACHLOR (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
40*	22.1	1.4	0.04	GC/HRMS	FAPAS 0685, IAEA-459
42	14.6	0.5[1]		GC/MSMS	spiked blank
54	20.2	10.1	3.30	GC/MSMS	
72*	24.2	0.9	0.10	GC/MS EI	NIST 1947
79	20.0	4.0	0.01	GC/ECD[2]	NIST 2974a, WMF-03

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

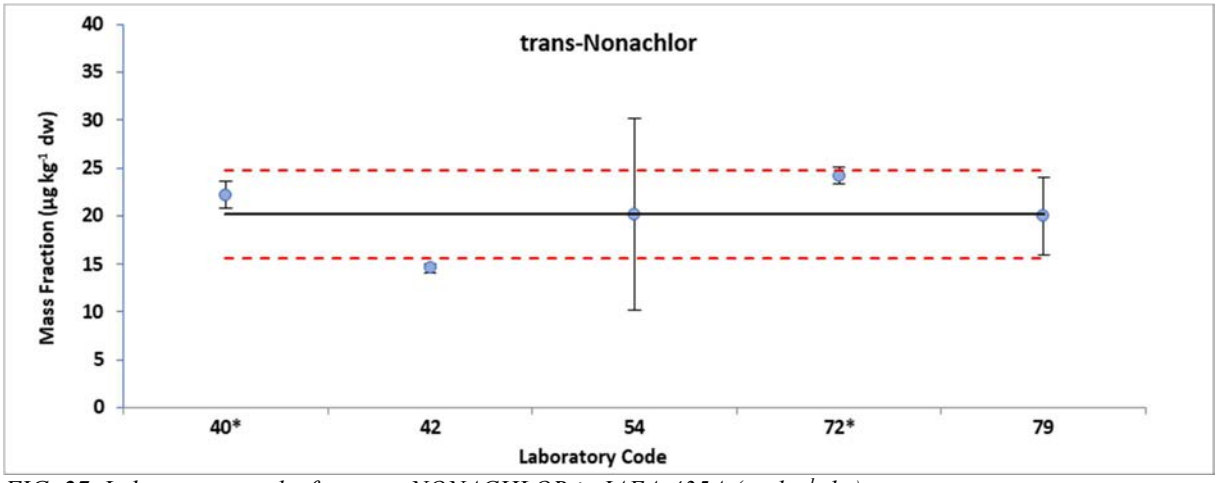

FIG. 27. Laboratory results for trans-NONACHLOR in IAEA-435A (µg kg^{-1} dw)

I.3. ASSIGNED VAULUES FOR PBDES

TABLE 39. RESULTS REPORTED BY PARTICIPANTS FOR BDE 28 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
10	1.49	0.09[1]	0.03	GC/MS EI	
14	1.45	0.64	0.001	GC/MSMS	WMF-02 Wellington
27	1.28	0.61[1]	0.01	GC/MSMS	
38	1.37	0.29	0.01	GC/MS NCI	In House Native Spike Solution
40*	1.34	0.12	0.01	GC/HRMS	IC2019SE
74*	1.26	0.01[1]	0.001	GC/MS EI	
78*	1.10	0.12[1]	0.30	GC/MSMS	
79	0.79	0.12	0.01	GC/MSMS	NIST 2974a, WMF-03

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

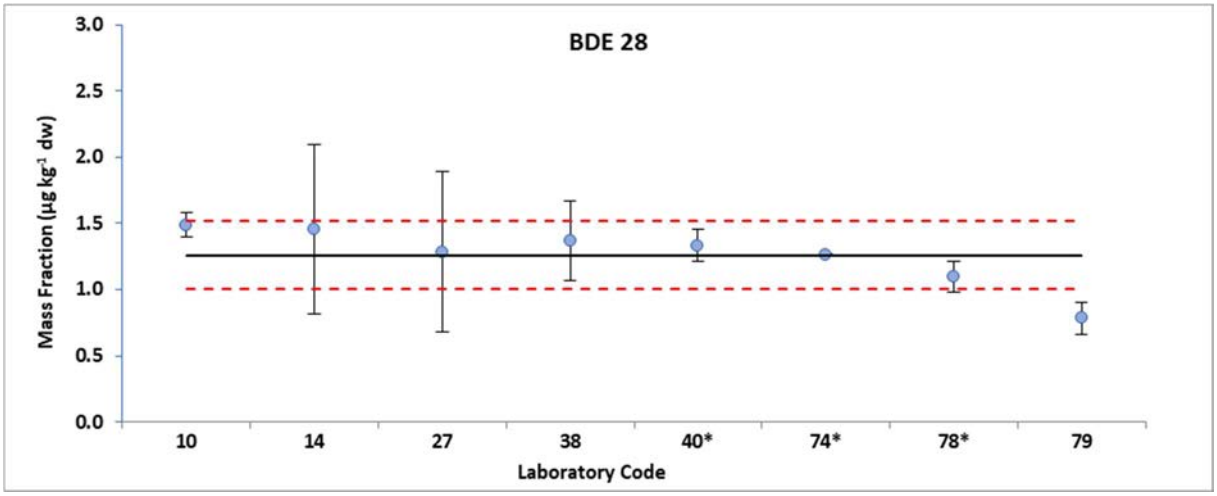

FIG. 28. Laboratory results for BDE 28 in IAEA-435A (µg kg^{-1} dw)

TABLE 40. RESULTS REPORTED BY PARTICIPANTS FOR BDE 47 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
10	25.5	1.6[1]	0.03	GC/MSMS	
14	23.4	10.3	0.001	GC/HRMS	WMF-02 Wellington
27	22.9	3.7[1]	0.01	GC/MSMS	
38	24.9	3.6	0.09	GC/HRMS	In House Native Spike Solution
40*	21.3	1.6	0.01	GC/HRMS	IC2019SE
74*	21.5	0.1[1]	0.01	GC/MSMS	
78*	20.9	3.4[1]	0.80	GC/MSMS	
79	23.0	4.6	0.15	GC/ECD[2]	NIST 2974a, WMF-03

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

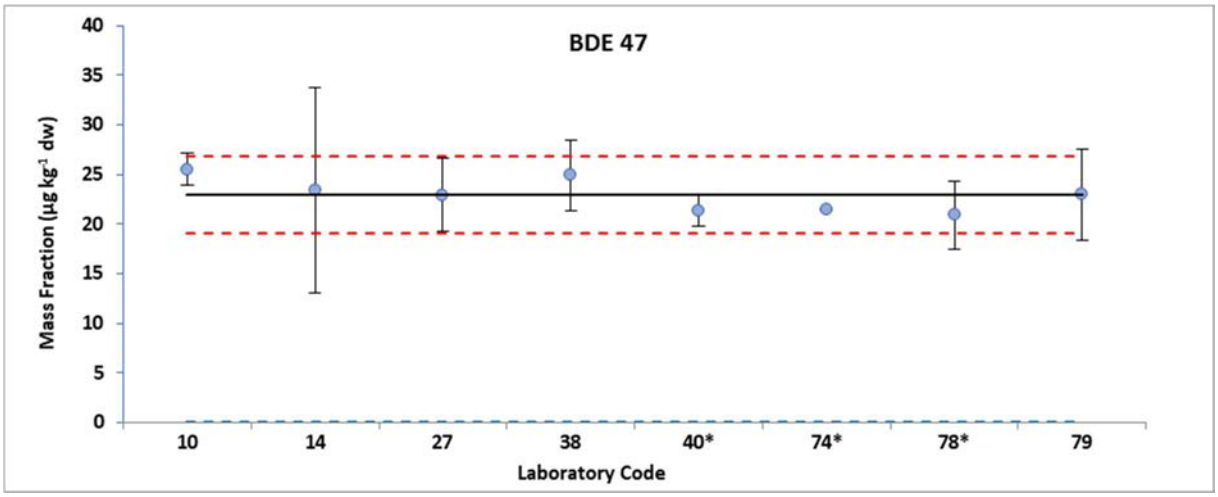

FIG. 29. Laboratory results for BDE 47 in IAEA-435A (µg kg^{-1} dw)

TABLE 41. RESULTS REPORTED BY PARTICIPANTS FOR BDE 66 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
27	2.73	0.41[1]	0.01	GC/MSMS	
38	2.81	0.82	0.005	GC/HRMS	In House Native Spike Solution
40*	2.22	0.77	0.02	GC/HRMS	IC2019SE
74*	2.02	0.06[1]	0.003	GC/MSMS	
79	2.63	0.52	0.05	GC/ECD[2]	NIST 2974a, WMF-03

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

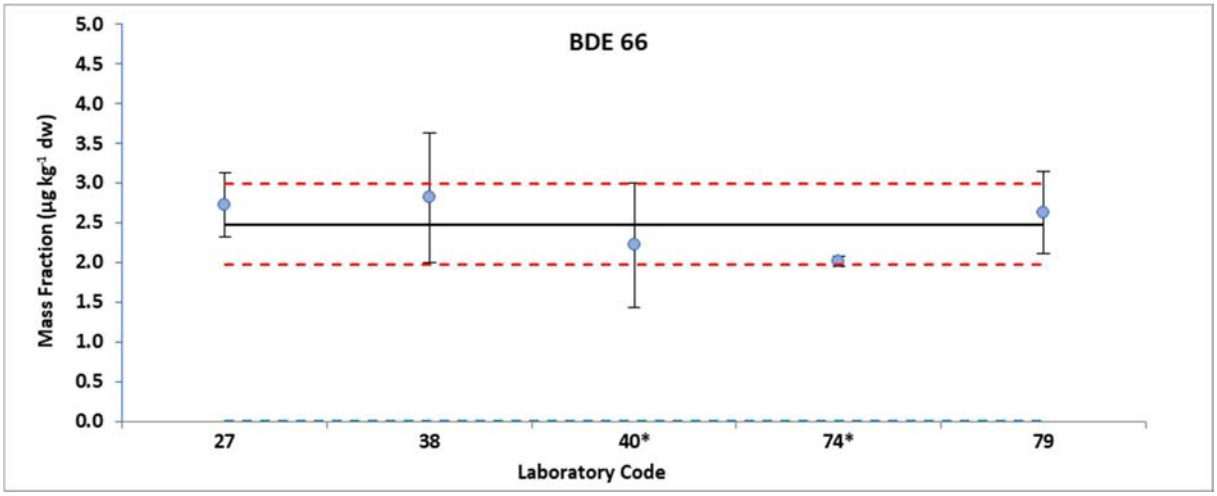

FIG. 30. Laboratory results for BDE 66 in IAEA-435A (µg kg^{-1} dw)

TABLE 42. RESULTS REPORTED BY PARTICIPANTS FOR BDE 99 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
10	1.28	0.05[1]	0.03	GC/MSMS	
14	1.37	0.60	0.001	GC/HRMS	WMF-02 Wellington
27	1.47	0.15[1]	0.01	GC/MSMS	
38	1.90	4.82	0.14	GC/HRMS	In House Native Spike Solution
40*	1.19	0.08	0.01	GC/HRMS	IC2019SE
74*	1.34	0.02[1]	0.01	GC/MSMS	
79	1.55	0.31	0.19	GC/ECD[2]	NIST 2974a, WMF-03
Results not used for the assignment of the value					
78*[1]	<2.50		0.80	GC/MSMS	

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

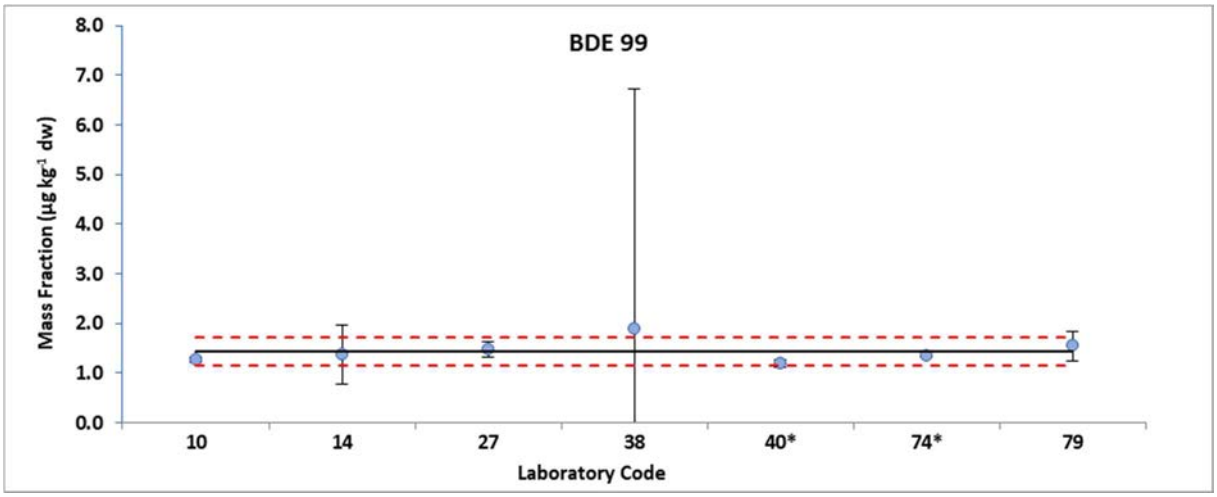

FIG. 31. Laboratory results for BDE 85 in IAEA-435A (µg kg^{-1} dw)

TABLE 43. RESULTS REPORTED BY PARTICIPANTS FOR BDE 100 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
10	8.29	0.49[1]	0.03	GC/MSMS	
14	7.72	3.40	0.001	GC/HRMS	WMF-02 Wellington
27	7.87	1.39[1]	0.01	GC/MSMS	
38	7.49	1.18	0.03	GC/HRMS	In House Native Spike Solution
40*	7.14	0.46	0.01	GC/HRMS	IC2019SE
74*	7.31	0.09[1]	0.01	GC/MSMS	
78*	5.70	0.61[1]	0.30	GC/MSMS	
79	6.78	1.36	0.08	GC/ECD[2]	NIST 2974a, WMF-03

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

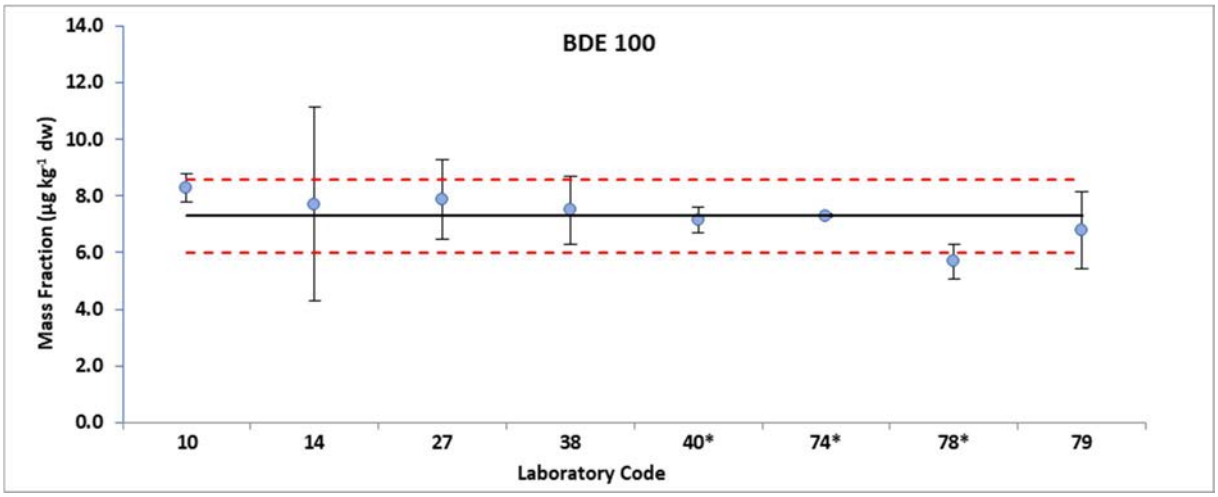

FIG. 32. Laboratory results for BDE 100 in IAEA-435A (µg kg^{-1} dw)

TABLE 44. RESULTS REPORTED BY PARTICIPANTS FOR BDE 153 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
10	0.69	0.06[1]	0.03	GC/MSMS	
14	0.77	0.34	0.001	GC/HRMS	WMF-02 Wellington
27	0.97	0.11[1]	0.01	GC/MSMS	
38	0.63	0.83	0.02	GC/HRMS	In House Native Spike Solution
40*	0.66	0.05	0.02	GC/HRMS	IC2019SE
74*	0.68	0.01[1]	0.01	GC/MSMS	
79	0.49	0.10	0.02	GC/ECD[2]	NIST 2974a, WMF-03
Results not used for the assignment of the value					
78*	<5.00		2.00	GC/MSMS	

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

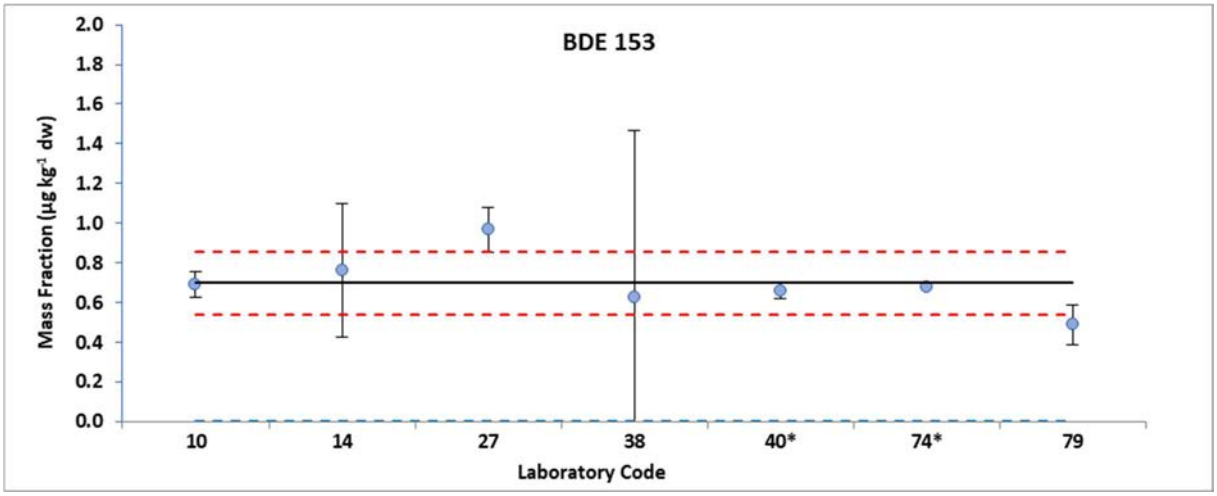

FIG. 33. Laboratory results for BDE 153 in IAEA-435A (µg kg^{-1} dw)

TABLE 45. RESULTS REPORTED BY PARTICIPANTS FOR BDE 154 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
10	5.24	0.47[1]	0.03	GC/MSMS	
14	5.84	2.56	0.001	GC/HRMS	WMF-02 Wellington
27	7.01	0.67[1]	0.01	GC/MSMS	
38	5.26	0.63	0.01	GC/HRMS	In House Native Spike Solution
40*	4.18	0.27	0.01	GC/HRMS	IC2019SE
74*	4.88	0.31[1]	0.01	GC/MSMS	
78*	3.73	1.62[1]	0.30	GC/MSMS	
79	4.14	0.83	0.01	GC/ECD[2]	NIST 2974a, WMF-03

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

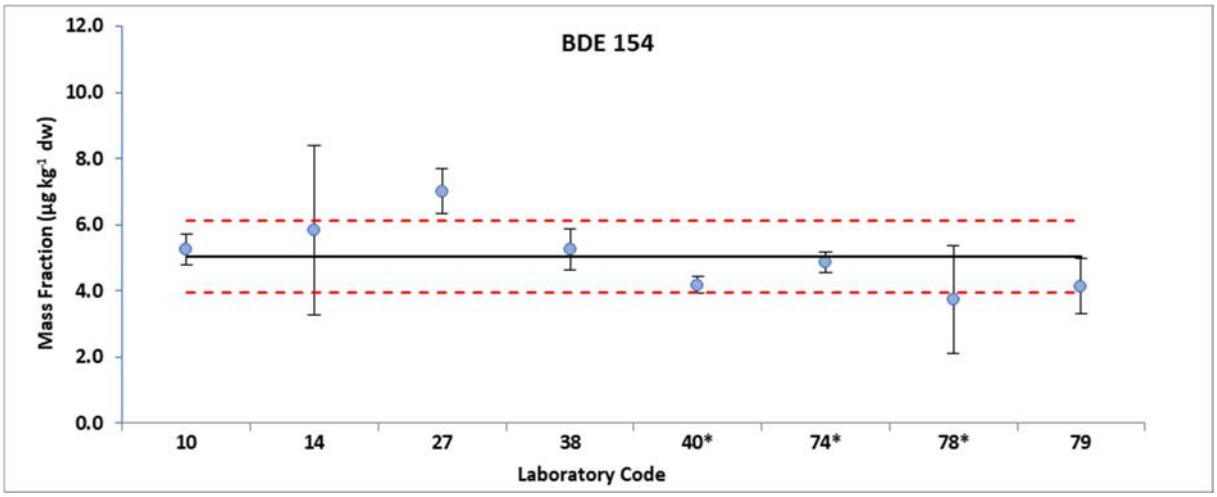

FIG. 34. Laboratory results for BDE 154 in IAEA-435A (µg kg^{-1} dw)

APPENDIX II: INFORMATION VALUES

II.1. INFORMATION VALUES FOR PCB CONGENERS

TABLE 46. RESULTS REPORTED BY PARTICIPANTS FOR PCB 8 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
40*	<0.30		0.10	GC/HRMS	FAPAS 0685, IAEA-459
79	<0.33		0.10	GC/ECD[1]	NIST 2974a, WMF-03

* Laboratory accredited
[1]GC/ECD and peak confirmation with GC/MSMS

TABLE 47. RESULTS REPORTED BY PARTICIPANTS FOR PCB 18 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
40*	0.36	0.13	0.14	GC/HRMS	FAPAS 0685, IAEA-459
79	0.68	0.14	0.21	GC/ECD[1]	NIST 2974a, WMF-03
Results not used for the assignment of the value					
78*	<1.00		0.30	GC/MSMS	

* Laboratory accredited
[1] GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a -- $X_a \pm U_a$ (k=2)

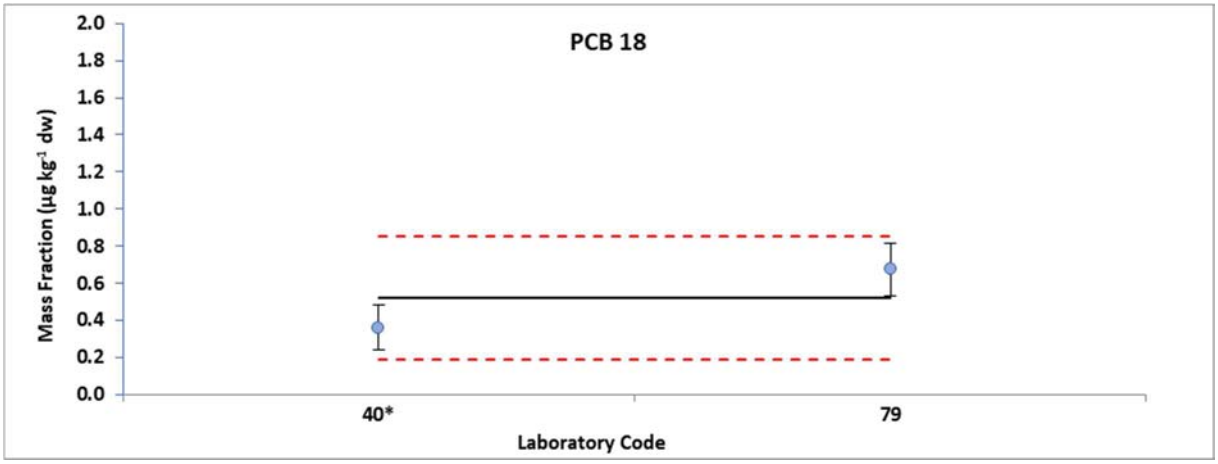

FIG. 35. Laboratory results for PCB 18 in IAEA-435A (µg kg^{-1} dw)

TABLE 48. RESULTS REPORTED BY PARTICIPANTS FOR PCB 44 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
40*	1.39	0.48	0.30	GC/HRMS	FAPAS 0685, IAEA-459
78*	1.80	0.60[1]	0.30	GC/MSMS	
79	1.99	0.40	0.04	GC/ECD[2]	NIST 2974a, WMF-03

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

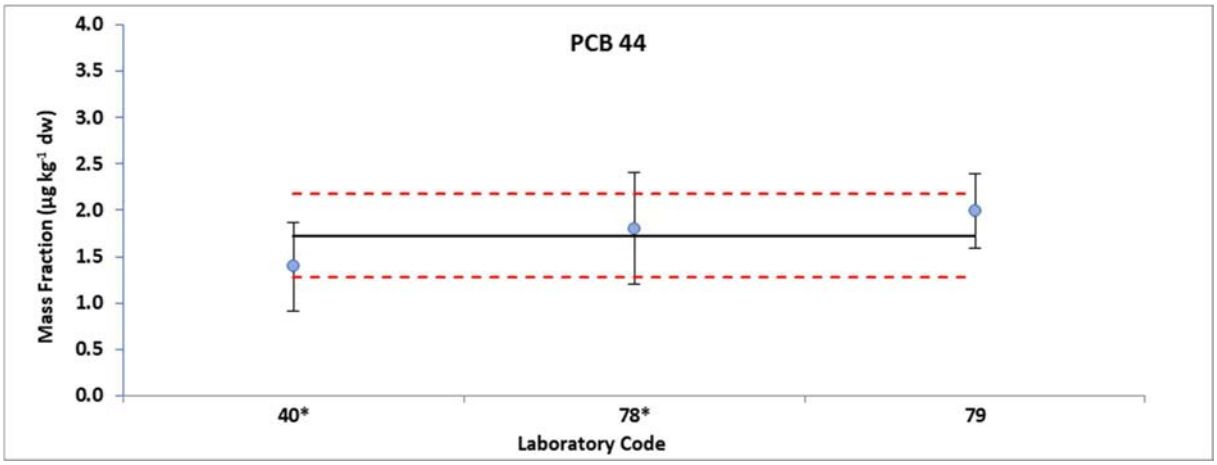

FIG. 36. Laboratory results for PCB 44 in IAEA-435A (µg kg^{-1} dw)

TABLE 49. RESULTS REPORTED BY PARTICIPANTS FOR PCB 49 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
40*	2.19	0.78	0.39	GC/HRMS	FAPAS 0685, IAEA-459
79	2.11	0.42	0.05	GC/ECD[1]	NIST 2974a, WMF-03

* Laboratory accredited

[1] GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

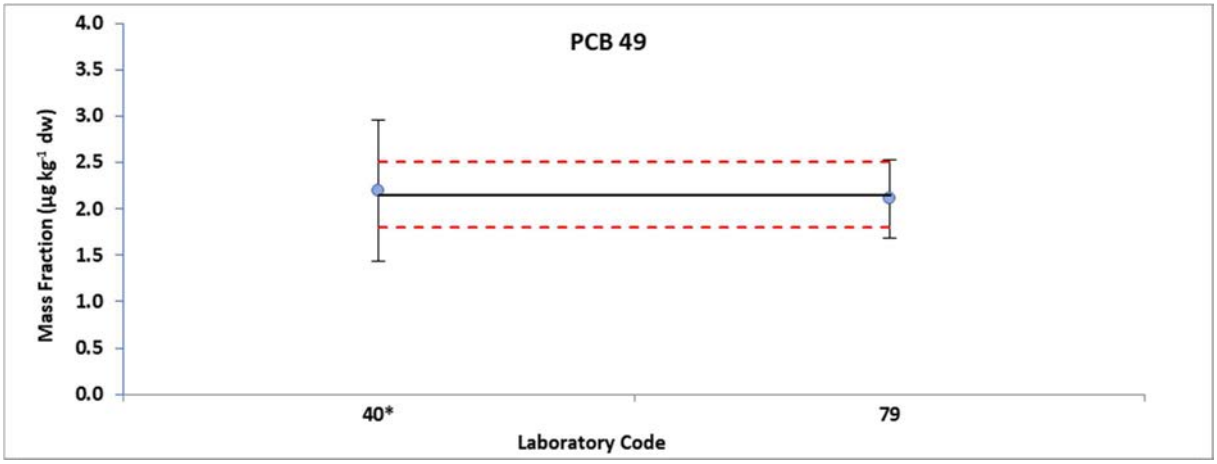

FIG. 37. Laboratory results for PCB 49 in IAEA-435A (µg kg^{-1} dw)

TABLE 50. RESULTS REPORTED BY PARTICIPANTS FOR PCB 66 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
40*	6.70	2.30	0.33	GC/HRMS	FAPAS 0685, IAEA-459
79	10.5	2.1	0.03	GC/ECD[1]	NIST 2974a, WMF-03

* Laboratory accredited
[1]GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

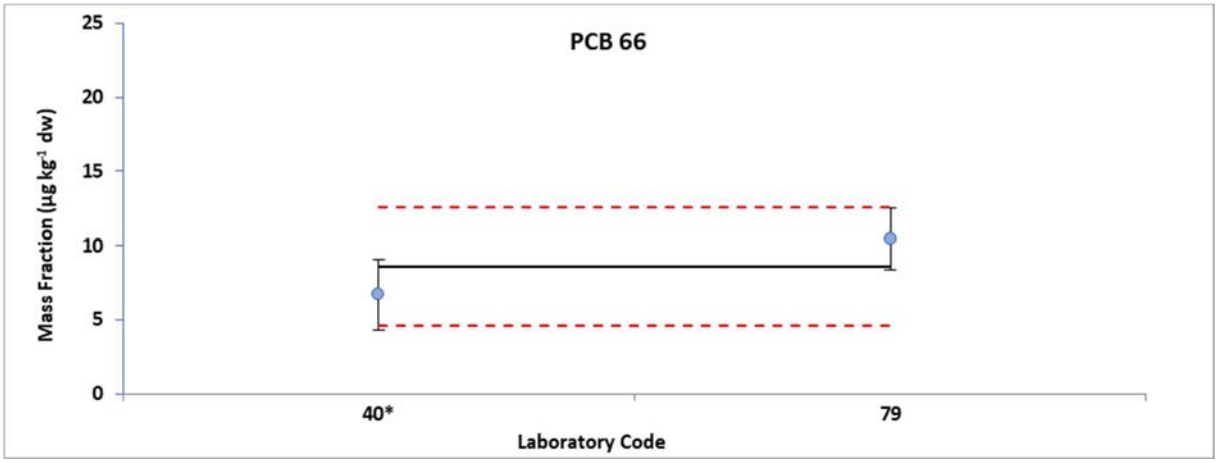

FIG. 38. Laboratory results for PCB 66 in IAEA-435A (µg kg^{-1} dw)

TABLE 51. RESULTS REPORTED BY PARTICIPANTS FOR PCB 70 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
40*	5.70	2.00	0.57	GC/HRMS	FAPAS 0685, IAEA-459

* Laboratory accredited

TABLE 52. RESULTS REPORTED BY PARTICIPANTS FOR PCB 74 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
40*	4.35	1.53	0.34	GC/HRMS	FAPAS 0685, IAEA-459

* Laboratory accredited

TABLE 53. RESULTS REPORTED BY PARTICIPANTS FOR PCB 87 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
40*	6.72	2.40	0.15	GC/HRMS	FAPAS 0685, IAEA-459
79	6.41	1.28	0.01	GC/ECD[1]	NIST 2974a, WMF-03

* Laboratory accredited
[1]GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a -- $X_a \pm U_a$ (k=2)

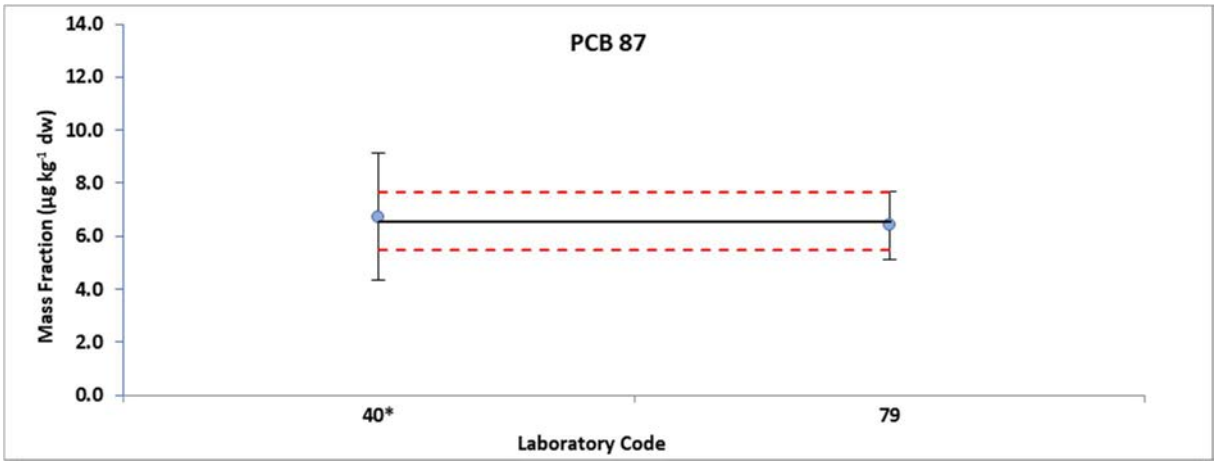

FIG. 39. Laboratory results for PCB 87 in IAEA-435A (µg kg^{-1} dw)

TABLE 54. RESULTS REPORTED BY PARTICIPANTS FOR PCB 95 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
10	8.98	0.16[1]	0.01	GC/MSMS	
25	4.68	0.21	0.03	GC/MSMS	
40*	7.41	2.60	0.40	GC/HRMS	FAPAS 0685, IAEA-459

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

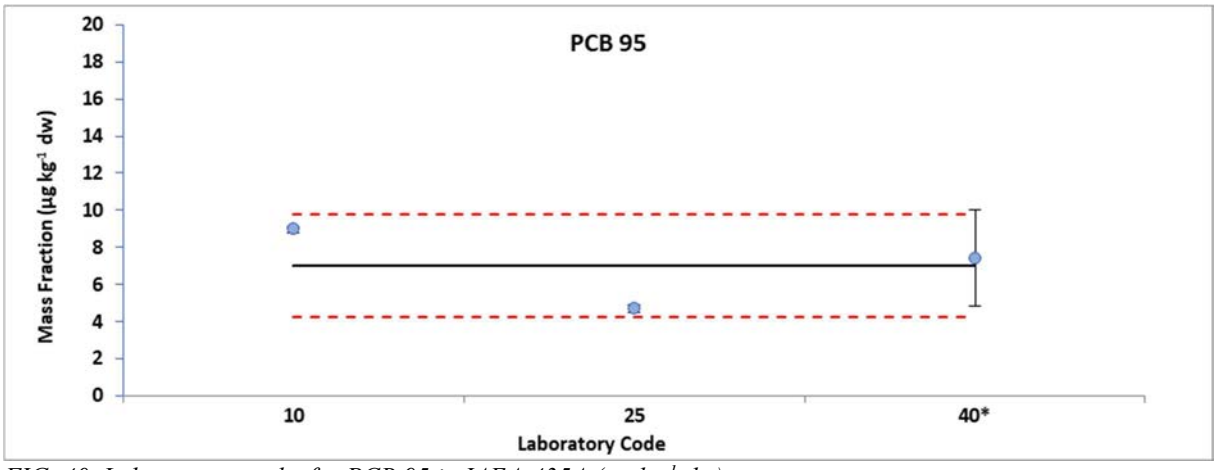

FIG. 40. Laboratory results for PCB 95 in IAEA-435A (µg kg^{-1} dw)

TABLE 55. RESULTS REPORTED BY PARTICIPANTS FOR PCB 97 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
40*	5.73	2.00	0.10	GC/HRMS	FAPAS 0685, IAEA-459
79	4.95	0.99	0.01	GC/ECD[1]	NIST 2974a, WMF-03

* Laboratory accredited
[1]GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a -- $X_a \pm U_a$ (k=2)

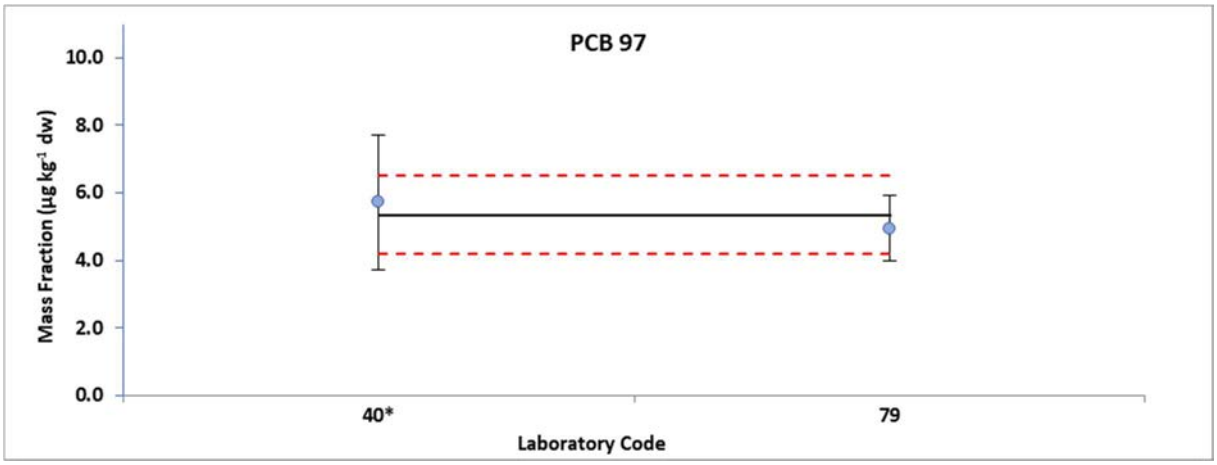

FIG. 41. Laboratory results for PCB 97 in IAEA-435A (µg kg^{-1} dw)

TABLE 56. RESULTS REPORTED BY PARTICIPANTS FOR PCB 99 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
10	20.5	0.2[1]	0.01	GC/MSMS	
25	10.7	0.3	0.03	GC/MSMS	
40*	19.5	6.9	0.42	GC/HRMS	FAPAS 0685, IAEA-459
79	24.5	5.3	0.03	GC/ECD[2]	NIST 2974a, WMF-03

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

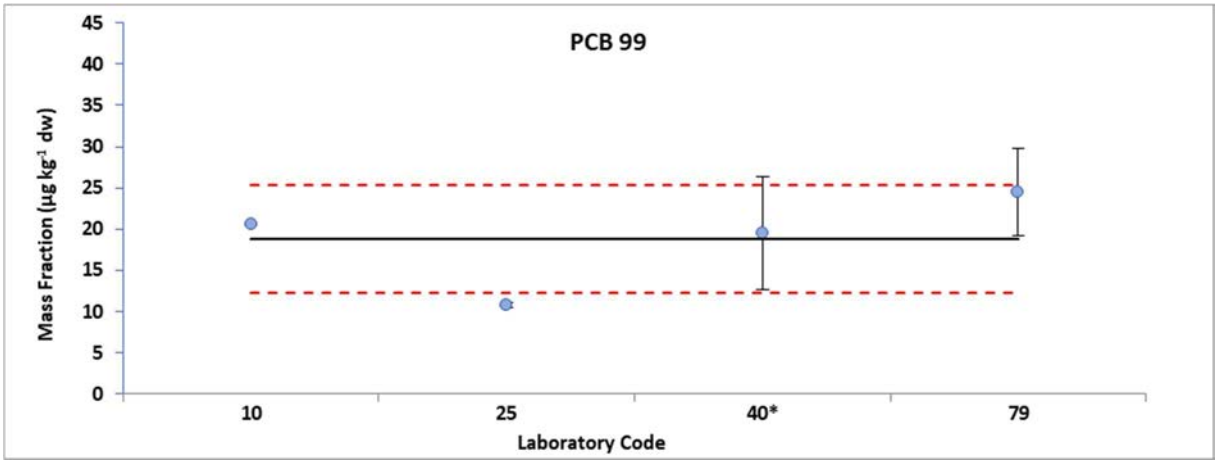

FIG. 42. Laboratory results for PCB 99 in IAEA-435A (µg kg^{-1} dw)

TABLE 57. RESULTS REPORTED BY PARTICIPANTS FOR PCB 110 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
10	15.4	0.5[1]	0.01	GC/MSMS	
79	14.8	3.0	0.12	GC/ECD[2]	NIST 2974a, WMF-03
Results not used for the assignment of the value					
25	9.41	0.55	0.03	GC/MSMS	
40*	20.4	7.1	0.42	GC/HRMS	FAPAS 0685, IAEA-459

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

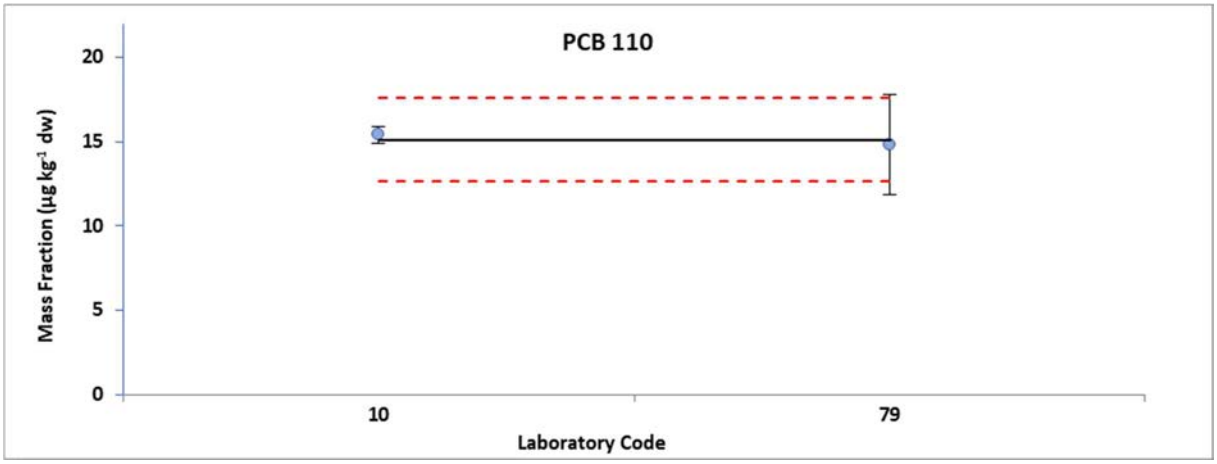

FIG. 43. Laboratory results for PCB 110 in IAEA-435A (µg kg^{-1} dw)

TABLE 58. RESULTS REPORTED BY PARTICIPANTS FOR PCB 128 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
25	8.84	0.29	0.03	GC/MSMS	
40*	8.88	3.10	0.10	GC/HRMS	FAPAS 0685, IAEA-459
79	13.8	2.8	0.02	GC/ECD[1]	NIST 2974a, WMF-03

* Laboratory accredited
[1]GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a -- $X_a \pm U_a$ (k=2)

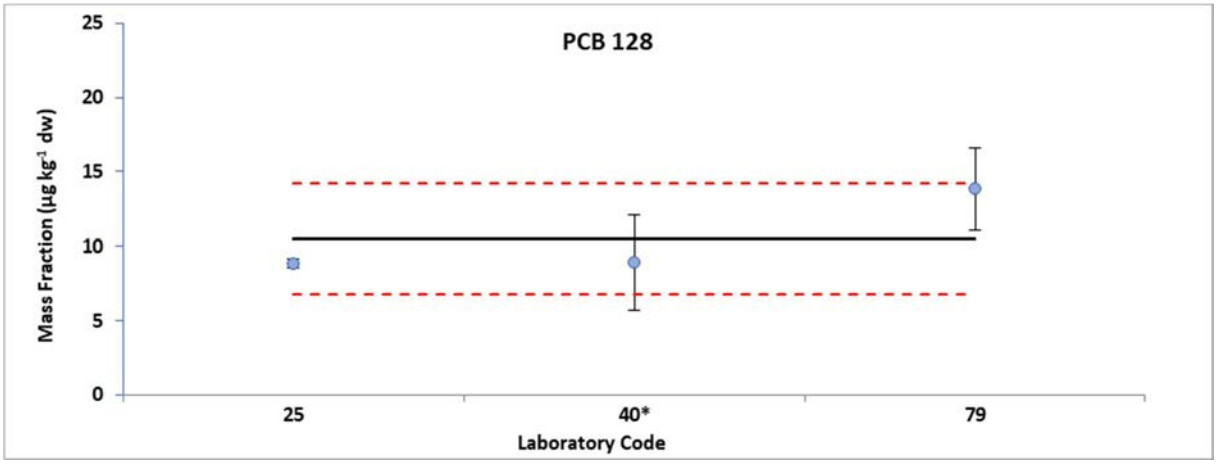

FIG. 44. Laboratory results for PCB 128 in IAEA-435A (µg kg^{-1} dw)

TABLE 59. RESULTS REPORTED BY PARTICIPANTS FOR PCB 146 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
10	19.0	0.6[1]	0.01	GC/MSMS	
40*	19.0	3.3	0.04	GC/HRMS	FAPAS 0685, IAEA-459
Results not used for the assignment of the value					
25	86.7	10.2	0.03	GC/MSMS	

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

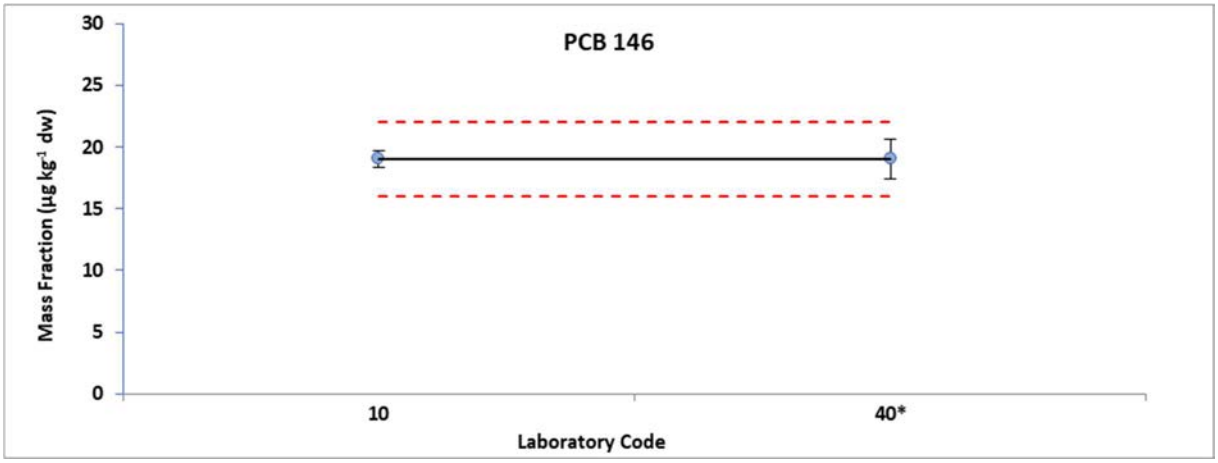

FIG. 45. Laboratory results for PCB 146 in IAEA-435A (µg kg^{-1} dw)

TABLE 60. RESULTS REPORTED BY PARTICIPANTS FOR PCB 151 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
10	9.78	0.23[1]	0.01	GC/MSMS	
25	7.54	1.07	0.03	GC/MSMS	
40*	10.6	3.8	0.03	GC/HRMS	FAPAS 0685, IAEA-459
79	9.52	1.90	0.01	GC/ECD[2]	NIST 2974a, WMF-03

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

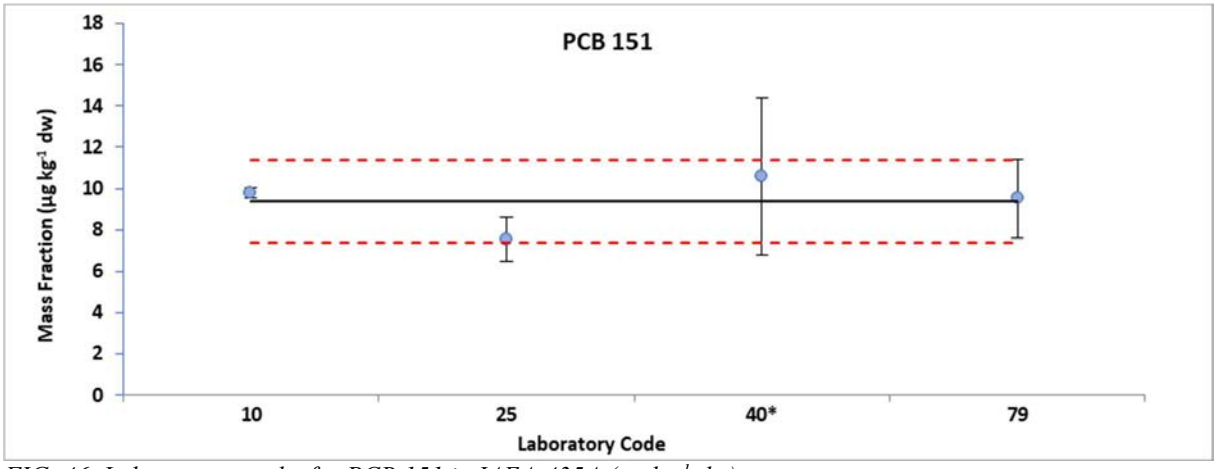

FIG. 46. Laboratory results for PCB 151 in IAEA-435A (µg kg^{-1} dw)

TABLE 61. RESULTS REPORTED BY PARTICIPANTS FOR PCB 174 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
40*	7.97	2.80	0.02	GC/HRMS	FAPAS 0685, IAEA-459
79	7.36	1.47	0.01	GC/ECD[1]	NIST 2974a, WMF-03

* Laboratory accredited
[1]GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

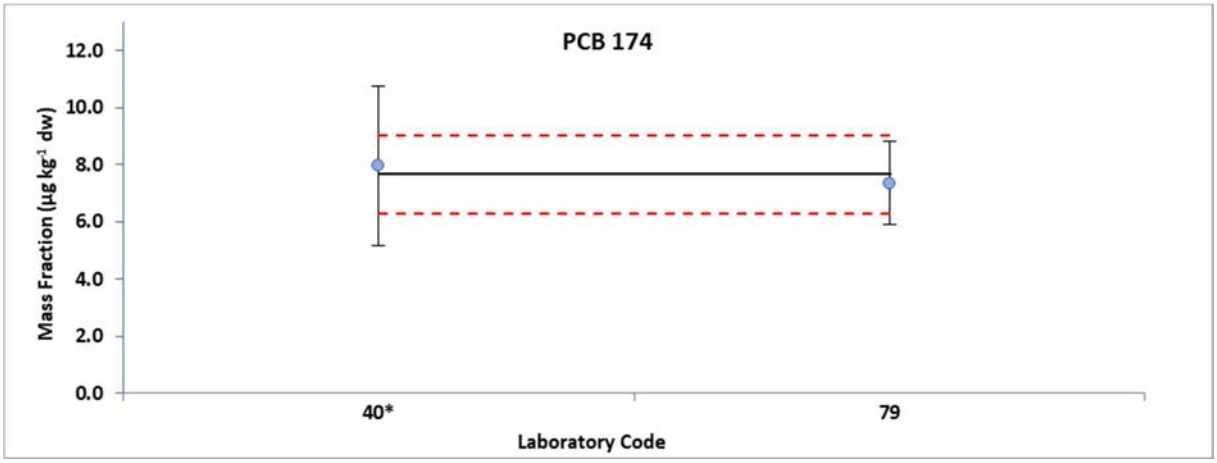

FIG. 47. Laboratory results for PCB 174 in IAEA-435A (µg kg^{-1} dw)

TABLE 62. RESULTS REPORTED BY PARTICIPANTS FOR PCB 177 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
10	10.7	0.5[1]	0.01	GC/MSMS	
25	6.52	0.86	0.03	GC/MSMS	
40*	9.92	3.50	0.20	GC/HRMS	FAPAS 0685, IAEA-459
79	8.63	1.73	0.01	GC/ECD[2]	NIST 2974a, WMF-03

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

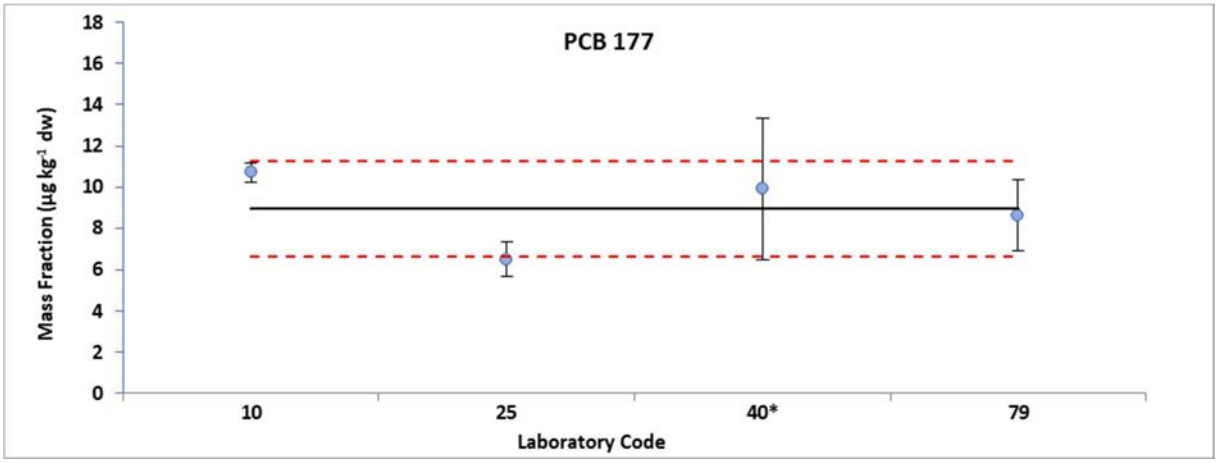

FIG. 48. Laboratory results for PCB 177 in IAEA-435A (µg kg^{-1} dw)

TABLE 63. RESULTS REPORTED BY PARTICIPANTS FOR PCB 183 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
10	9.70	0.31[1]	0.01	GC/MSMS	
25	6.77	0.27	0.03	GC/MSMS	
40*	14.1	4.9	0.03	GC/HRMS	FAPAS 0685, IAEA-459
79	9.99	2.00	0.02	GC/ECD[2]	NIST 2974a, WMF-03

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

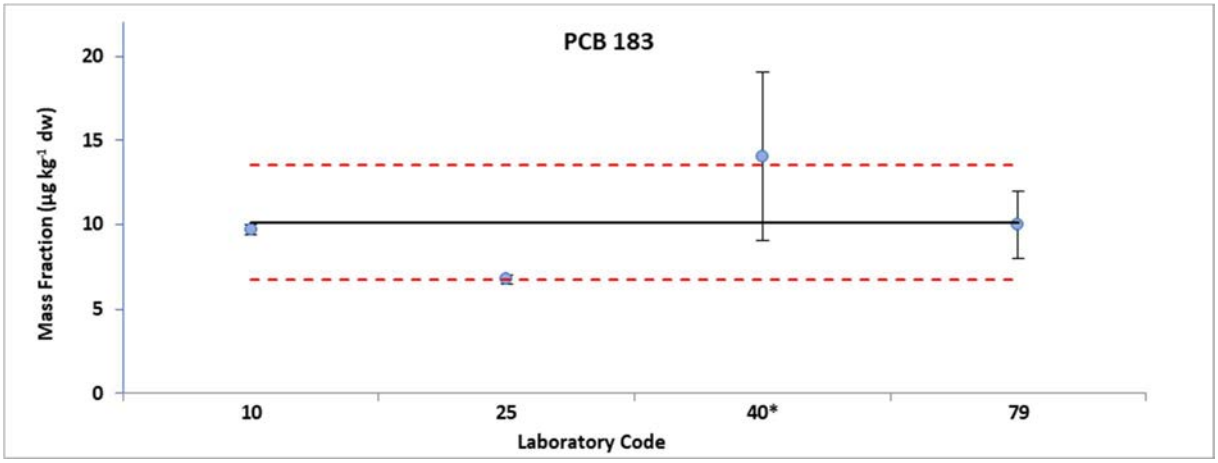

FIG. 49. Laboratory results for PCB 183 in IAEA-435A (µg kg^{-1} dw)

TABLE 64. RESULTS REPORTED BY PARTICIPANTS FOR PCB 195 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
40*	1.13	0.40	0.03	GC/HRMS	FAPAS 0685, IAEA-459
79	1.51	0.30	0.004	GC/ECD[1]	NIST 2974a, WMF-03

* Laboratory accredited
[1]GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

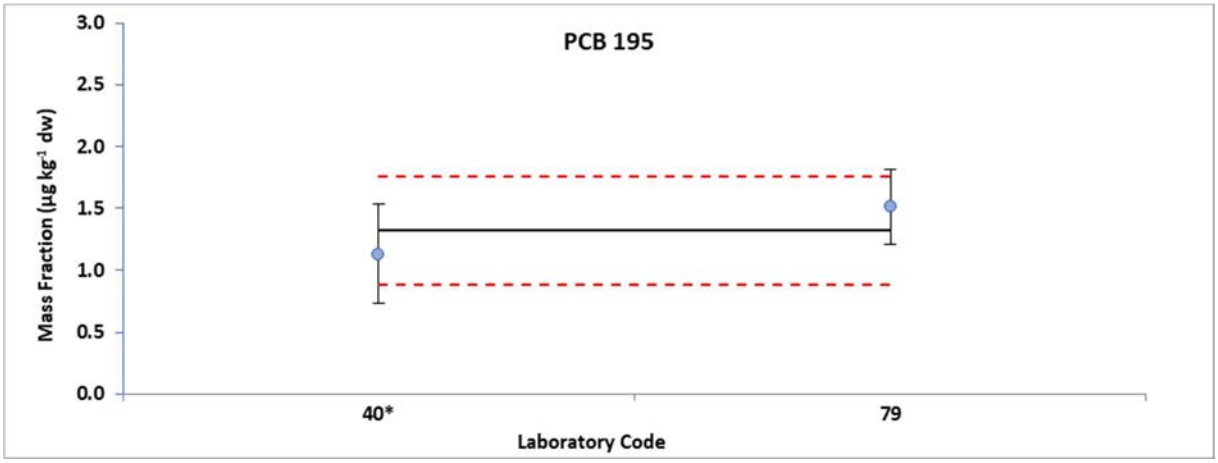

FIG. 50. Laboratory results for PCB 195 in IAEA-435A (µg kg^{-1} dw)

TABLE 65. RESULTS REPORTED BY PARTICIPANTS FOR PCB 201 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
40*	4.03	1.40	0.04	GC/HRMS	FAPAS 0685, IAEA-459
79	3.14	0.63	0.01	GC/ECD[1]	NIST 2974a, WMF-03

* Laboratory accredited
[1]GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

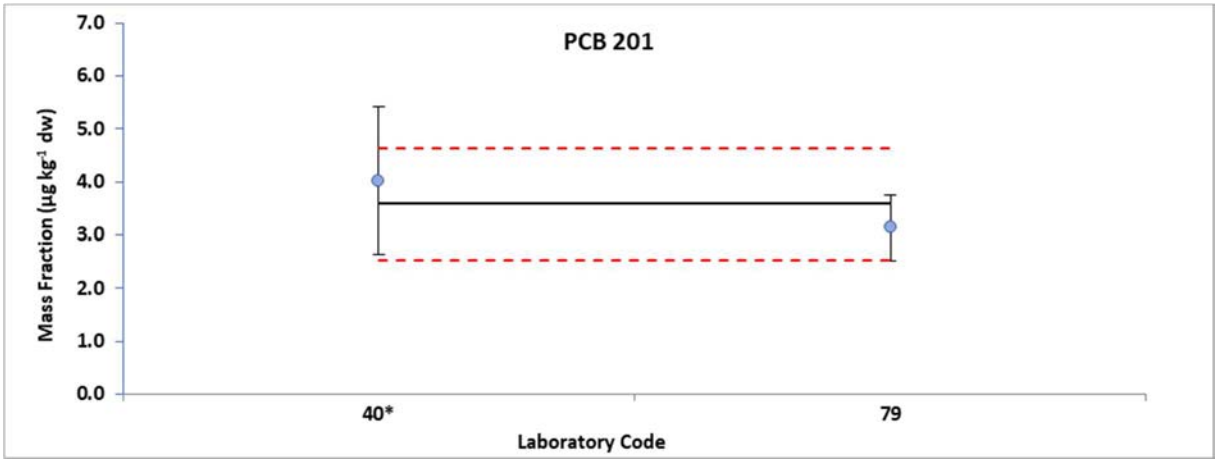

FIG. 51. Laboratory results for PCB 201 in IAEA-435A (µg kg^{-1} dw)

TABLE 66. RESULTS REPORTED BY PARTICIPANTS FOR PCB 206 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
40*	3.28	1.10	0.06	GC/HRMS	FAPAS 0685, IAEA-459
79	3.16	0.63	0.01	GC/ECD[1]	NIST 2974a, WMF-03

* Laboratory accredited
[1]GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

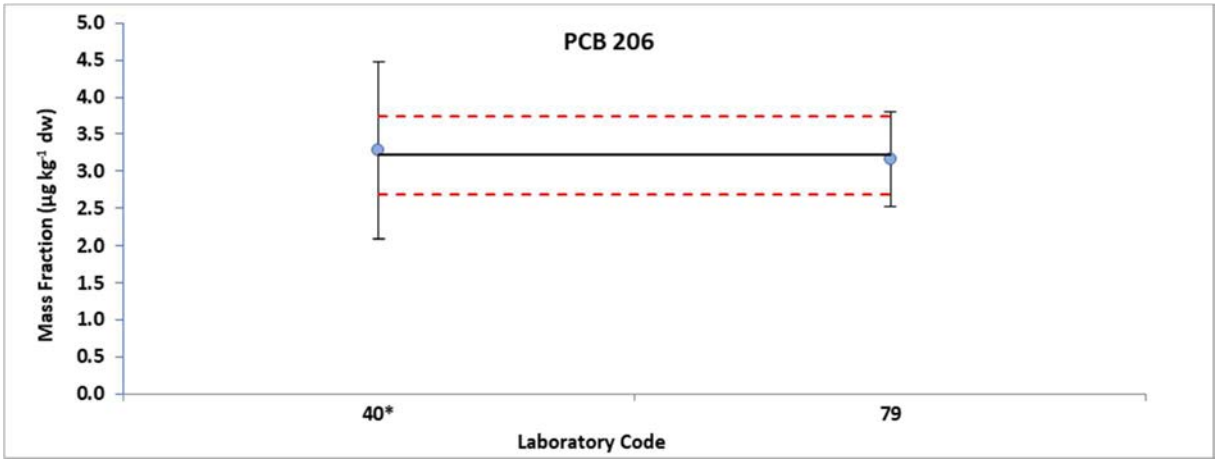

FIG. 52. Laboratory results for PCB 206 in IAEA-435A (µg kg^{-1} dw)

TABLE 67. RESULTS REPORTED BY PARTICIPANTS FOR PCB 209 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
40*	2.11	0.13	0.06	GC/HRMS	FAPAS 0685, IAEA-459
78*	1.67	0.35[1]	0.30	GC/MSMS	
79	2.01	0.40	0.01	GC/ECD[2]	NIST 2974a, WMF-03

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

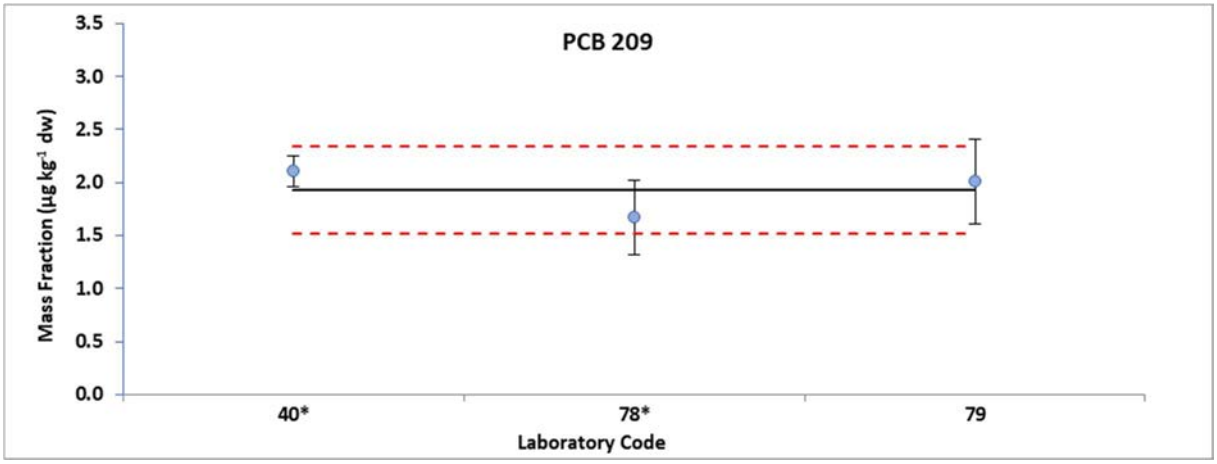

FIG. 53. Laboratory results for PCB 209 in IAEA-435A (µg kg^{-1} dw)

II.2. INFORMATION VALUES FOR ORGANOCHLORINE PESTICIDES

TABLE 68. RESULTS REPORTED BY PARTICIPANTS FOR α-HCH (μg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
5	0.02	0.01[1]		GC/MSMS	IAEA-435, IAEA-432
25	<0.10		0.03	GC/MSMS	
40*	<0.15		0.09	GC/HRMS	FAPAS 0685, IAEA-459
42	<0.08			GC/MSMS	spiked blank
54	<1.70		1.70	GC/MSMS	
78*	<2.00		0.70	GC/MSMS	
79	<0.01		0.01	GC/ECD[2]	NIST 2974a, WMF-03
Results not used for the assignment of the value					
27	0.19	0.01[1]	0.02	GC/ECD	
72*	0.42	0.08	0.10	GC/MS EI	NIST 1947

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

TABLE 69. RESULTS REPORTED BY PARTICIPANTS FOR β-HCH (μg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
3	0.05		0.05	GC/MSMS	NIST 2974a
5	0.17	0.01[1]		GC/MSMS	IAEA-435, IAEA-432
25	<0.10		0.03	GC/MSMS	
40*	0.24	0.02	0.06	GC/HRMS	FAPAS 0685, IAEA-459
42	0.13	0.01[1]		GC/MSMS	spiked blank
54	<1.70		1.70	GC/MSMS	
72*	<0.30		0.10	GC/MS EI	NIST 1947
78*	<2.00		0.70	GC/MSMS	
Results not used for the assignment of the value					
79	0.64	0.13	0.13	GC/ECD[2]	NIST 2974a, WMF-03

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

TABLE 70. RESULTS REPORTED BY PARTICIPANTS FOR γ-HCH (LINDANE) (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
3	0.18	0.03[1]	0.05	GC/MSMS	NIST 2974a
5	0.09	0.02[1]		GC/MSMS	IAEA-435, IAEA-432
25	<0.10		0.03	GC/MSMS	
27	0.30	0.04[1]	0.02	GC/ECD	
40*	0.12	0.01	0.06	GC/HRMS	FAPAS 0685, IAEA-459
42	0.12	0.01[1]		GC/MSMS	spiked blank
54	<1.70		1.70	GC/MSMS	
72*	<0.30		0.10	GC/MS EI	NIST 1947
78*	<2.00		0.70	GC/MSMS	
79	0.10	0.02	0.01	GC/ECD[2]	NIST 2974a, WMF-03
Results not used for the assignment of the value					
27	0.30	0.04[1]	0.02	GC/ECD	

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

TABLE 71. RESULTS REPORTED BY PARTICIPANTS FOR δ-HCH (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
25	<0.10		0.03	GC/MSMS	
40*	<0.15		0.15	GC/HRMS	FAPAS 0685, IAEA-459
54	<1.70		1.70	GC/MSMS	
78*	<2.00		0.70	GC/MSMS	
79	0.10	0.02	0.10	GC/ECD[1]	NIST 2974a, WMF-03

* Laboratory accredited

[1] GC/ECD and peak confirmation with GC/MSMS

TABLE 72. RESULTS REPORTED BY PARTICIPANTS FOR HEPTACHLOR (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
10	0.06	0.01[1]		GC/MSMS	
25	<0.10		0.03	GC/MSMS	
40*	<0.16		0.05	GC/HRMS	FAPAS 0685, IAEA-459
42	<0.08			GC/MSMS	spiked blank
54	<1.70		1.70	GC/MSMS	
72*	<0.30		0.10	GC/MS EI	NIST 1947
78*	<1.00		0.30	GC/MSMS	
79	<0.01		0.01	GC/ECD[2]	NIST 2974a, WMF-03

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

TABLE 73. RESULTS REPORTED BY PARTICIPANTS FOR ALDRIN (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
3	0.08	0.07[1]	0.05	GC/MSMS	NIST 2974a
25	<0.10		0.03	GC/MSMS	
40*	<0.06		0.03	GC/HRMS	FAPAS 0685, IAEA-459
54	<1.70		1.70	GC/MSMS	
72*	<0.30		0.10	GC/MS EI	NIST 1947
78*	<12.5		4.0	GC/MSMS	
79	<0.09		0.09	GC/ECD[2]	NIST 2974a, WMF-03
Results not used for the assignment of the value					
27	0.28	0.02[1]	0.02	GC/ECD	

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

TABLE 74. RESULTS REPORTED BY PARTICIPANTS FOR ENDRIN (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
3	0.05		0.05	GC/MSMS	NIST 2974a
25	<0.10		0.03	GC/MSMS	
40*	<0.15		0.09	GC/HRMS	FAPAS 0685, IAEA-459
54	<3.30		3.30	GC/MSMS	
78*	<12.5		4.0	GC/MSMS	
79	<0.50		0.20	GC/ECD[1]	NIST 2974a, WMF-03

* Laboratory accredited

[1] GC/ECD and peak confirmation with GC/MSMS

TABLE 75. RESULTS REPORTED BY PARTICIPANTS FOR *trans*-CHLORDANE (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
40*	0.33	0.03	0.01	GC/HRMS	FAPAS 0685, IAEA-459
72*	0.39	0.02	0.10	GC/MS EI	NIST 1947
79	0.42	0.08	0.003	GC/ECD[2]	NIST 2974a, WMF-03
Results not used for the assignment of the value					
27	<0.05		0.02	GC/ECD	
42	5.43	0.21[1]		GC/MSMS	spiked blank
54	<3.30		3.30	GC/MSMS	
78*	7.60		0.30	GC/MSMS	

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

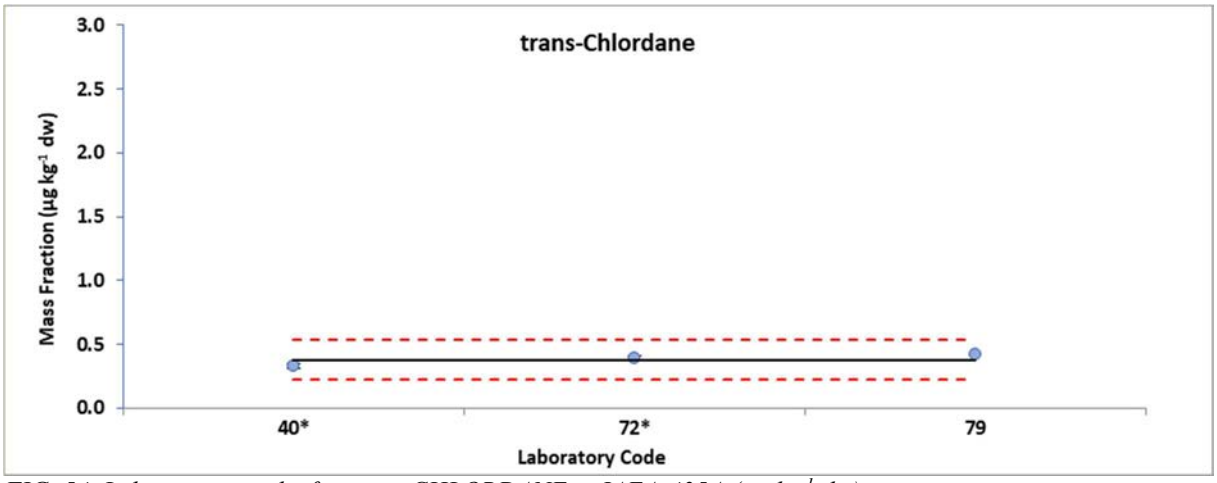

FIG. 54. Laboratory results for trans-CHLORDANE in IAEA-435A (µg kg^{-1} dw)

TABLE 76. RESULTS REPORTED BY PARTICIPANTS FOR *cis*-NONACHLOR (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
40*	11.3	0.7	0.02	GC/HRMS	FAPAS 0685, IAEA-459
54	11.2	5.6	3.30	GC/MSMS	
79	11.3	2.3	0.02	GC/ECD[1]	NIST 2974a, WMF-03
Results not used for the assignment of the value					
72*	13.8	0.9	0.10	GC/MS EI	NIST 1947

* Laboratory accredited

[1] GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

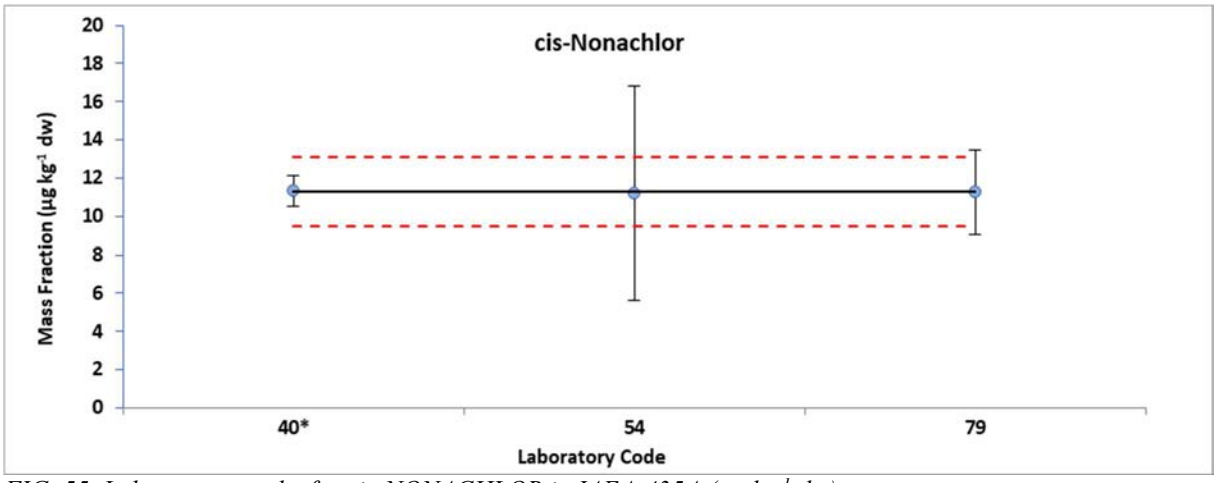

FIG. 55. Laboratory results for cis-NONACHLOR in IAEA-435A (µg kg^{-1} dw)

** Robust mean of the reported values

TABLE 77. RESULTS REPORTED BY PARTICIPANTS FOR α-ENDOSULFAN (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
25	<0.10		0.03	GC/MSMS	
40*	<0.50		0.30	GC/HRMS	FAPAS 0685, IAEA-459
54	<3.30		3.30	GC/MSMS	
72*	<0.30		0.10	GC/MS EI	NIST 1947
78*	<12.5		4.0	GC/MSMS	
79	<0.52		0.16	GC/ECD[1]	NIST 2974a, WMF-03

* Laboratory accredited

[1] GC/ECD and peak confirmation with GC/MSMS

TABLE 78. RESULTS REPORTED BY PARTICIPANTS FOR β-ENDOSULFAN (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
25	<0.10		0.03	GC/MSMS	
40*	<1.00		0.60	GC/HRMS	FAPAS 0685, IAEA-459
54	<3.30		3.30	GC/MSMS	
78*	<12.5		4.0	GC/MSMS	
79	<0.09		0.09	GC/ECD[1]	NIST 2974a, WMF-03

* Laboratory accredited

[1] GC/ECD and peak confirmation with GC/MSMS

TABLE 79. RESULTS REPORTED BY PARTICIPANTS FOR ENDOSULFAN SULFATE (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
54	<3.30		3.30	GC/MSMS	
78*	<12.5		4.0	GC/MSMS	
79	<0.18		0.18	GC/ECD[1]	NIST 2974a, WMF-03

* Laboratory accredited

[1] GC/ECD and peak confirmation with GC/MSMS

TABLE 80. RESULTS REPORTED BY PARTICIPANTS FOR HEPTACHLOR EPOXIDE (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
10	0.48	0.02[1]		GC/MSMS	
40*	0.84	0.05	0.05	GC/HRMS	FAPAS 0685, IAEA-459
72*	0.97	0.05	0.10	GC/MS EI	NIST 1947
79	0.61	0.12	0.12	GC/ECD[2]	NIST 2974a, WMF-03
Results not used for the assignment of the value					
25	<0.10		0.03	GC/MSMS	
54	<3.30		3.30	GC/MSMS	
78*	<12.5		4.0	GC/MSMS	

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

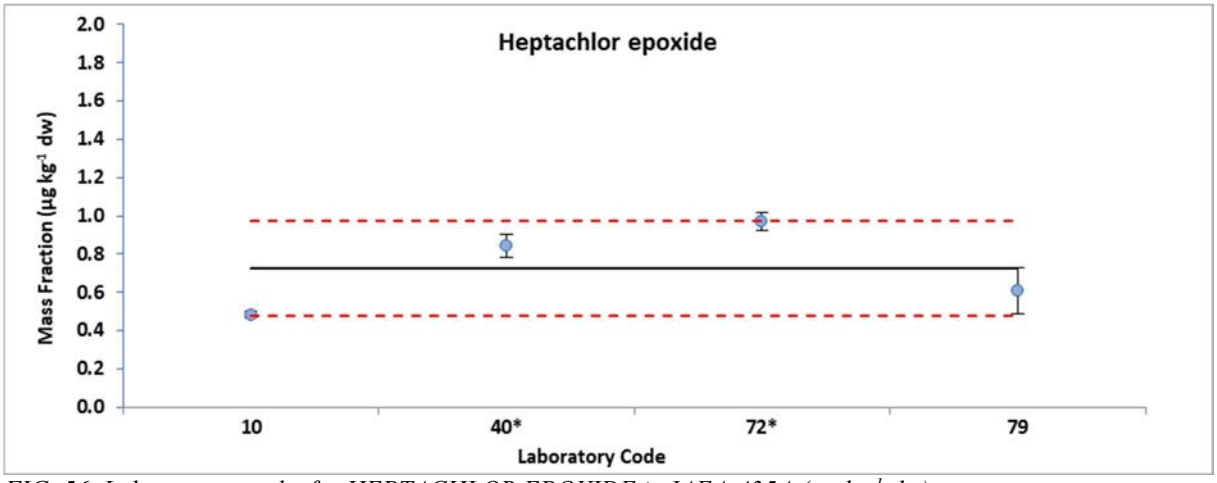

FIG. 56. Laboratory results for HEPTACHLOR EPOXIDE in IAEA-435A (µg kg^{-1} dw)

II.3. INFORMATION VALUES FOR PBDES

TABLE 81. RESULTS REPORTED BY PARTICIPANTS FOR BDE 17 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
38	0.04	0.27	0.003	GC/HRMS	In House Native Spike Solution
40*	0.03	0.01	0.01	GC/HRMS	IC2019SE
79	<0.06		0.02	GC/ECD[1]	NIST 2974a, WMF-03

* Laboratory accredited

[1] GC/ECD and peak confirmation with GC/MSMS

TABLE 82. RESULTS REPORTED BY PARTICIPANTS FOR BDE 49 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
38	2.46	0.50	0.01	GC/HRMS	In House Native Spike Solution
40*	1.99	0.70	0.01	GC/HRMS	IC2019SE
74*	2.12	0.03[1]	0.003	GC/MSMS	

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

Reported results and expanded uncertainties:

$X_{lab} \pm U_{lab}$ (k=2); — X_a - - $X_a \pm U_a$ (k=2)

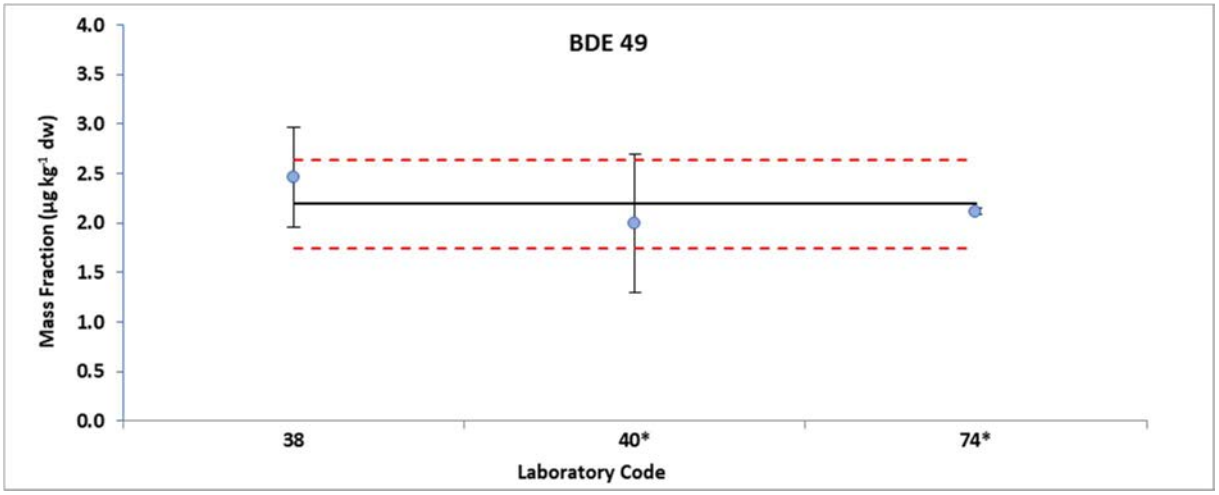

FIG. 57. Laboratory results for BDE 49 in IAEA-435A (µg kg^{-1} dw)

TABLE 83. RESULTS REPORTED BY PARTICIPANTS FOR BDE 85 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
38	0.022	1.010	0.016	GC/HRMS	In House Native Spike Solution
40*	<0.028		0.028	GC/HRMS	IC2019SE
74*	0.021	0.013[1]	0.003	GC/MSMS	
79	0.103	0.030	0.010	GC/ECD[2]	NIST 2974a, WMF-03

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

TABLE 84. RESULTS REPORTED BY PARTICIPANTS FOR BDE 183 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
14	<0.008		0.003	GC/HRMS	WMF-02 Wellington
38	<0.010	1.520	0.013	GC/HRMS	In House Native Spike Solution
40*	0.022	0.002	0.013	GC/HRMS	IC2019SE
74*	0.002	0.001[1]	0.005	GC/MSMS	

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

TABLE 85. RESULTS REPORTED BY PARTICIPANTS FOR BDE 209 (µg kg^{-1} dw)

Lab code	X_{lab}	U_{lab} (k=2)	Detection Limit	Instrument Type	Quality Control
38	<2.00	12.33	2.95	GC/HRMS	In House Native Spike Solution
40*	0.05	0.01	0.02	GC/HRMS	IC2019SE
74*	0.10	0.05[1]	0.01	GC/MSMS	
79	<2.05		2.05	GC/ECD[2]	NIST 2974a, WMF-03

* Laboratory accredited

[1] Uncertainty not reported by participant and calculated as: $2\frac{s}{\sqrt{n}}$ where s is the standard deviation and n is the number of measurements reported

[2] GC/ECD and peak confirmation with GC/MSMS

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ANNEX: DOCUMENTS SENT TO PARTICIPATING LABORATORIES

Atoms for Peace and Development

الوكالة الدولية للطاقة الذرية
国 际 原 子 能 机 构
International Atomic Energy Agency
Agence internationale de l'énergie atomique
Международное агентство по атомной энергии
Organismo Internacional de Energía Atómica

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Email: el@iaea.org • Internet: http://www.iaea.org/environmentlaboratories
In reply please refer to: CHARACTERIZATION-IAEA-MESL-2019-01-OC

INFORMATION SHEET: CHARACTERIZATION-IAEA-MESL-2019-01-OC
PCBs, organochlorine pesticides, PBDEs and PAHs in a fish sample.

PLEASE READ THIS INFORMATION CAREFULLY BEFORE STARTING THE ANALYSES AND FILLING IN THE ONLINE DATA REPORTING FORM!

The present exercise is specifically organized for the determination of **PCBs, organochlorine pesticides, PBDEs and PAHs in a fish sample: CHARACTERIZATION-IAEA-MESL-2019-01-OC.**

Description of the material:

A marine fish sample was collected for interlaboratory comparison purpose. This sample was deep-frozen, freeze-dried, ground and sieved through a 250 µm stainless steel sieve.
This powder, with a particle size of less than 250 µm was homogenized by mixing in a stainless-steel rotating drum for two weeks. Then aliquots of about 30 grams were packaged into glass bottles with aluminum screw caps and sealed with Teflon tape.

Homogeneity test:

The homogeneity of the material for PCBs, organochlorine pesticides, PBDEs and PAHs was checked by determining the concentration of these compounds in several samples taken randomly in the bulk of the powder. A one-way variance analysis indicated that the material can be considered as homogenous.

Moisture content:

Since the moisture content may change with the ambient humidity and temperature, it is recommended that the water content of this material always be determined in a separate sub-sample (not taken for analysis) by drying to **constant weight at 105 °C for 8 hrs**.

All results are to be reported on a dry mass basis using the unit specified on the reporting form.

Compounds to be determined:

The participants are requested to determine as many compounds as possible from the attached list using their established analytical methods.

Analytical quality control:

Procedures of quality control and laboratory quality assurance are recommended to be applied. The results of the analyses of the quality control (QC) sample should be reported together with the results from PT sample on the same reporting forms.

Reporting of results:

1.The participants are requested to make at least **six independent determinations** and to report the results together with a short **description of the method used**. Participants are also required to provide **proof of traceability** of obtained results to the International System of Units (SI), via standard calibration solutions and CRM applied, as a part of their analytical procedures.

2.The concentrations reported must be calculated on a dry mass basis and given as ng g^{-1}, leaving as many significant figures as justified by the precision of the method used.

3.If a compound is not detected by the method used, the corresponding limit of detection should be given rather than the statement "not detected".

4.For each class of compounds, the participants **MUST** report information about:

- Water content (in %);
- % Lipid;
- Extraction technique;
- Quantity extracted;
- Solvent used;
- Samples clean up;
- Samples fractionation;
- Detection method;
- Injector;
- GC or HPLC column;
- Results quantification;
- Statement on traceability of the obtained measurement results (standards and reference materials used);
- QA/QC;
- Surrogates spiked before extraction;
- Internal standards spiked before injection;
- Use of CRMs;
- Use of validated method;
- Accreditation;

5. For each compound the participants **MUST** report:

- Detection limit;
- Quantification limit;
- At least three determinations;
- Combined uncertainty;
- Expanded uncertainty;
- Coverage factor;
- Surrogate used;
- Result of CRM used for QC;
- CRM value (on certificate);

OTHER NOTES

1. About two weeks before the deadline, the organizers of the Proficiency Test will send to all participating laboratories deadline reminder and further instructions for the on-line submission of results by Email.

   The deadline for returning the results is **3 March 2020**.

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List of Chlorinated pesticides and PCB compounds to be measured:

HCB
α HCH
β HCH
g HCH- Lindane
pp′ DDE
pp′ DDD
pp′ DDT
δ HCH
op' DDE
op' DDD
op' DDT
Heptachlor
Aldrin
Dieldrin
Endrin
cis-Chlordane
trans-Chlordane
cis-Nonachlor
trans-Nonachlor
α Endosulfan
β Endosulfan
Endosulfan sulfate
Heptachlor epoxide
PCB 8
PCB 18
PCB 28
PCB 31
PCB 44
PCB 49
PCB 52
PCB 66
PCB 70
PCB 74
PCB 95
PCB 87
PCB 97
PCB 99
PCB 101
PCB 105
PCB 110
PCB 118
PCB 128
PCB 138
PCB 138 (+169)
PCB 149
PCB 146
PCB 151
PCB 153
PCB 156
PCB 170
PCB 174
PCB 177
PCB 180
PCB 180 (+193)
PCB 183
PCB 187
PCB 194
PCB 195
PCB 201
PCB 206
PCB 209

List of Polybrominated diphenyl ethers (PBDEs) to be measured:

BDE 17

BDE 28

BDE 28 + 33

BDE 47

BDE 49

BDE 66

BDE 85

BDE 99

BDE 100

BDE 153

BDE 154

BDE 183

BDE 209

List of Polycyclic Aromatic Hydrocarbons (PAHs) and aliphatic hydrocarbons to be measured:

Naphthalene

2-Methylnaphthalene

1-Methylnaphthalene

1,6-Dimethylnaphthalene + 2,7 -Dimethylnaphthalene

Biphenyl

Acenaphthene

Acenaphthylene

Fluorene

Phenanthrene

Anthracene

1-Methylphenanthrene

2-Methylphenanthrene

3,6-Dimethylphenanthrene

Fluoranthene

Pyrene

Benz(a)anthracene

Chrysene

Benzo(b)fluoranthene

Benzo(e)pyrene

Benzo(a)pyrene

Benzo(g,h,i)perylene

List of aliphatic hydrocarbons to be measured:

n-C17

Pristane

n-C18

Phytane

ABBREVIATIONS

ANOVA	Analysis of variance
CRM	Certified reference material
BDE	Brominated diphenyl ether
GC/ECD	Gas Chromatography/Electron Capture Detector
GC/MS EI	Gas chromatography/Mass Spectrometry, Electron Impact
GC/HRMS	Gas Chromatography/High Resolution Mass Spectrometry
GC/MSMS	Gas Chromatography/Tandem Mass Spectrometry
GUM	Guide to the Expression of Uncertainty in Measurement
ILC	Interlaboratory Comparison
IAEA-NAML	International Atomic Energy Agency-Nuclear Applications Marine Laboratories
ISO	International Organization for Standardization
JCGM	Joint Committee for Guides in Metrology
MESL	Marine Environmental Studies Laboratories
NIST	National Institute of Standards and Technology
OCs Pest.	Organochlorine Pesticides
PAH	Polycyclic Aromatic Hydrocarbon
PCB	Polychlorinated Biphenyl
PBDE	Polybrominated diphenyl ether
POP	Persistent organic pollutant
PT	Proficiency test
RM	Reference material
SI	International System of Units
SC	Stockholm Convention
SPE	Solid Phase Extraction
SRM	Standard Reference Material
QA/QC	Quality Assurance/Quality Control
SPE	Solid Phase Extraction
RM	Reference material
WMF	Wellington Laboratories
PT	Proficiency test

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