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Certification of Polycyclic Aromatic Hydrocarbon Mass Fractions in IAEA-477 Sediment Sample

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CERTIFICATION OF POLYCYCLIC AROMATIC HYDROCARBON MASS FRACTIONS IN IAEA-477 SEDIMENT SAMPLE

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INTERNATIONAL ATOMIC ENERGY AGENCY
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For further information on this publication, please contact:

IAEA Environment Laboratories, Monaco
Marine Environmental Studies Laboratory
International Atomic Energy Agency
4a Quai Antoine 1er, 98000
Principality of Monaco

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FOREWORD

The IAEA Environment Laboratories assist Member States in understanding, monitoring and protecting both the terrestrial and the marine environment. To assess the impact of land based and sea based pollution sources on marine coastal environments, it is imperative to ensure the quality and comparability of the analytical data generated by national and regional pollution monitoring programmes. For this purpose, the IAEA Environment Laboratories have been assisting national laboratories and regional laboratory networks through the Reference Products for Science and Trade programme since the early 1970s. This is accomplished through the production of certified reference materials, the provision of training in quality assurance, and the evaluation of measurement performances through worldwide and regional interlaboratory comparison exercises and proficiency tests.

This publication describes the production of the certified reference material IAEA-477 produced by the IAEA Environment Laboratories following ISO Standard ISO 17034:2016 and ISO/Guide 35:2017. This material is a sediment sample with certified mass fractions of polycyclic aromatic hydrocarbons. The assigned final values and their associated uncertainties were derived from the results provided by selected laboratories with demonstrated technical and quality competence, following the guidance provided in ISO/Guide 35:2017 and in the Guide to the Expression of Uncertainty in Measurement.

The material is intended to be used for quality control and assessment of method performance in the determination of mass fractions of polycyclic aromatic hydrocarbons, which are included in the group of priority substances in many environment monitoring programmes.

The IAEA is grateful to the Government of Monaco for the support provided to its Environment Laboratories and to the Permanent Mission of Australia to the United Nations in New York and its International Development Fund for their financial support under the IAEA's Peaceful Uses Initiative. The IAEA also acknowledges all the laboratories that participated in the characterization study of this reference material, and the Australian Nuclear Science and Technology Organisation and James Cook University for conducting the sampling mission. Planning of the sampling mission and collection of the sediment samples was performed by J. Daniell of James Cook University, and M. Johansen, E. Prentice, H. Heijnis and M. Corry of ANSTO. The IAEA officers responsible for this publication were I. Tolosa and R. Cassi of the IAEA Environment Laboratories.

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1. INTRODUCTION

The Marine Environmental Studies Laboratory (MESL) of the IAEA-EL provides assistance to Member States' laboratories to enhance the quality of the analytical measurement results for trace elements and organic contaminants in marine environmental samples. This is achieved through the production of CRMs, organization of Interlaboratory Comparisons and Proficiency Tests, and by conducting Training Courses on the analysis of contaminants in marine samples. This activity is undertaken in the framework of the IAEA-EL's Sub-Programme 2.4.1 "Reference Products for Science and Trade" and the Project 2.4.1.1 "Provision of Reference Products and Assurance of Laboratory Performance".

CRMs are valuable tools for analytical method development and validation to improve the measurements and quality control in analytical laboratories. More specifically, marine matrix CRMs are needed to ensure the reliability of the analytical measurements and ensure the use of high quality data as the basis for decision making in national or regional marine pollution monitoring programmes. Polycyclic aromatic hydrocarbons (PAHs) comprise a large group of more than several hundred chemical compounds containing two or more fused aromatic rings. They are produced during incomplete combustion of organic matter or released from oil spills. Because many of the PAHs compounds are known to be carcinogenic and genotoxic, they are included under the group of priority substances (PSs) in many environment monitoring programmes. The most important lists of monitored PAHs are a group of 16 PAHs listed by the U. S. Environmental Protection Agency (EPA), and the 15+1 European Union (EU) priority PAHs to be monitored in foods. While there are several CRMs certified for PAHs, there is still a noticeable lack of matrix CRMs, in particular in marine sediments, where the concentrations levels are in the low ng g^{-1} range. To meet this need, MESL has developed a CRM in a natural and remote sediment for the determination of PAHs in the environment monitoring programmes.

This publication describes and provides information on the sample preparation methodology followed and on the assignment of property values with their associated uncertainties for a number of polycyclic aromatic hydrocarbons in a marine sediment sample. Certification of the mass fractions was made for major polycyclic aromatic hydrocarbons (PAHs). 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], which combines the different uncertainties of characterization, heterogeneity and instability.

The CRM IAEA-477 was produced to satisfy the needs of laboratories to strengthen data quality assurance in the analysis of priority substances in marine samples. The low concentration levels of this CRM make it a suitable material for monitoring PAHs in marine environments where the concentration levels are relatively low and close to the method detection limits.

2. METHODOLOGY

2.1 COLLECTION AND PREPARATION OF THE MATERIAL

Two sediment samples were collected in Queensland region, Australia using a Van Veen type sampler. One site was sampled in Townsville Marina and the other in Townsville Ross River. Frozen samples were sent from Brisbane to IAEA laboratories in Monaco, and then sent for freeze-drying to LYOFAL (France) and to micronization at 20 µm to AFT-Micro-Macinazione (France). Sediment was further γ-irradiated at doses ranging from 27 to 39.5 kGy at Steris (France). The final powdered composite sediment, about 15 kg, was homogenized by mixing it in a stainless-steel rotating drum for three weeks. Then, aliquots of about 50 grams were packaged into cleaned amber glass bottles with screw caps and labeled IAEA-477.

The particle size analysis was performed by laser diffraction light using a Mastersizer 2000 equipped with a Sirocco dispenser for a range of particles from 0.1 to 1000 µm. The sample showed 100% of particulates below 20 µm (Figure 1).

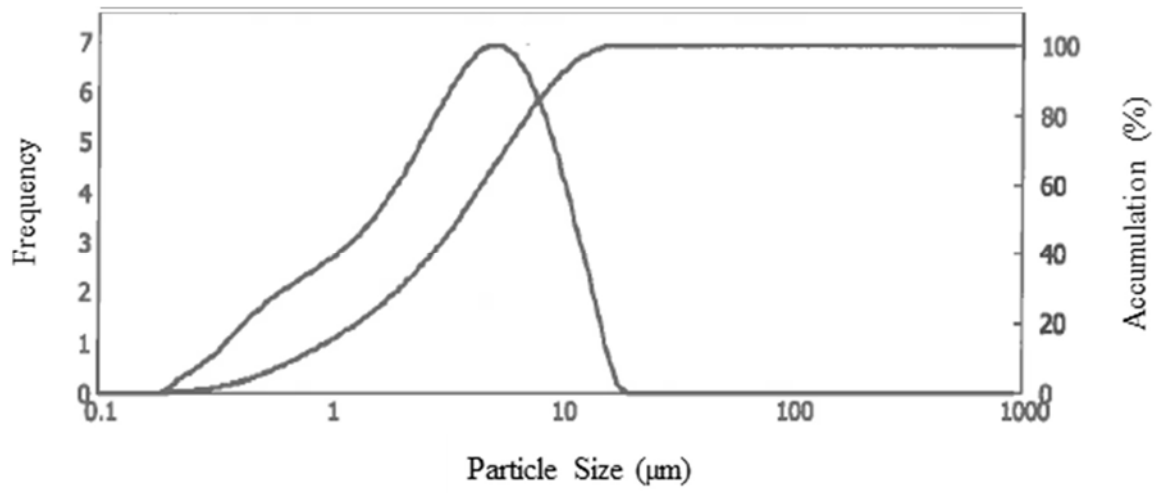

Fig. 1. Particle size distribution of IAEA-477.

2.2. SELECTION OF LABORATORIES FOR THE CHARACTERIZATION STUDY

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

One bottle of sediment sample was sent to each participating laboratory, accompanied by an information sheet and a reporting form to include the data results for polycyclic aromatic

hydrocarbons (PAHs). The participating laboratories were requested to analyse six aliquots using their usual technique, and the reported results had to be accompanied with the description of the method used together with the applied quality control procedures, including results for the organic contaminants in a CRM with a matrix similar to the candidate reference material.

The list of laboratories participating in the characterization study is provided on page 83.

2.3. HOMOGENEITY ASSESSMENT

In order to establish the degree of homogeneity of the reference material with respect to the properties of interest, both within- and between-unit homogeneity have to be evaluated to ensure that all distributed units are the same within the stated uncertainty [1].

Homogeneity test was performed by IAEA-EL in Monaco after the bottling of the sample material. The between-bottle homogeneity of the material was assessed by determining the concentration of selected parent PAHs in duplicate analysis from 10 bottles (5 g sample intake) randomly selected during the whole bottling process of the bulk dry powder. The analytic method used included microwave extraction with hexane/methylene chloride (1:1), sulphur removal by copper and fractionation by solid phase extraction (SPE) to isolate PAHs from aliphatic hydrocarbons. The final measurements of PAHs were performed by gas chromatography coupled to mass spectrometry using selected ion monitoring (GC-MS-SIM) under “quasi” repeatability conditions and in a randomized order to be able to separate an analytical drift from a trend in the filling sequence.

2.4. CHARACTERIZATION

Characterization refers to the process of assigning a property value that can be reliably assessed when its value is confirmed by several laboratories working independently and using different methods, for each of which the accuracy has been well established [3]. The material was initially analysed in the IAEA-EL in Monaco. The final characterization was based on the results provided by selected laboratories with demonstrated technical and quality competence. Although having a formal accreditation was not mandatory, all laboratories participating in the characterization campaign provided their results with their method validation data in accordance with the guidelines of ISO 17025 [4].

As it is shown in Table 1, the characterization and the value assignment approach for the PAH analytes included a combination of results derived from analyses using different solvents and extraction techniques, a variety of different cleanup and fractionation procedures and diverse instrumental detection techniques. Consequently, 12 independent datasets were obtained for PAHs.

TABLE 1. ANALYTICAL METHODOLOGIES USED FOR THE CERTIFICATION OF PAHS IN CRM IAEA-477

Lab	Extraction Procedure	Solvent	Cleanup	Fractionation	Equipment	Chromatographic Column
1	Microwave	DCM	SPE	Silica/Alumina	GC-MS	5% Phenyl DMPsiloxane & Select PAH
2	Microwave	Acetone/n-Hexane	GPC[2]	Silica	GC-MS/MS	5% Phenyl DMPsiloxane
3	ASE	n-Hexane/DCM	None	None	HPLC-FLD	C18
4	Sohxlet	DCM	Silica	Silica	GC-MS	5% Phenyl DMPsiloxane
5	Sohxlet	n-Hexane/ DCM	SPE	Silica	GC-MS	5% Phenyl DMPsiloxane
6	ASE	n-Hexane/ DCM	SPE	Silica	GC-MS	50% Phenyl DMPsiloxane
7	HP hot LE[1]	Toluene/Acetone	None	None	GC-HRMS	5% Phenyl DMPsiloxane
9	Microwave	Acetone/n-Hexane	Adsorption Column	Silica	GC-MSMS	5% Phenyl DMPsiloxane
10	Sohxlet	Methanol/ DCM	Saponification	Silica	GC-MS	5% Phenyl DMPsiloxane
11	ASE	n-Hexane/ DCM	Silica	Silica	GC-MS	5% Phenyl DMPsiloxane
12	ASE	Acetone/DCM	None	None	GC-MS	5% Phenyl DMPsiloxane
13	Microwave	n-Hexane/ DCM	SPE	Silica/Cyanopropyl	GC-MS	5% Phenyl DMPsiloxane

[1] High performance hot liquid extraction

[2] Gel permeation chromatography

The extraction procedures of the PAHs were performed through accelerated solvent extraction (ASE), microwave, Soxhlet, high performance hot liquid extraction by using different solvents mixtures, including dichloromethane (DCM), hexane, acetone, toluene and methanol. After further cleanup with copper/sorbents and/or fractionation with different adsorbents, PAHs were characterized using four different analytical techniques as summarized in Table 2 and Figure 2: gas chromatography/mass spectrometry (GC-MS), gas chromatography/high resolution mass spectrometry (GC-HRMS), high performance liquid chromatography/fluorescence detector (HPLC–FLD), and gas chromatography coupled to tandem mass spectrometry. A reversed-phase C18 column was used in the HPLC and three GC stationary phases of different selectivity, e.g. the 5 % phenyl-substituted methylpolysyloxane phase, the 50% phenyl-substituted methylpolysiloxane phase and the Select PAH Agilent phase.

TABLE 2. INSTRUMENTAL TECHNIQUES

Method code	**Instrumental technique**
GC-MS	Gas chromatography/mass spectrometry
GC-HRMS	Gas chromatography/high resolution mass spectrometry
HPLC–FLD	High performance liquid chromatography/fluorescence detector
GC-MS/MS	Gas chromatography coupled to tandem mass spectrometry

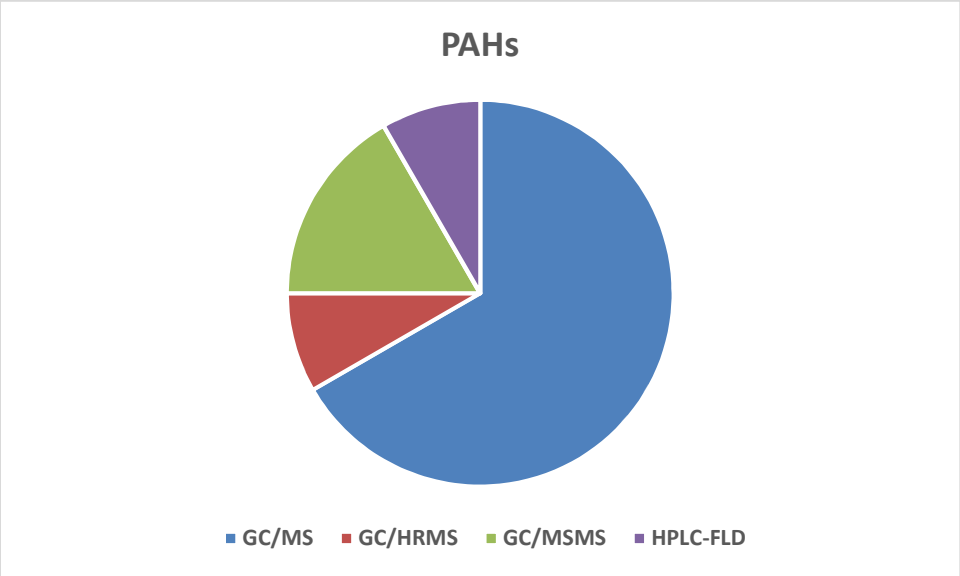

Fig. 2. Analytical methods used for the characterization of PAHs in the IAEA-477 sediment sample. Abbreviations used to describe the instrumental techniques are given in Table 1.

2.5. MOISTURE CONTENT

Participants were required to determine the moisture content of the lyophilized material received by drying an aliquot to a constant mass at 105°C. The moisture content of the lyophilized material, as the average of the mean value of 9 participants was found to be 0.9 ± 0.4%.

3. RESULTS AND DISCUSSION

3.1. RESULTS OF THE HOMOGENEITY STUDY

3.1.1. Between-unit and within-unit homogeneity

To assess the uncertainty contribution related to the inhomogeneity, 10 bottle units (about 3% of the total batch) of sediment samples were analysed for selected PAHs under repeatability conditions. Each bottle unit was extracted and analysed in duplicate, resulting in 2 independent data values by bottle unit.

Grubbs-tests at 95% and 99% confidence levels were performed to identify potentially outlying individual results as well as outlying bottle means. Few individual results and unit means were detected as outliers at 95% and 99% confidence levels. One technical reason for outlying results was identified for the high molecular weight (HMW) PAHs due to a blockage of a SPE cleanup column, which required the use of the vacuum pump to collect the HMW PAHs. In this context, the individual results of the outlying bottle means were rejected, whereas all other individual results following a normal distribution, were retained for evaluating the between-unit homogeneity.

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} using Eq. (1) and also between-units standard deviation s_{bb} ,using Eq. (2):

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

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

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. [5] through Eq. (3):

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

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 3, the between-unit variations (s_{bb} and u_{bb}*) for most of the selected PAHs were between 2.1 and 5% small enough to ensure the homogeneity of the material at 5 g sample size. Only benzo[e]pyrene exhibited a relatively higher value (7.8%), probably related to the higher measurement variability of this HMW PAH at such low concentration level. The within-unit variation (s_{wb}) derived from the ANOVA calculation was higher than the typical method repeatability (s_{meas}: 1-5%) derived from the analyses of six replicates from the same bottle ($s_{mes} = \frac{s}{\sqrt{6}}$), and also higher than the average s_{wbp} derived from the replicate data provided by the participant laboratories. Taking in account the close agreement among the replicate analysis provided by the laboratory participants, we might conclude that the within-unit homogeneity fits the purpose of this CRM.

The uncertainty u_{hom} associated with inhomogeneity of the material was estimated according to the ISO Guide 35 [1] by Eq. 4:

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

where the u_{wb} was derived from the participant laboratories data and the u_{bb} was taken as the maximum values of the between-unit variations (s_{bb} and u_{bb}*). As it is presented in Table 3, the uncertainty contribution related to inhomogeneity was estimated to range from 3.3 to 8.4 %, and, thus, we set the uncertainty associated with inhomogeneity at 8 % for all PAH analytes.

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

	ANOVA			METHOD	PART.	
Compounds	s_{wb}	s_{bb}	u_{bb}*	s_{meas} (%)	s_{wbp}	u_{hom}
	%	%	%	%	%	%
Napthalene	8.3	-	4.1	4	4.2±2.0	5.8
Phenanthrene	5.1	5	2.5	3	2.6±1.7	5.6
Pyrene	5.5	2.1	2.7	1	2.4±1.3	3.6
Chrysene+triphenylene	5.1	-	2.5	1	2.1±1.0	3.3
Benzo[*e*]Pyrene	15.7	-	7.8	5	3.0±1.9	8.4
Perylene	6.6	3.4	3.3	5	3.1±1.8	4.6

3.2. STABILITY STUDY

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

As previous results on PAHs stability studies did not show any significant trend of degradation over the timeframe at different temperatures: +20°C and +40°C [6], no special precautions regarding temperature control during transport was taken. This approach is supported by the chemical nature of the PAHs which owe a high chemical stability and persistence. Therefore, no additional uncertainty with respect to instability due to transport was taken into account and the uncertainty associated with short term stability under transport conditions is taken as zero.

3.2.1. Long-term stability study

Long-term stability evaluation aims to determine if the certified values of the analyte(s) remain valid during the lifetime of the certified reference material. Judging from similar materials issued from our IAEA laboratory and other organizations such as NIST, the expiry date of the CRMs was given to be 5-10 years after the date of certification if they were stored at temperatures less than 25-30°C and away from direct sunlight [7, 8]. As no measurements of long-term instability has been performed in this CRM, the uncertainty for long-term stability has been set as the intrinsic variability of the method $s_{meas,}$ which was set at 3% (phenantrene from Table 3). This is based on the experience on previous IAEA organic reference material where changes for long-term stability have not been detected, and therefore the uncertainty associated to the long-term stability is considered lower than the variability of the method. The CRM will be monitored at regular intervals and changes, which may occur, will be reflected in an updated certificate of analysis provided to the users.

3.3 DETERMINATION OF ASSIGNED VALUES AND UNCERTAINTIES

The data provided by the participant laboratories was first checked for their validity based on the fully documented method which it was tested by using an appropriated CRM for PAHs or participating in interlaboratory comparisons. Values given as below limit of detection or below limit of quantification were excluded for the statistical evaluation, excepting the values of naphthalene from laboratory 3 and anthracene from laboratories 1 and 3, due to the close values to the other reported values. Also, all data set of Acenapthene and Dibenzo[*a,h*]anthracene were retained due to the consistent dataset. Based on this criterion, the following datasets were rejected:

Lab. 1: 1-Methylnaphthalene, 2-Methylnaphthalene and Naphthalene

Lab 2: 1-Methylnaphthalenes, 2-Methylnaphthalene

Most of the participating laboratories applying the GC used the common 5% phenylmethylpolysiloxane phase where several important PAH isomers are not completely resolved, i.e. Chrysene and Triphenylene, Benzo[b]fluoranthene and Benzo[*j*]fluoranthene. Therefore, for those laboratories that achieved the separation of these isomers by using a 50% phenylmehtylpolysiloxane or a Select PAH column (lab 1 and lab 6), or using a brand new 5% phenylmehtylpolysiloxane (lab 7), their individual values were combined and included in the

final data set for the statistical evaluation as Chrysene (+Triphenylene) and Benzo[*b+j*]fluoranthene.

The characterization datasets accepted based on the technical reasons resulted in 2 to 12 measurement results for the target PAH compounds. As each participant used different extraction techniques/solvents followed by their own fractionation procedures and GC or HPLC separation, the data set is expected to provide a certain scatter. Therefore, different statistical tests were carried out to confirm that the single values provided by the participant laboratories follow a common mother distribution and are compliant with the certification requirements. 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. All data sets were normally distributed, and outliers were found for Acenapthylene (lab 6 and 7), C2-Phen/Anth (lab 10 and 13), C3-Chrysenes (lab. 13), Benzo[*b+j*]fluoranthene (lab 6 and 10) and Dibenzo[*a,h*]anthracene (lab 6 and 7). All data were retained for statistical analysis, except for the 2 outliers of Benzo[*b+j*]fluoranthene and the outlier at 99% of acenaphthylene.

The medians, unweighted mean of the means and robust mean were calculated and compared (Table 4). No significant differences were observed for the major PAHs compounds and the reference values obtained with the unweighted mean of the means was considered the most reliable estimates of the property values of the selected PAHs.

TABLE 4. COMPARISON OF DIFFERENT MEANS

	No. Results accepted	Mean of the means µg Kg^{-1}	Median µg Kg^{-1}	Robust mean µg Kg^{-1}	outliers 95%	outliers 99%
Naphthalene	9	4.50	4.34	4.42	0	0
2-Methylnaphthalene	5	2.42	2.50	2.44	0	0
1-Methylnaphthalene	5	1.41	1.44	1.41	0	0
C2- Naphthalenes	6	8.03	6.67	7.66	0	0
C3- Naphthalenes	6	5.16	5.19	5.05	0	0
Biphenyl	3	2.00	2.10	2.00	0	0
Acenaphthylene	9	0.71	0.71	0.71	1	1
Fluorene	10	0.80	0.64	0.68	0	0
Acenaphthene	8	0.33	0.24	0.26	0	0
C1-Fluorenes	3	2.74	2.20	2.28	0	0
C2-Fluorenes	3	3.76	3.09	3.22	0	0
C3-Fluorenes	3	4.29	5.45	5.24	0	0
Dibenzothiophene	6	0.55	0.52	0.54	0	0
C1-Dibenzothiophenes	4	2.49	2.05	2.17	0	0
C2-Dibenzothiophenes	4	7.62	6.31	7.26	0	0
C3-Dibenzothiophenes	3	7.53	6.89	7.53	0	0
Phenanthrene	12	4.55	4.38	4.45	0	0
Anthracene	10	1.31	1.29	1.26	0	0
1-Methylphenanthrene	5	1.51	1.30	1.47	0	0
2-Methylphenanthrene	6	2.41	2.47	2.40	0	0
C1- Phen/Anth	6	7.58	7.37	7.58	0	0
C2- Phen/Anth	6	8.58	8.37	8.41	2	0
C3- Phen/Anth	6	4.91	4.62	4.91	0	0
C4- Phen/Anth	4	2.41	2.44	2.42	0	0
Fluoranthene	12	6.65	6.22	6.61	0	0
Pyrene	12	6.17	5.63	6.15	0	0
1-MetylPyrene	2	0.71	0.71		0	0
C1-Fluoranthenes/pyrenes	5	4.42	4.40	4.45	0	0
C2-Fluoranthenes/pyrenes	4	3.49	3.13	3.35	0	0
C3-Fluoranthenes/pyrenes	2	2.10	2.10		0	0
Benz[*a*]anthracene	12	2.43	2.29	2.40	0	0
Chrysene	4	4.00	3.37	3.66	0	0
Chrysene (+ Triphenylene)	10	3.53	3.21	3.37	0	0
Triphenylene	2	0.81	0.81		0	0
C1-Chrysenes	5	4.13	4.60	4.28	0	0
C2-Chrysenes	5	3.52	3.90	3.52	0	0
C3-Chrysenes	3	3.18	1.55	1.58	1	1
Benzo[*b*]fluoranthene	4	5.76	4.43	4.75	0	0
Benzo[*j*]fluoranthene	3	2.16	1.59	1.87	0	0
Benzo[*b+j*]fluoranthene	9	5.00	4.68	4.98	2	1

Table 4. (cont.)

	No. Results	Mean of the means µg Kg^{-1}	Median µg Kg^{-1}	Robust mean µg Kg^{-1}	outliers 95%	outliers 99%
Benzo[*k*]fluoranthene	10	2.09	1.75	2.04	0	0
Benzo[*a*]fluoranthene	2	0.70	0.70		0	0
Benzo[*e*]pyrene	9	3.95	2.95	3.22	0	0
Benzo[*a*]pyrene	12	2.87	2.81	2.80	0	0
Indeno[*1,2,3-c,d*]pyrene	12	2.65	2.51	2.65	0	0
Dibenz[*a,h*]anthracene	9	0.43	0.43	0.42	2	0
Benzo[*g,h,i*]perylene	11	2.98	3.04	2.98	0	0
Perylene	9	35.64	31.57	33.85	0	0

The uncertainties associated with the assigned property values were evaluated according to ISO Guide 35 [1]. The relative combined uncertainty of the assigned property value of the CRM involved combining the standard uncertainties associated with the characterization *(*u_{char}), homogeneity (u_{hom}), short term stability (u_{short}) and long-term stability (u_{stab}). Because the uncertainty component derived from the short-term stability was insignificant and assumed to be zero, the final expanded uncertainty was a combination of the other three different contributions using the law of propagation of uncertainty as shown in Eq. (5):

$$U = k \times \sqrt{u_{char}^2 + u_{stab}^2 + u_{hom}^2} \qquad (5)$$

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

u_{hom} was evaluated as described in section 3.1.1, and set at 8 % for all PAH analytes

u_{stab} was evaluated as described in section 3.2.1 and set at 3% for all PAH analytes.

u_{char} was evaluated as described in ISO 35 [1] using Eq. (6):

$$u_{char} = \frac{s}{\sqrt{p}} \qquad (6)$$

Where: *s* is the standard deviation and p is the number of data sets accepted.

The final assigned values derived by the mean of the means are shown in Table 5 together with their individual uncertainty associated to the characterization and final uncertainty budget.

TABLE 5. MEAN OF THE MEAN, CHARACTERIZATION UNCERTAINTY AND TOTAL UNCERTAINTY

	No. Results	Mean of the means µg Kg^{-1}	u_{char} %	*U* total (*k*=2) %
Naphthalene	9	4.50	18	40
2-Methylnaphthalene	5	2.42	12	30
1-Methylnaphthalene	5	1.41	13	32
C2- Naphthalenes	6	8.03	23	50
C3- Naphthalenes	6	5.16	20	44
Biphenyl	3	2.00	16	36
Acenaphthylene	9	0.71	14	33
Fluorene	10	0.80	18	40
Acenaphthene	8	0.33	31	64
C1-Fluorenes	3	2.74	24	50
C2-Fluorenes	3	3.76	26	55
C3-Fluorenes	3	4.29	33	68
Dibenzothiophene	6	0.55	13	32
C1-Dibenzothiophenes	4	2.49	27	56
C2-Dibenzothiophenes	4	7.62	37	77
C3-Dibenzothiophenes	3	7.53	23	48
Phenanthrene	12	4.55	12	29
Anthracene	10	1.31	16	36
1-Methylphenanthrene	5	1.51	18	39
2-Methylphenanthrene	6	2.41	15	35
C1- Phen/Anth	6	7.58	13	30
C2- Phen/Anth	6	8.58	16	37
C3- Phen/Anth	6	4.91	21	46
C4- Phen/Anth	4	2.41	10	27
Fluoranthene	12	6.65	9.2	25
Pyrene	12	6.17	9.4	25
1-MetylPyrene	2	0.71	--	--
C1-Fluoranthenes/pyrenes	5	4.42	23	49
C2-Fluoranthenes/pyrenes	4	3.49	33	69
C3-Fluoranthenes/pyrenes	2	2.10	--	--
Benz[*a*]anthracene	12	2.43	7.7	23
Chrysene	4	4.00	32	67
Chrysene (+ Triphenylene)	10	3.53	8.7	24
Triphenylene	2	0.81	--	--
C1-Chrysenes	5	4.13	13	32
C2-Chrysenes	5	3.52	19	42
C3-Chrysenes	3	3.18	52	105
Benzo[*b*]fluoranthene	4	5.76	32	67
Benzo[*j*]fluoranthene	3	2.16	43	88
Benzo[*b*+*j*]fluoranthene	11	5.00	8.3	24
Benzo[*k*]fluoranthene	10	2.09	11	28

Table 5. (cont.)

	No. Results	Mean of the means µg Kg^{-1}	u_{char} %	U total (k=2) %
Benzo[*a*]fluoranthene	2	0.70	--	--
Benzo[*e*]pyrene	9	3.95	14	33
Benzo[*a*]pyrene	12	2.87	9.0	25
Indeno[*1,2,3-c,d*]pyrene	12	2.65	8.9	25
Dibenz[*a,h*]anthracene	9	0.43	9.2	25
Benzo[*g,h,i*]perylene	11	2.98	7.0	22
Perylene	9	35.64	22	47

The results for the mass fractions of the PAHs as reported by the participants in this characterization are presented in Appendix I and II. The laboratory means are plotted together with the mean of the means denoted by a bold line, while the dashed lines represent mean ± expanded uncertainty (k=2) in all figures (as calculated in Eq. 5). The error bars represent the expanded uncertainty of the participant 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. In this context, it should be noted that this expanded uncertainty as a measurement of the experimental standard deviation of the mean only reflect the repeatability of the participant method, neglecting other key contributors to the uncertainty as it is described within the GUM guide [2]. Reasonable and comparability estimates of uncertainty in organic contaminant analysis is still a priority to be achieved in accordance to the GUM guide.

As shown previously in Table 1, methods using different independent analytical techniques (GC-MS; GC-MS/MS, GC-HRMS and HPLC-FLD) with different extraction and purification procedures 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 and its relative expanded uncertainties was less than 40 % of the assigned value. These criteria were fulfilled for 19 PAHs: Naphthalene, 2-Methylnaphthalene, 1-Methylnaphthalene, Phenantrene, Anthracene, 1-Methylphenanthrene, 2-Methylphenanthrene, C1-Phenanthrenes/ Anthracenes, Fluoranthene, Pyrene, Benz[*a*]anthracene, Chrysene (+Triphenylene), C1-Chrysenes, Benzo[*b+j*]fluoranthene; Benzo[*k*]fluoranthene, Benzo[*e*]pyrene, Benzo[*a*]pyrene, Indeno[*1,2,3-c,d*]pyrene, Benzo[*g,h,i*]perylene. The certified values are presented in Table 6, together with their expanded uncertainty.

Compounds that did not fulfill the criteria of certification are considered information values. They include C2-Naphthalenes, C3-Naphthalenes, Biphenyl, Acenaphthylene, Fluorene,

Acenaphthene, C1-Fluorenes, C2-Fluorenes, C3-Fluorenes, Dibenzothiophene, C1-Dibenzothiophenes, C2-Dibenzothiophenes, C3-Dibenzothiophenes, C2-Phen/Anth, C3-Phen/Anth, C4-Phen/Anth, 1-Metylpyrene, C1-Fluoranthenes/pyrenes, C2-Fluoranthenes/pyrenes, C3-Fluoranthenes/pyrenes, Triphenylene, C2-Chrysenes, C3-Chrysenes, Benzo[*b*]fluoranthene, Benzo[*j*]fluoranthene, Benzo[*a*]fluoranthene, Dibenz[*a,h*]anthracene, Perylene. Table 7, shows the information values for PAHs together with the expanded uncertainty for the compounds that could be calculated.

TABLE 6. CERTIFED VALUES FOR PAHS MASS FRACTIONS AND THEIR EXPANDED UNCERTAINTY (*k=2*) IN THE IAEA-477 SEDIMENT SAMPLE

Compound	**Unit**	**Certified value**[1]	***U* (*k*=2)** [2]
Naphthalene	µg kg^{-1}	4.5	1.8
2-Methylnaphthalene	µg kg^{-1}	2.4	0.7
1-Methylnaphthalene	µg kg^{-1}	1.4	0.5
Phenanthrene	µg kg^{-1}	4.6	1.3
Anthracene	µg kg^{-1}	1.3	0.5
1-Methylphenanthrene	µg kg^{-1}	1.5	0.6
2-Methylphenanthrene	µg kg^{-1}	2.4	0.9
C1- Phen/Anth	µg kg^{-1}	7.6	2.3
Fluoranthene	µg kg^{-1}	6.6	1.7
Pyrene	µg kg^{-1}	6.2	1.6
Benz[*a*]anthracene	µg kg^{-1}	2.4	0.6
Chrysene (+ Triphenylene)	µg kg^{-1}	3.5	0.9
C1-Chrysenes	µg kg^{-1}	4.1	1.3
Benzo[*b+j*]fluoranthene	µg kg^{-1}	5.0	1.2
Benzo[*k*]fluoranthene	µg kg^{-1}	2.1	0.6
Benzo[*e*]pyrene	µg kg^{-1}	3.9	1.3
Benzo[*a*]pyrene	µg kg^{-1}	2.9	0.7
Indeno[*1,2,3-c,d*]pyrene	µg kg^{-1}	2.6	0.7
Benzo[*g,h,i*]perylene	µg kg^{-1}	3.0	0.7

[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 7. INFORMATION VALUES FOR PAHs MASS FRACTIONS AND THEIR EXPANDED UNCERTAINTY (*k=2*) IN THE IAEA-477 SEDIMENT SAMPLE

Compound	**Unit**	**Information value[1]**	**U (*k*=2) [2]**
C2- Naphthalenes	μg kg^{-1}	8.0	4.0
C3- Naphthalenes	μg kg^{-1}	5.2	2.3
Biphenyl	μg kg^{-1}	2.0	0.7
Acenaphthylene	μg kg^{-1}	0.7	0.3
Fluorene	μg kg^{-1}	0.8	0.3
Acenaphthene	μg kg^{-1}	0.3	0.2
C1-Fluorenes	μg kg^{-1}	2.7	1.4
C2-Fluorenes	μg kg^{-1}	3.8	2.1
C3-Fluorenes	μg kg^{-1}	4.3	2.9
Dibenzothiophene	μg kg^{-1}	0.6	0.2
C1-Dibenzothiophenes	μg kg^{-1}	2.5	1.4
C2-Dibenzothiophenes	μg kg^{-1}	7.6	5.9
C3-Dibenzothiophenes	μg kg^{-1}	7.5	3.7
C2- Phen/Anth	μg kg^{-1}	8.6	3.2
C3- Phen/Anth	μg kg^{-1}	4.9	2.3
C4- Phen/Anth	μg kg^{-1}	2.4	0.6
1-Metylpyrene	μg kg^{-1}	0.7	--
C1-Fluoranthenes/pyrenes	μg kg^{-1}	4.4	2.2
C2-Fluoranthenes/pyrenes	μg kg^{-1}	3.5	2.4
C3-Fluoranthenes/pyrenes	μg kg^{-1}	2.1	--
Chrysene	μg kg^{-1}	4.0	2.7
Triphenylene	μg kg^{-1}	0.8	--
C2-Chrysenes	μg kg^{-1}	3.5	1.5
C3-Chrysenes	μg kg^{-1}	1.6	--
Benzo[*b*]fluoranthene	μg kg^{-1}	5.8	3.8
Benzo[*j*]fluoranthene	μg kg^{-1}	2.2	1.9
Benzo[*a*]fluoranthene	μg kg^{-1}	0.7	--
Dibenz[*a,h*]anthracene	μg kg^{-1}	0.4	0.1
Perylene	μg kg^{-1}	35.6	16.9

[1] The value is the mean of the mean of the accepted sets of data, except for C3-Chrysenes where the value is the robust mean. Each set of data is 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 [9]. The information on the calibration standards, surrogate standards and matrix standard reference materials (CRMs) standards are summarized in Appendix III. The methods used by all participating laboratories were validated by using matrix standard reference materials (CRMs) from NIST (SRM1941b, SRM1944), IAEA (IAEA-408, IAEA-459, IAEA-383) and materials characterized by QUASIMEME proficiency tests (QPH094MS). Their reported values are based on calibration standard solutions of known purity, issued by accredited commercial companies with documented unbroken chain of calibrations. Consequently, 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 measurand.

Commutability is a property of an RM, demonstrated by the closeness of agreement between the relation among the measurement results for a stated quantity in this material, obtained according to two given measurement procedures, and the relation obtained among the measurement results for other specified materials [10].

A material is said to be commutable when the measurand in the routine test samples behaves similar as in the CRM with respect to the different measurement procedures. This implies that the application of the procedures to the CRM would produce the same quantitative value as normal routine samples containing the same concentration of the measurand. In this respect, IAEA-477 is a natural marine sediment sample and their analytical behaviour should be the same as for a routine sample of dried sediment samples. The agreement between the data acquired with different analytical procedures for the IAEA-477 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 two different analytical techniques has allowed the assignment of certified concentrations for 19 PAHs following the recommendation of ISO Guide 35. The extensive characterization with very low concentration levels and associated uncertainties will make CRM 477 a valuable sediment reference material for use in the validation of analytical methods for the determination of polycyclic aromatic hydrocarbons included as priority substances (PSs), within the environmental monitoring programmes.

APPENDIX I: CHARACTERIZATION RESULTS: ASSIGNED VALUES OF PAHS

TABLE 8. NAPHTHALENE RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1}):

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
2	2.93	0.23	0.500	GC/MSMS	IAEA-408
3	1.23	0.22	0.500	HPLC-FLD	QPH094MS
4	4.95	0.19	0.950	GC/MS	NIST 1941b
5	1.97	0.05	0.100	GC/MS	IAEA 459
6	6.53	0.64	0.500	GC/MS	NIST 1941b
9	4.34	0.40	0.128	GC/MSMS	IAEA-459
10	3.25	0.18	0.160	GC/MS	NIST 1941b
11	8.82	0.79	2.000	GC/MS	IAEA-383
13	6.49	0.50	0.050	GC/MS	IAEA-459
Results not used for the assignment value					
1	0.87	0.09	5.000	GC/MS	NIST 1941b
7	<4.9		7.000	GC/HRMS	IAEA-459

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

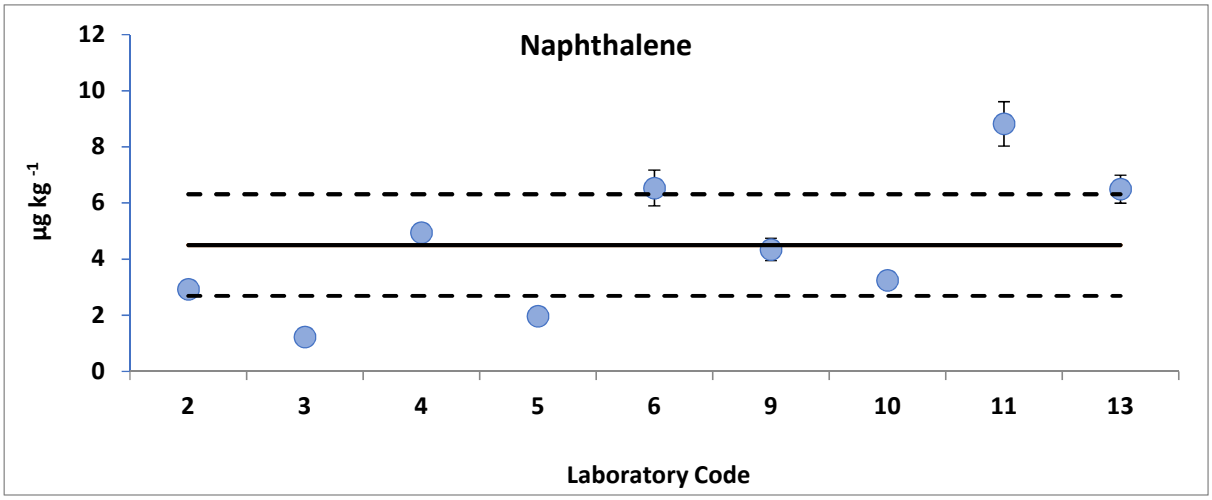

*FIG. 3 Laboratory results used to calculate the assignment mass fraction of Naphthalene in IAEA-477 (*µg kg^{-1}).

TABLE 9. 2-METHYLNAPHTHALENE RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
4	2.74	0.15	0.110	GC/MS	NIST 1941b
5	1.41	0.07	0.050	GC/MS	IAEA 459
6	2.28	0.28	0.500	GC/MS	NIST 1941b
10	2.50	0.30	0.080	GC/MS	NIST 1941b
13	3.18	0.39	0.015	GC/MS	IAEA-459
			Results not used for the assignment value		
1	0.55	0.07	0.700	GC/MS	NIST 1941b
2	0.83	0.07	0.170	GC/MSMS	IAEA-408
7	<3.9		0.130	GC/HRMS	IAEA-459

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

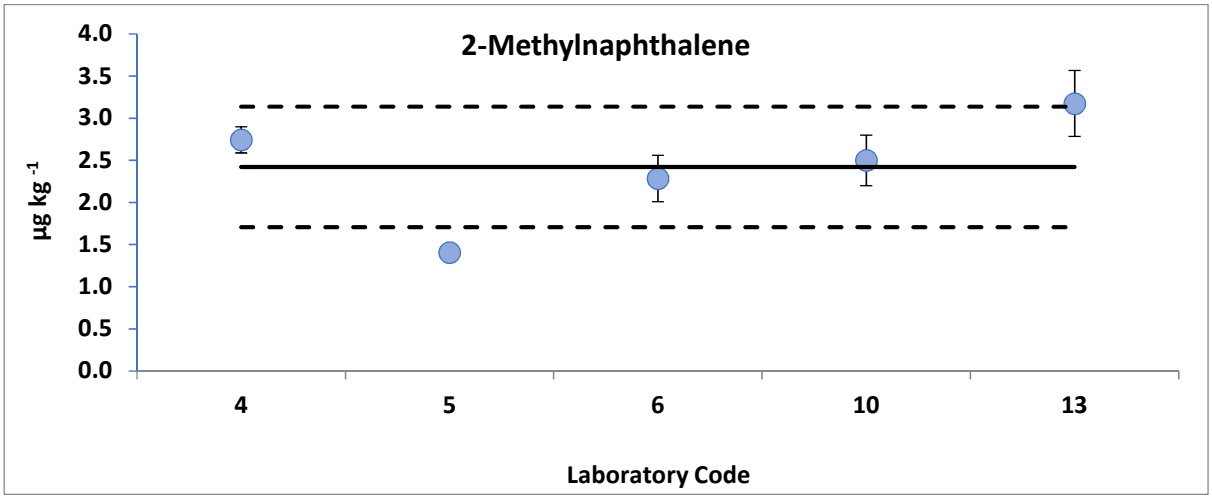

*FIG. 4. Laboratory results used to calculate the assignment mass fraction of 2-Methylnaphthalene in IAEA-477 (*µg kg^{-1}).

TABLE 10. 1-METHYLNAPHTHALENE RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
4	1.44	0.14	0.140	GC/MS	NIST 1941b
5	0.83	0.07	0.050	GC/MS	IAEA 459
6	1.17	0.21	0.500	GC/MS	NIST 1941b
10	1.92	0.16	0.060	GC/MS	IAEA-459
13	1.69	0.19	0.014	GC/MS	NIST 1941b
Results not used for the assignment value					
1	0.27	0.04	0.700	GC/MS	NIST 1941b
2	0.36	0.06	0.100	GC/MSMS	IAEA-408
7	<1.9		0.130	GC/HRMS	IAEA-459

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

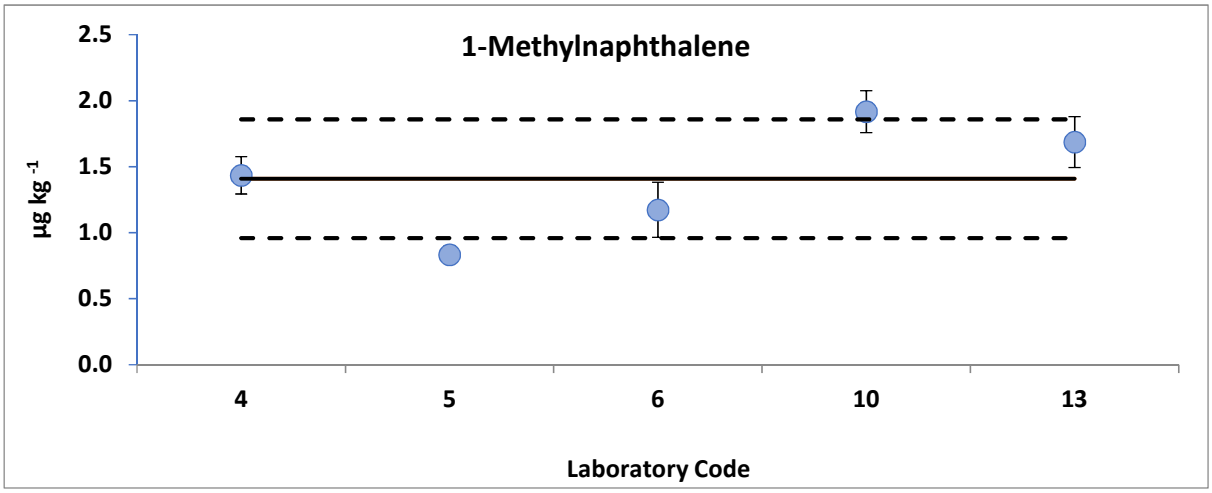

*FIG. 5. Laboratory results used to calculate the assignment mass fraction of 1-Methylnaphthalene in IAEA-477 (*µg kg^{-1}).

TABLE 11. PHENANTHRENE RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
1	2.85	0.14	0.700	GC/MS	NIST 1941b
2	5.41	0.10	0.100	GC/MSMS	IAEA-408
3	2.57	0.17	0.500	HPLC-FLD	QPH094MS
4	2.63	0.07	0.430	GC/MS	NIST 1941b
5	3.97	0.13	0.050	GC/MS	IAEA 459
6	8.37	0.64	0.500	GC/MS	NIST 1941b
7	2.72	0.06	0.020	GC/HRMS	IAEA-459
9	6.64	0.23	0.018	GC/MSMS	IAEA-459
10	4.80	0.05	0.090	GC/MS	NIST 1941b
11	3.18	0.42	0.400	GC/MS	IAEA-383
12	5.41	0.38	0.800	GC/MS	IAEA-383
13	6.04	0.42	0.014	GC/MS	IAEA-459

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

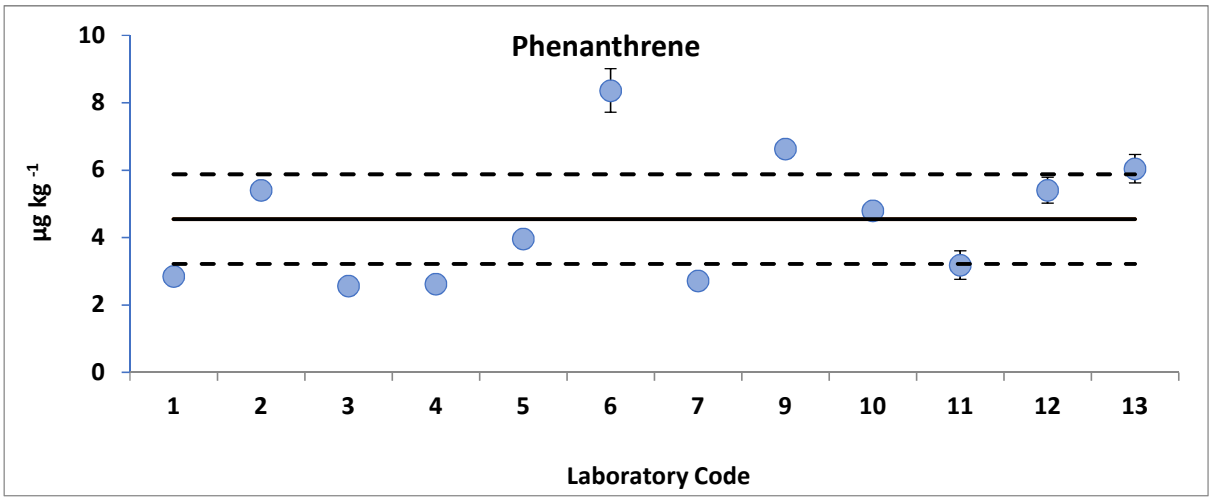

*FIG. 6. Laboratory results used to calculate the assignment mass fraction of Phenanthrene in IAEA-477 (*µg kg^{-1}*).*

TABLE 12. ANTHRACENE RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
1	0.72	0.05	0.700	GC/MS	NIST 1941b
2	1.41	0.04	0.120	GC/MSMS	IAEA-408
3	0.83	0.03	0.500	HPLC-FLD	QPH094MS
5	0.79	0.03	0.050	GC/MS	IAEA 459
6	2.68	0.18	0.500	GC/MS	NIST 1941b
7	0.56	0.04	0.020	GC/HRMS	IAEA-459
9	1.89	0.10	0.020	GC/MSMS	IAEA-459
10	1.28	0.08	0.090	GC/MS	NIST 1941b
11	1.67	0.16	0.400	GC/MS	IAEA-383
13	1.30	0.22	0.030	GC/MS	IAEA-459
Results not used for the assignment value					
4	<LOQ		0.280	GC/MS	NIST 1941b
12	<1.44		0.800	GC/MS	IAEA-383

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

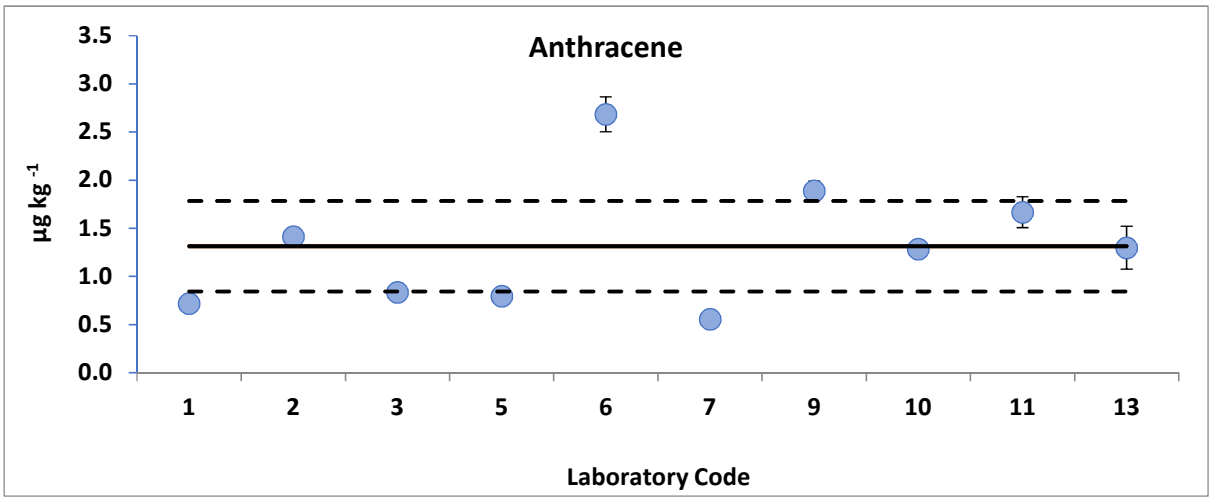

*FIG. 7. Laboratory results used to calculate the assignment mass fraction of Anthracene in IAEA-477 (*µg kg^{-1}*).*

TABLE 13. 1-METHYLPHENANTHRENE RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
1	1.14	0.05	0.700	GC/MS	NIST 1941b
4	1.30	0.10	0.290	GC/MS	NIST 1941b
6	2.37	0.08	0.500	GC/MS	NIST 1941b
7	0.89	0.05	0.020	GC/HRMS	IAEA-459
13	1.85	0.06	0.014	GC/MS	IAEA-459

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

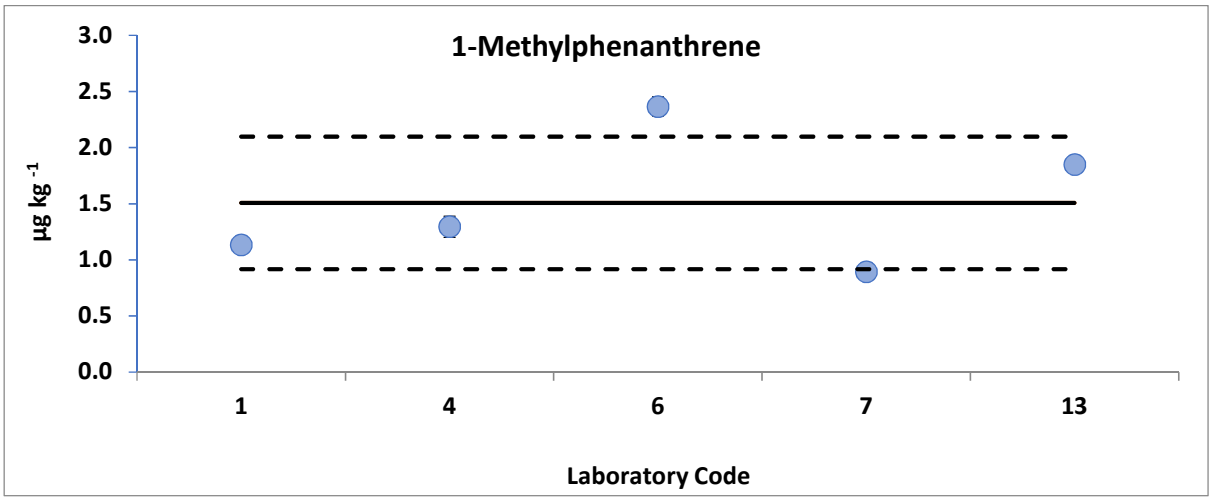

*FIG. 8. Laboratory results used to calculate the assignment mass fraction of 1-Methylphenanthrene in IAEA-477 (*µg kg^{-1}).

TABLE 14. 2-METHYLPHENANTHRENE RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
1	1.46	0.05	0.700	GC/MS	NIST 1941b
2	2.91	0.21	0.450	GC/MSMS	IAEA-408
4	2.06	0.15	0.110	GC/MS	NIST 1941b
6	3.70	0.31	0.500	GC/MS	NIST 1941b
7	1.43	0.07		GC/HRMS	IAEA-459
13	2.87	0.14	0.014	GC/MS	IAEA-459

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

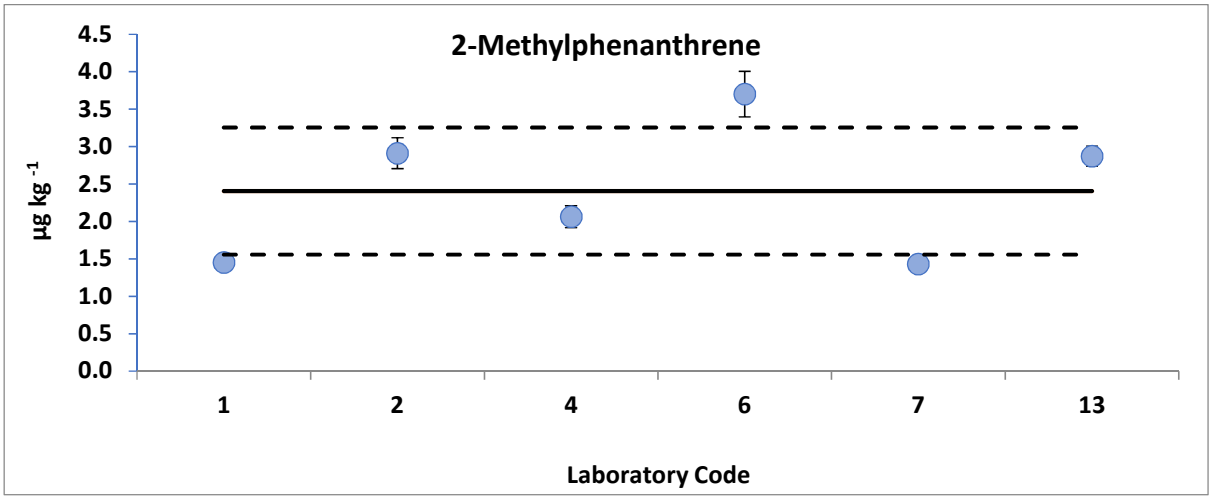

FIG. 9. Laboratory results used to calculate the assignment mass fraction of 2-Methylphenanthrene *in IAEA-477 (*µg kg^{-1}).

TABLE 15. C1- PHEN/ANTH RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
1	5.23	0.21	0.700	GC/MS	NIST 1941b
2	7.50	0.49	0.450	GC/MSMS	IAEA-408
4	7.24	0.13	0.910	GC/MS	NIST 1941b
7	4.93	0.17	0.080	GC/HRMS	IAEA-459
10	10.50	0.70	0.110	GC/MS	NIST 1941b
13	10.08	0.47	0.014	GC/MS	IAEA-459

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

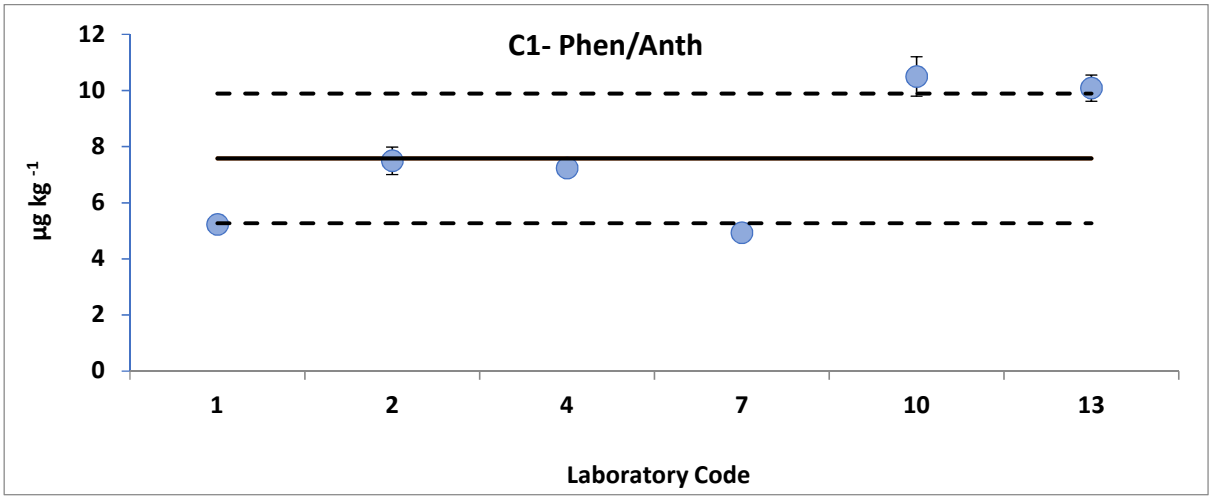

*FIG. 10. Laboratory results used to calculate the assignment mass fraction of C1-Phen/Anth in IAEA-477 (*µg kg^{-1}).

TABLE 16. FLUORANTHENE RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
1	4.95	0.21	0.300	GC/MS	NIST 1941b
2	7.88	0.14	0.110	GC/MSMS	IAEA-408
3	5.10	0.21	0.500	HPLC-FLD	QPH094MS
4	3.65	0.18	0.640	GC/MS	NIST 1941b
5	5.21	0.09	0.050	GC/MS	IAEA 459
6	10.25	0.88	0.500	GC/MS	NIST 1941b
7	4.72	0.11	0.010	GC/HRMS	IAEA-459
9	9.63	0.47	0.006	GC/MSMS	IAEA-459
10	7.23	0.10	0.080	GC/MS	NIST 1941b
11	7.88	0.44	0.400	GC/MS	IAEA-383
12	5.20	0.30	0.740	GC/MS	IAEA-383
13	8.16	0.32	0.024	GC/MS	IAEA-459

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

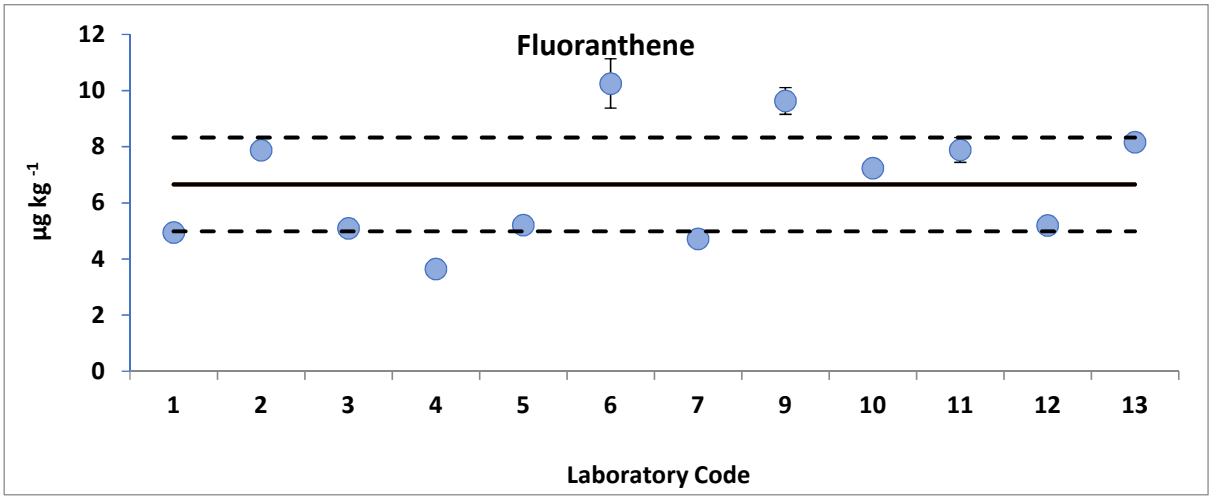

*FIG. 11. Laboratory results used to calculate the assignment mass fraction of Fluoranthene in IAEA-477 (*µg kg^{-1}).

TABLE 17. PYRENE RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
1	4.58	0.18	0.300	GC/MS	NIST 1941b
2	6.27	0.20	0.210	GC/MSMS	IAEA-408
3	4.47	0.20	0.500	HPLC-FLD	QPH094MS
4	3.94	0.17	0.460	GC/MS	NIST 1941b
5	4.28	0.16	0.050	GC/MS	IAEA 459
6	9.52	1.07	0.500	GC/MS	NIST 1941b
7	4.35	0.12	0.010	GC/HRMS	IAEA-459
9	8.98	0.48	0.006	GC/MSMS	IAEA-459
10	6.93	0.10	0.080	GC/MS	NIST 1941b
11	7.65	0.59	0.400	GC/MS	IAEA-383
12	5.00	0.29	0.950	GC/MS	IAEA-383
13	8.01	0.23	0.023	GC/MS	IAEA-459

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

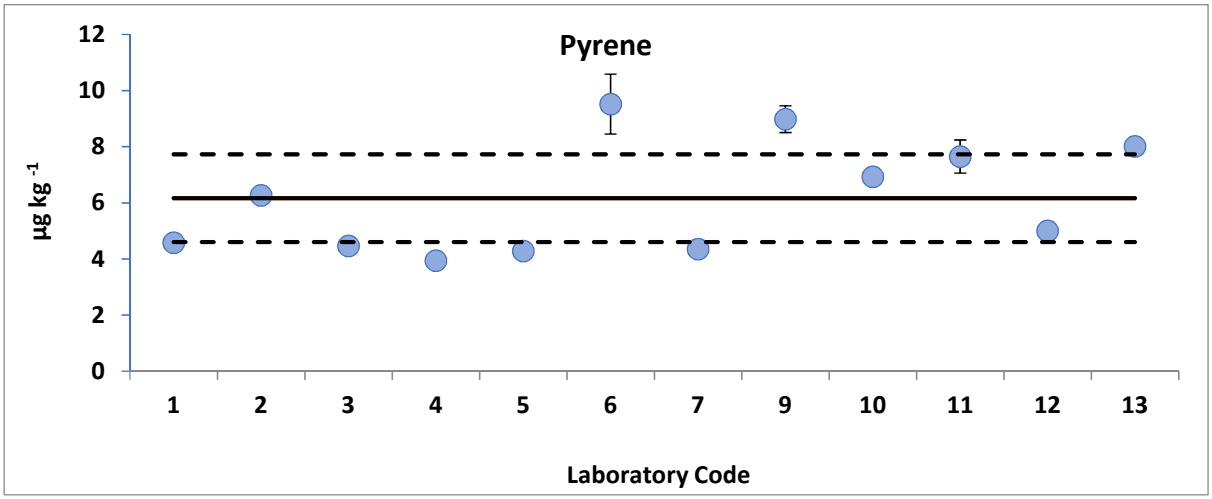

*FIG. 12. Laboratory results used to calculate the assignment mass fraction of Pyrene in IAEA-477 (*µg kg^{-1}).

TABLE 18. BENZ[*A*]ANTHRACENE RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
1	1.81	0.07	0.300	GC/MS	NIST 1941b
2	2.39	0.07	0.310	GC/MSMS	IAEA-408
3	1.47	0.10	0.500	HPLC-FLD	QPH094MS
4	1.83	0.11	0.430	GC/MS	NIST 1941b
5	2.13	0.11	0.100	GC/MS	IAEA 459
6	3.25	0.33	0.500	GC/MS	NIST 1941b
7	2.02	0.08	0.020	GC/HRMS	IAEA-459
9	2.74	0.09	0.008	GC/MSMS	IAEA-459
10	3.47	0.10	0.100	GC/MS	NIST 1941b
11	3.32	0.24	0.400	GC/MS	IAEA-383
12	2.18	0.16	0.230	GC/MS	IAEA-383
13	2.50	0.21	0.020	GC/MS	IAEA-459

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

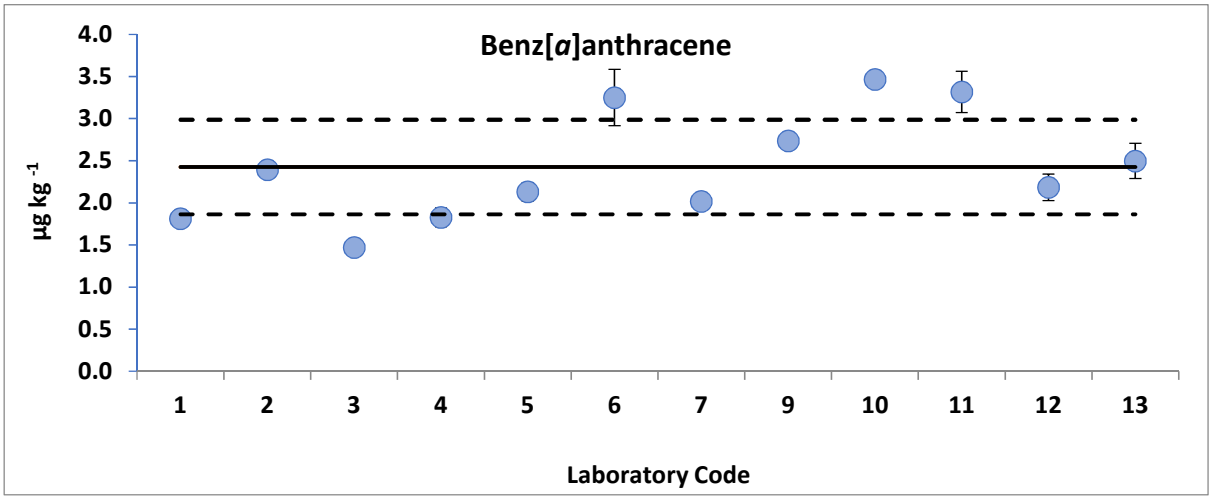

*FIG. 13. Laboratory results used to calculate the assignment mass fraction of Benz(a)anthracene in IAEA-477 (*µg kg^{-1}).

TABLE 19. CHRYSENE (+ TRIPHENYLENE) RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
1	5.07	0.18	0.300	GC/MS	NIST 1941b
2	3.72	0.12	0.080	GC/MSMS	IAEA-408
4	2.66	0.17	0.440	GC/MS	NIST 1941b
5	2.61	0.14	0.100	GC/MS	IAEA 459
7	3.29	0.12	0.020	GC-HRMS	IAEA-459
9	3.12	0.14	0.010	GC/MSMS	IAEA-459
10	5.15	0.07	0.090	GC/MS	NIST 1941b
11	2.65	0.18	0.400	GC/MS	IAEA-383
12	2.90	0.18	1.260	GC/MS	IAEA-383
13	4.16	0.12	0.011	GC/MS	IAEA-459

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

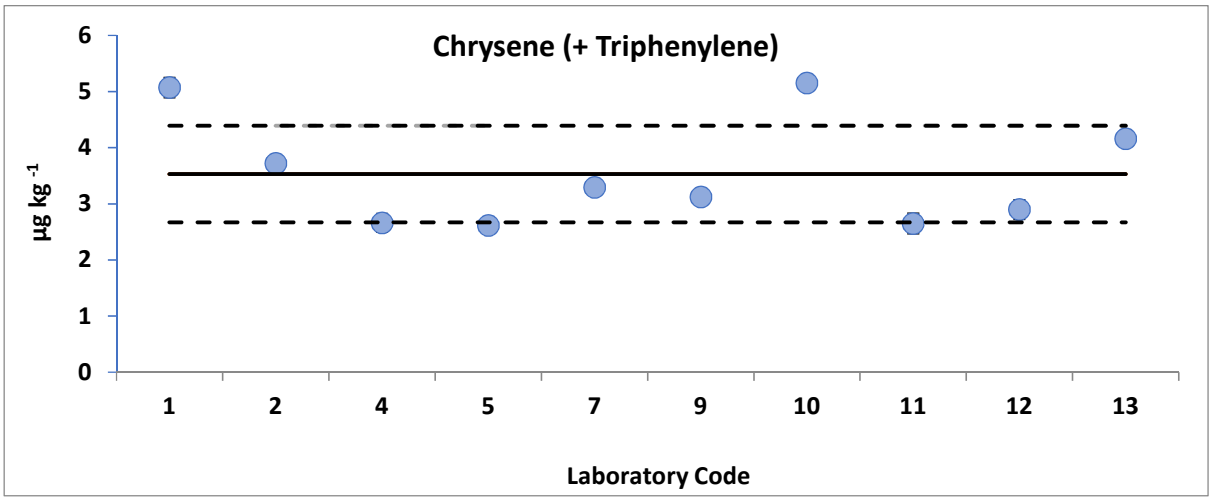

*FIG. 14. Laboratory results used to calculate the assignment mass fraction of Chrysene (+ Triphenylene) in IAEA-477 (*µg kg^{-1}*).*

TABLE 20. C1-CHRYSENES RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
1	3.35	0.16	0.300	GC/MS	NIST 1941b
4	2.35	0.15	0.170	GC/MS	NIST 1941b
7	5.27	0.37	0.360	GC/HRMS	IAEA-459
10	5.10	0.30	0.120	GC/MS	NIST 1941b
13	4.60	0.35	0.011	GC/MS	IAEA-459

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

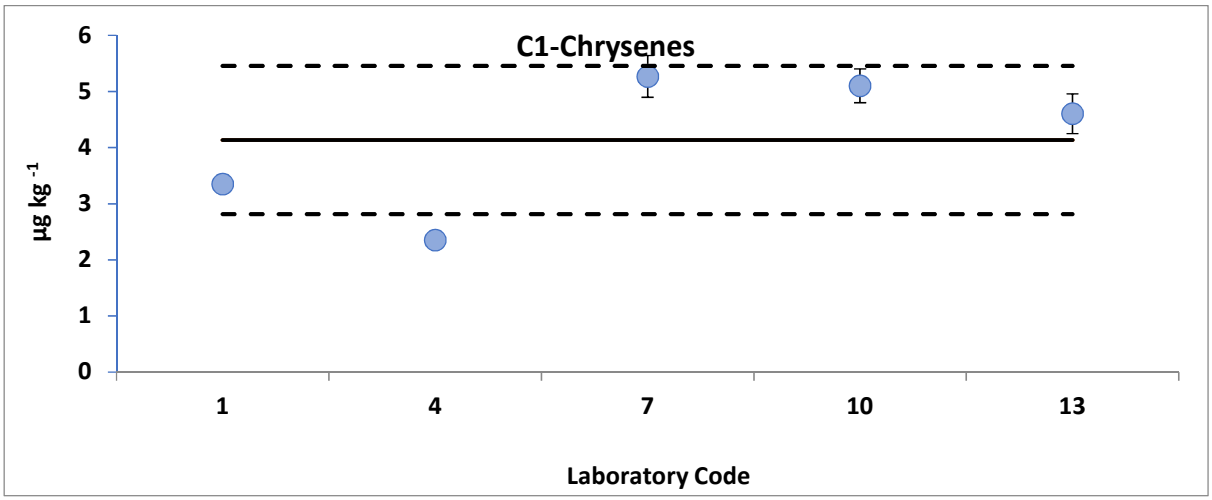

*FIG. 15. Laboratory results used to calculate the assignment mass fraction of C1-Chrysenes in IAEA-477 (*µg kg^{-1}).

TABLE 21. BENZO[B+J] FLUORANTHENE RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
1	4.68	0.16	0.700	GC/MS	NIST 1941b
2	6.60	0.15	0.310	GC/MSMS	IAEA-408
4	3.79	0.22	0.520	GC/MS	NIST 1941b
5	3.52	0.21	0.100	GC/MS	IAEA 459
7	4.32	0.14	0.010	GC/HRMS	IAEA-459
9	6.78	0.07	0.016	GC/MSMS	IAEA-459
11	4.10	0.21	0.400	GC/MS	IAEA-383
12	6.28	0.48	2.110	GC/MS	IAEA-383
13	4.93	0.80	0.018	GC/MS	IAEA-459
Results not used for the assignment value					
6	15.10	1.70	0.500	GC/MS	NIST 1941b
10	10.35	0.27	0.190	GC/MS	NIST 1941b

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

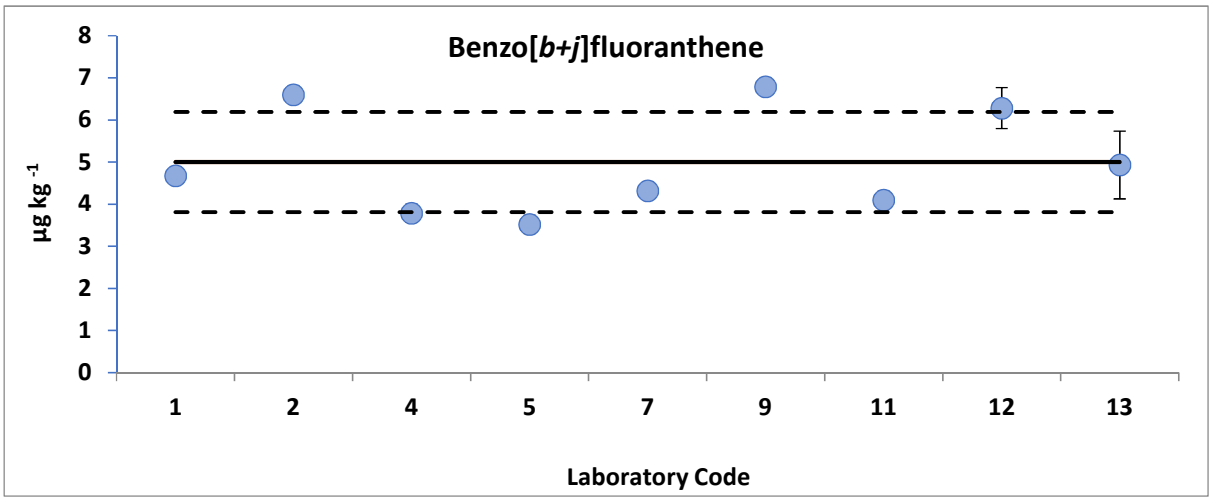

*FIG. 16. Laboratory results used to calculate the assignment mass fraction of Benzo(b+j) fluoranthene in IAEA-477 (*µg kg^{-1}*).*

TABLE 22. BENZO[K]FLUORANTHENE RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
1	1.40	0.12	0.700	GC/MS	NIST 1941b
2	1.80	0.06	0.300	GC/MSMS	IAEA-408
3	1.15	0.09	0.500	HPLC-FLD	QPH094MS
5	2.86	0.16	0.100	GC/MS	IAEA 459
6	3.15	0.31	0.500	GC/MS	NIST 1941b
7	1.70	0.05	0.010	GC/HRMS	IAEA-459
9	1.67	0.06	0.010	GC/MSMS	IAEA-459
10	2.97	0.12	0.180	GC/MS	NIST 1941b
11	1.47	0.13	0.400	GC/MS	IAEA-383
13	2.78	0.42	0.016	GC/MS	IAEA-459
Results not used for the assignment value					
4	<LOQ		0.770	GC/MS	NIST 1941b

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

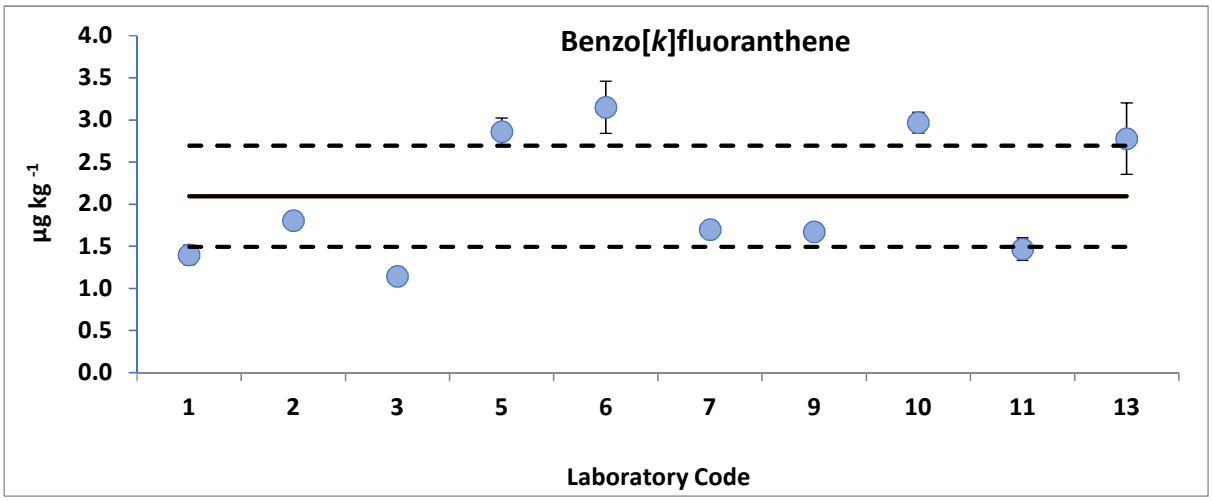

*FIG. 17. Laboratory results used to calculate the assignment mass fraction of Benzo(k)fluoranthene in IAEA-477 (*µg kg^{-1}).

TABLE 23. BENZO[E]PYRENE RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
1	2.56	0.08	0.300	GC/MS	NIST 1941b
2	3.87	0.07	0.350	GC/MSMS	IAEA-408
4	2.95	0.15	0.460	GC/MS	NIST 1941b
6	6.98	0.42	0.500	GC/MS	NIST 1941b
7	2.80	0.12	0.010	GC/HRMS	IAEA-459
10	6.13	0.18	0.210	GC/MS	NIST 1941b
11	2.75	0.37	0.400	GC/MS	IAEA-383
12	2.64	0.19	0.440	GC/MS	IAEA-383
13	4.82	0.52	0.012	GC/MS	IAEA-459

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

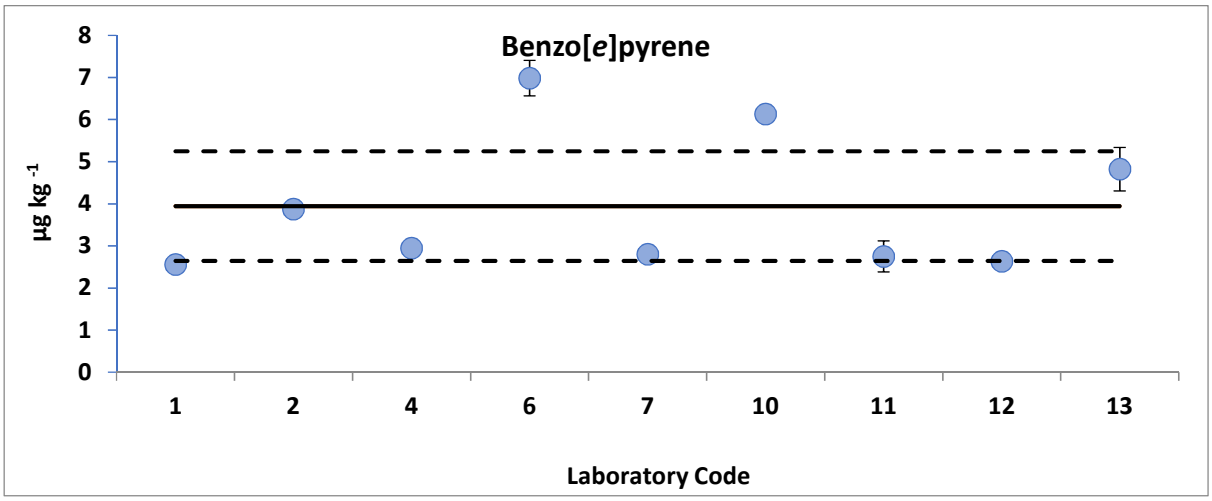

*FIG. 18. Laboratory results used to calculate the assignment mass fraction of Benzo(e)pyrene in IAEA-477 (*µg kg^{-1}).

TABLE 24. BENZO[A]PYRENE RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
1	2.39	0.10	0.700	GC/MS	NIST 1941b
2	3.13	0.05	0.350	GC/MSMS	IAEA-408
3	1.89	0.09	0.500	HPLC-FLD	QPH094MS
4	1.62	0.06	0.340	GC/MS	NIST 1941b
5	2.62	0.07	0.100	GC/MS	IAEA 459
6	4.45	0.41	0.500	GC/MS	NIST 1941b
7	2.55	0.10	0.010	GC/HRMS	IAEA-459
9	3.12	0.05	0.013	GC/MSMS	IAEA-459
10	4.32	0.11	0.230	GC/MS	NIST 1941b
11	1.92	0.14	0.400	GC/MS	IAEA-383
12	3.00	0.19	0.880	GC/MS	IAEA-383
13	3.42	0.64	0.019	GC/MS	IAEA-459

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

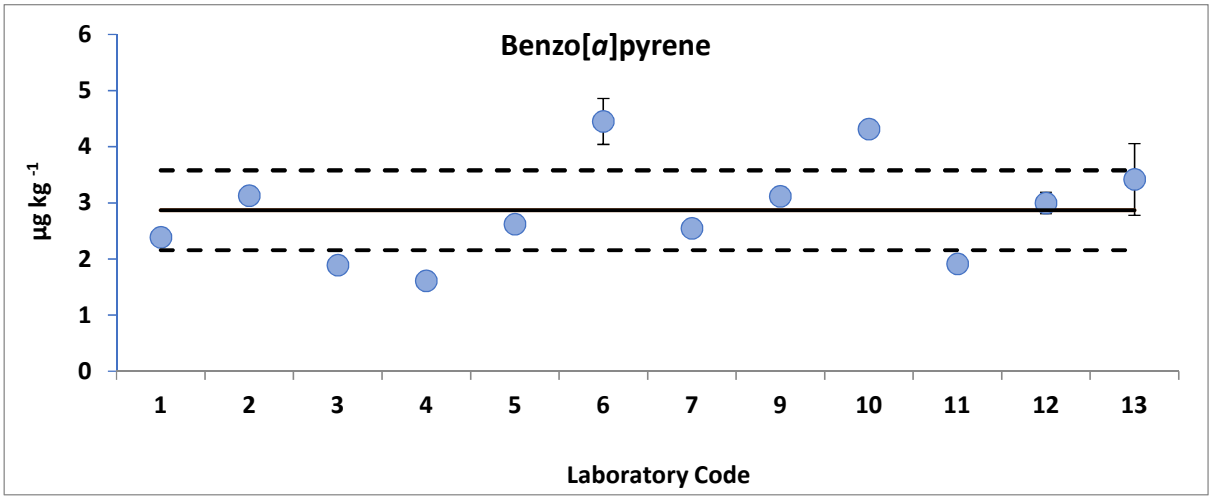

*FIG. 19. Laboratory results used to calculate the assignment mass fraction of Benzo(a)pyrene in IAEA-477 (*µg kg^{-1}).

TABLE 25. INDENO[1,2,3-C,D]PYRENE RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
1	2.43	0.11	0.700	GC/MS	NIST 1941b
2	3.32	0.07	0.330	GC/MSMS	IAEA-408
3	1.77	0.17	0.500	HPLC-FLD	QPH094MS
4	1.66	0.01	0.510	GC/MS	NIST 1941b
5	3.11	0.21	0.100	GC/MS	IAEA 459
6	3.77	0.48	0.500	GC/MS	NIST 1941b
7	3.12	0.17	0.020	GC/HRMS	IAEA-459
9	2.54	0.06	0.007	GC/MSMS	IAEA-459
10	2.48	0.10	0.150	GC/MS	NIST 1941b
11	1.67	0.25	0.400	GC/MS	IAEA-383
12	1.90	0.20	0.730	GC/MS	IAEA-383
13	4.02	0.51	0.038	GC/MS	IAEA-459

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

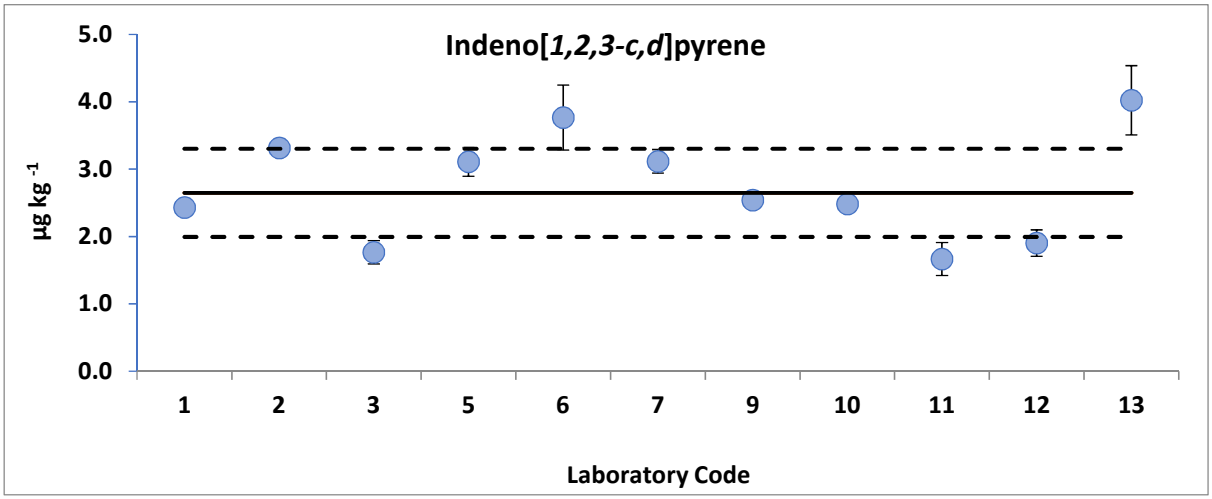

*FIG. 20. Laboratory results used to calculate the assignment mass fraction of Indeno[1,2,3-c,d]pyrene in IAEA-477 (*µg kg^{-1}).

TABLE 26. BENZO(G,H,I)PERYLENE RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
1	2.69	0.12	0.700	GC/MS	NIST 1941b
2	4.12	0.05	0.050	GC/MSMS	IAEA-408
3	2.44	0.09	0.500	HPLC-FLD	QPH094MS
4	1.89	0.08	0.350	GC/MS	NIST 1941b
5	3.04	0.25	0.100	GC/MS	IAEA 459
6	2.58	0.54	0.500	GC/MS	NIST 1941b
7	3.38	0.19	0.020	GC/HRMS	IAEA-459
9	3.05	0.13	0.006	GC/MSMS	IAEA-459
10	3.58	0.06	0.260	GC/MS	NIST 1941b
11	2.22	0.31	0.400	GC/MS	IAEA-383
13	3.81	0.51	0.070	GC/MS	IAEA-459
			Results not used for the assignment value		
12	<1.40		0.820	GC/MS	IAEA-383

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

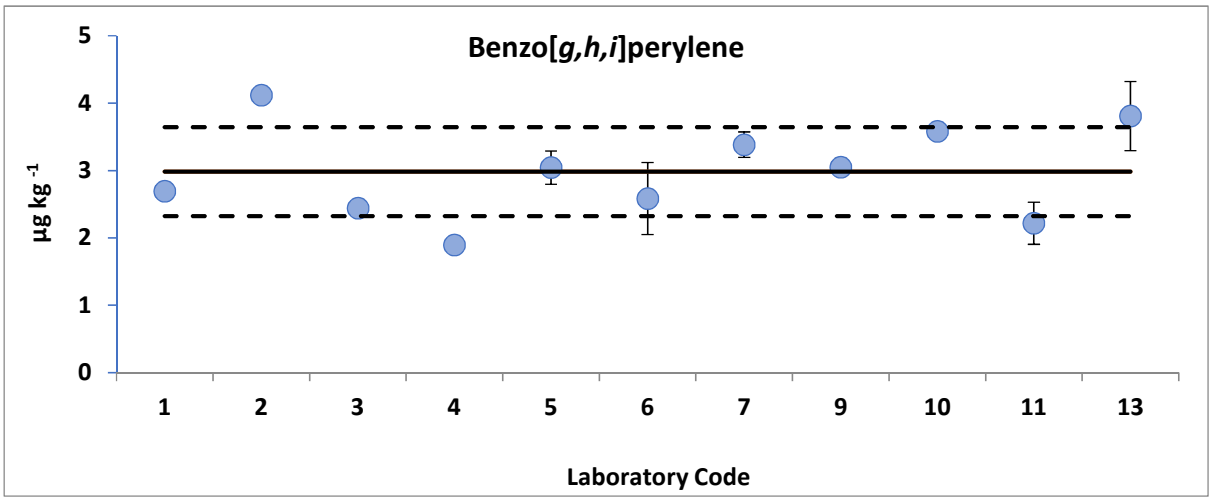

*FIG. 21. Laboratory results used to calculate the assignment mass fraction of Benzo(g,h,i)perylene in IAEA-477 (*µg kg^{-1}).

APPENDIX II: CHARACTERIZATION RESULTS: INFORMATION VALUES OF PAHS

TABLE 27. C2- NAPHTHALENES RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
1	2.82	0.48	0.700	GC/MS	NIST 1941b
2	5.31	0.28	0.070	GC/MSMS	IAEA-408
4	8.03	0.13	0.120	GC/MS	NIST 1941b
7	12.0	1.47	0.350	GC/HRMS	IAEA-459
10	5.17	0.11	0.120	GC/MS	NIST 1941b
13	14.8	1.81	0.017	GC/MS	IAEA-459

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

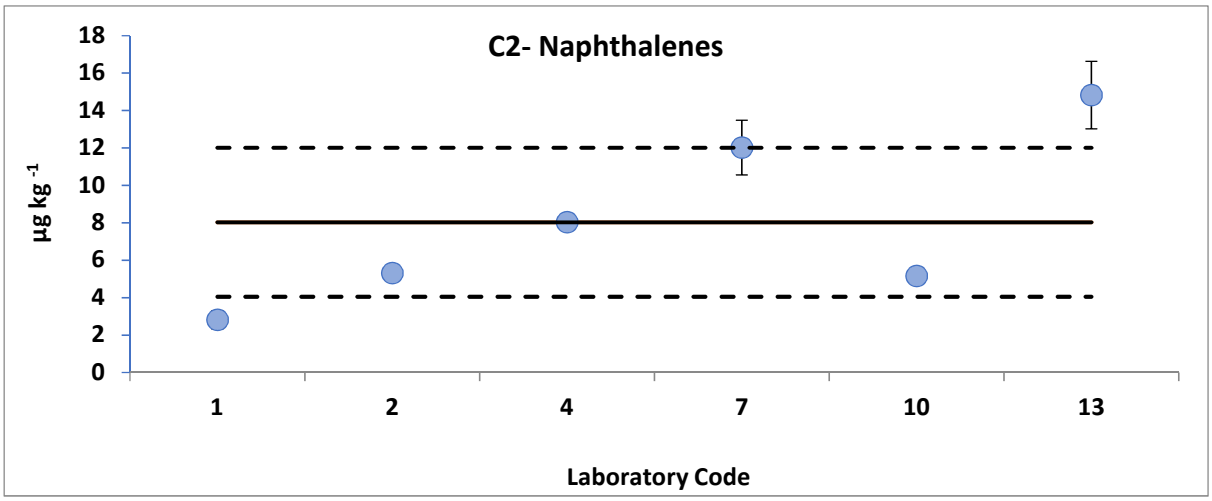

*FIG. 22. Laboratory results used to calculate the information mass fraction of C2- Naphthalenes in IAEA-477 (*µg kg^{-1}*).*

TABLE 28. C3- NAPHTHALENES RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
1	2.82	0.63	0.700	GC/MS	NIST 1941b
2	2.47	0.30	0.250	GC/MSMS	IAEA-408
4	5.62	0.29	0.120	GC/MS	NIST 1941b
7	4.77	0.52	0.380	GC/HRMS	IAEA-459
10	5.95	0.66	0.100	GC/MS	NIST 1941b
13	9.31	1.84	0.017	GC/MS	IAEA-459

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

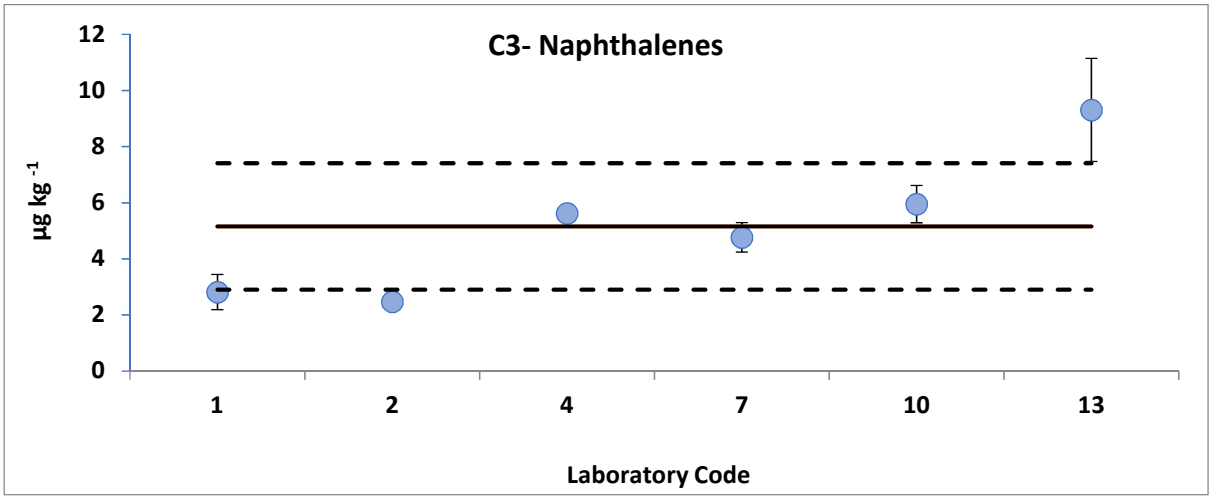

*FIG. 23. Laboratory results used to calculate the information mass fraction of C3- Naphthalenes in IAEA-477 (*µg kg^{-1}).

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

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
6	2.48	0.18	0.500	GC/MS	NIST 1941b
7	1.42	0.06	0.130	GC/HRMS	IAEA-459
13	2.10	0.25	0.026	GC/MS	IAEA-459

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

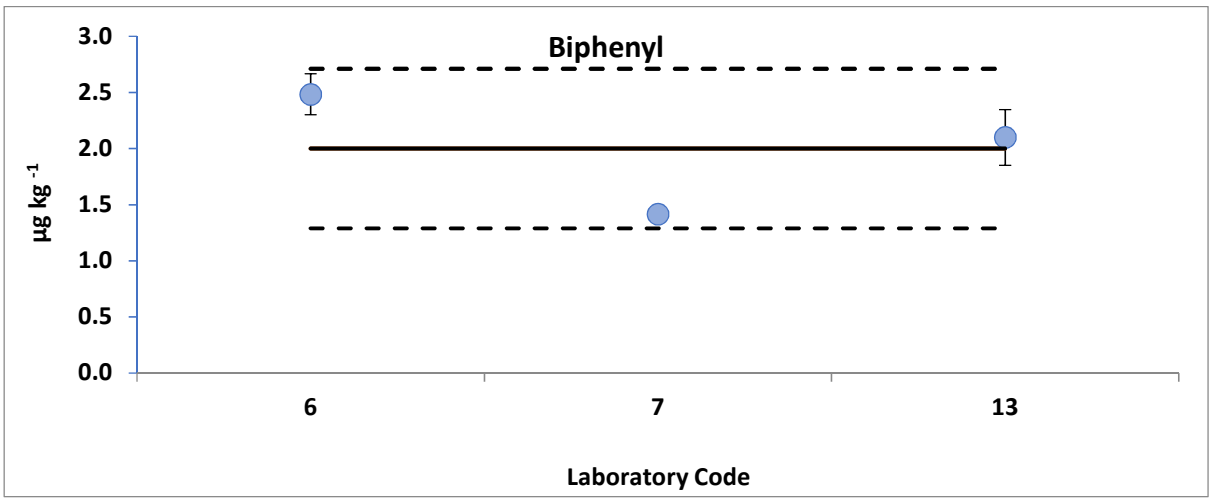

*FIG. 24. Laboratory results used to calculate the information mass fraction of Biphenyl in IAEA-477 (*µg kg^{-1}*).*

TABLE 30. ACENAPHTHYLENE RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
1	0.41	0.03	0.700	GC/MS	NIST 1941b
2	0.38	0.05	0.170	GC/MSMS	IAEA-408
5	0.47	0.01	0.050	GC/MS	IAEA 459
6	1.19	0.15	0.500	GC/MS	NIST 1941b
9	0.62	0.07	0.024	GC/MSMS	IAEA-459
10	0.80	0.16	0.130	GC/MS	NIST 1941b
11	0.82	0.13	0.400	GC/MS	IAEA-383
13	0.96	0.08	0.022	GC/MS	IAEA-459
			Results not used for the assignment value		
4	<LOQ		0.630	GC/MS	NIST 1941b
7	4.77	0.52	0.130	GC/HRMS	IAEA-459

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

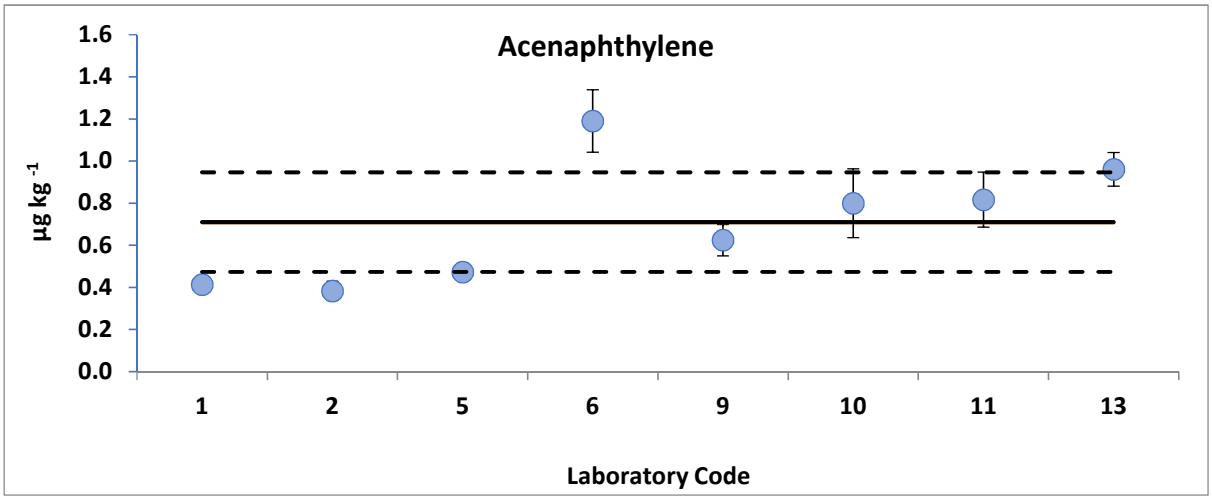

*FIG. 25. Laboratory results used to calculate the information mass fraction of Acenaphthylene in IAEA-477 (*µg kg^{-1}).

TABLE 31. FLUORENE RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
1	0.52	0.03	0.700	GC/MS	NIST 1941b
2	0.69	0.05	0.180	GC/MSMS	IAEA-408
3	0.26	0.07	0.500	HPLC-FLD	QPH094MS
5	0.51	0.01	0.050	GC/MS	IAEA 459
6	1.62	0.32	0.500	GC/MS	NIST 1941b
7	0.39	0.01	0.110	GC/HRMS	IAEA-459
9	1.43	0.07	0.037	GC/MSMS	IAEA-459
10	0.58	0.03	0.110	GC/MS	NIST 1941b
11	1.13	0.26	0.400	GC/MS	IAEA-383
13	0.89	0.10	0.039	GC/MS	IAEA-459
Results not used for the assignment value					
4	<LOQ		0.460	GC/MS	NIST 1941b
12	<2.15		1.300	GC/MS	IAEA-383

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

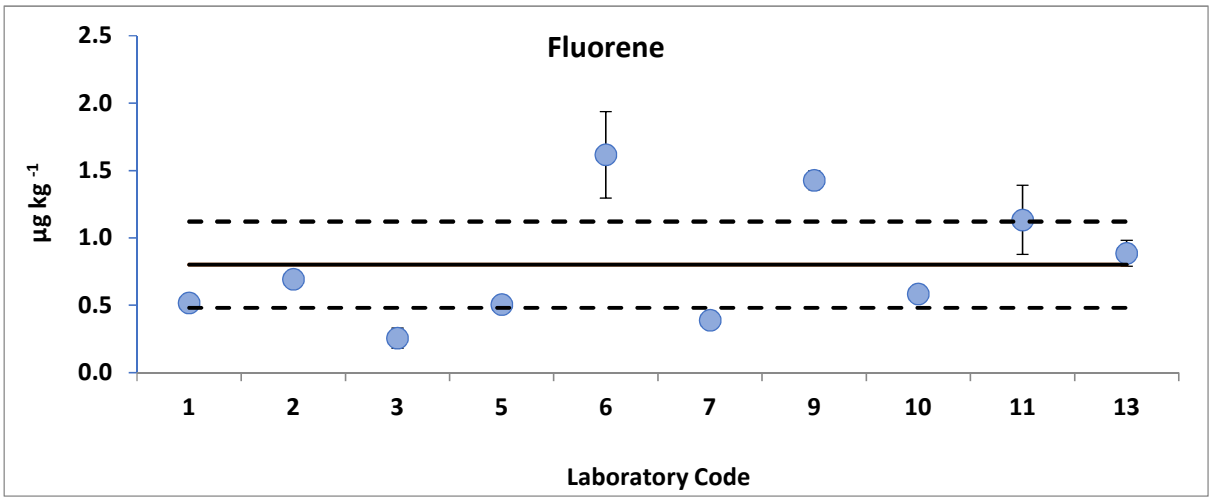

*FIG. 26. Laboratory results used to calculate the information mass fraction of Fluorene in IAEA-477 (*µg kg^{-1}*).*

TABLE 32. ACENAPHTHENE RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
1	0.11	0.02	0.700	GC/MS	NIST 1941b
2	0.09	0.02	0.210	GC/MSMS	IAEA-408
3	0.05	0.02	0.500	HPLC-FLD	QPH094MS
5	0.25	0.02	0.050	GC/MS	IAEA 459
6	0.56	0.03	0.500	GC/MS	NIST 1941b
10	0.43	0.04	0.130	GC/MS	NIST 1941b
11	0.88	0.03	0.400	GC/MS	IAEA-383
13	0.23	0.04	0.033	GC/MS	IAEA-459
			Results not used for the assignment value		
4	<LOQ		0.280	GC/MS	NIST 1941b
7	<1.3		0.220	GC/HRMS	IAEA-459
9	<1.518		0.455	GC/MSMS	IAEA-459

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

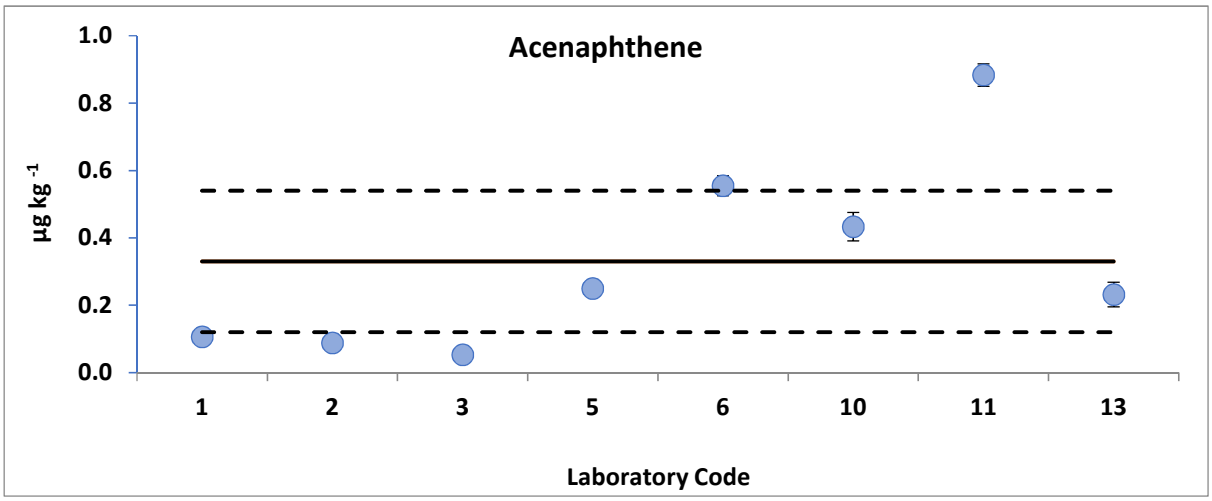

*FIG. 27. Laboratory results used to calculate the information mass fraction of Acenaphthene in IAEA-477 (*µg kg^{-1}).

TABLE 33. C1-FLUORENES RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
4	2.00	0.12	0.140	GC/MS	NIST 1941b
7	2.20	0.15	0.330	GC/HRMS	IAEA-459
13	4.03	0.39	0.039	GC/MS	IAEA-459

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

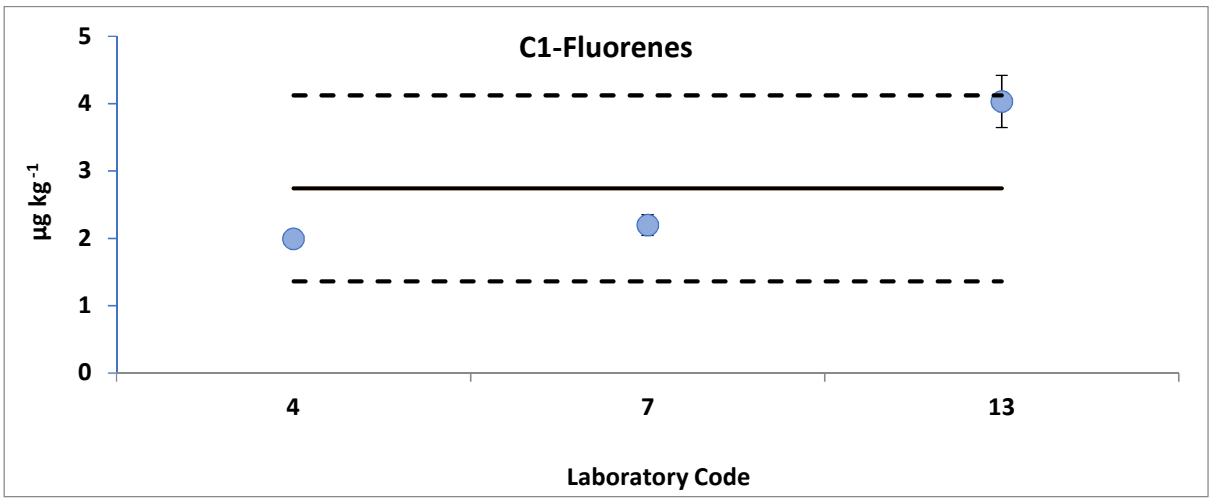

FIG. 28. Laboratory results used to calculate the information mass fraction of C1-Fluorenes *in IAEA-477 (*µg kg^{-1}).

TABLE 34. C2-FLUORENES RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
4	3.09	0.19	0.140	GC/MS	NIST 1941b
7	2.77	0.08	0.650	GC/HRMS	IAEA-459
13	5.42	0.46	0.039	GC/MS	IAEA-459

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

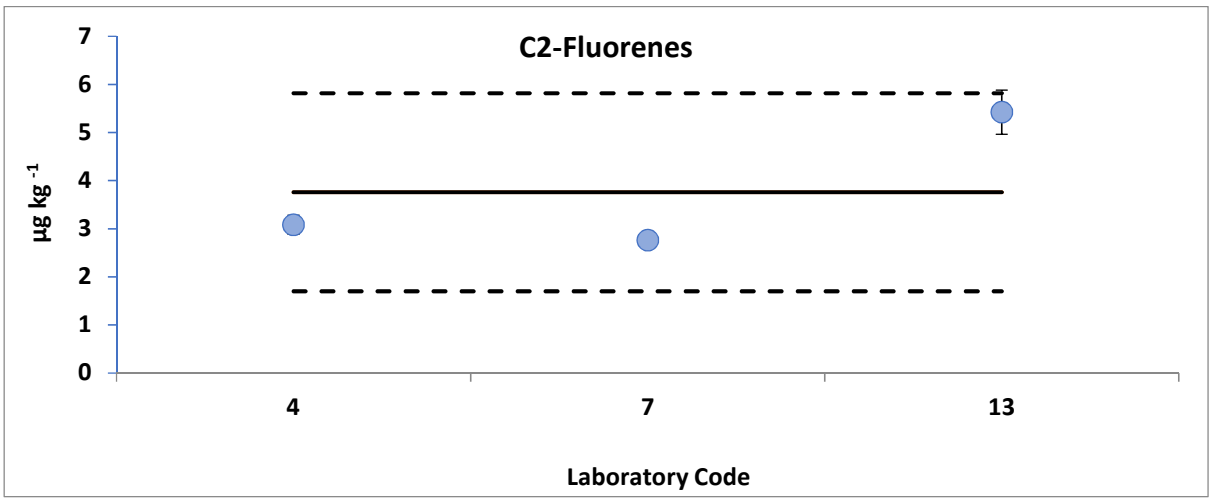

*FIG. 29. Laboratory results used to calculate the information mass fraction of C2-Fluorenes in IAEA-477 (*µg kg^{-1}).

TABLE 35. C3-FLUORENES RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
4	5.45	0.65	0.140	GC/MS	NIST 1941b
7	1.47	0.15	0.650	GC/HRMS	IAEA-459
13	5.94	0.63	0.039	GC/MS	IAEA-459

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

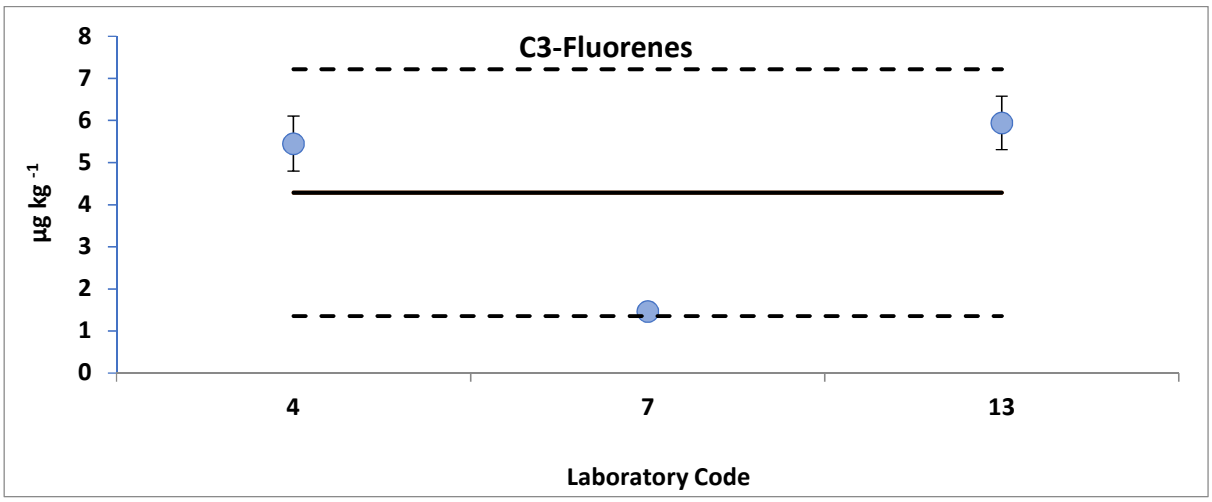

*FIG. 30. Laboratory results used to calculate the information mass fraction of C3-Fluorenes in IAEA-477 (*µg kg^{-1}*).*

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

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
2	0.33	0.04	0.300	GC/MSMS	IAEA-408
4	0.43	0.02	0.130	GC/MS	NIST 1944
6	0.56	0.05	0.500	GC/MS	NIST 1941b
7	0.66	0.02	0.110	GC/HRMS	IAEA-459
10	0.47	0.04	0.070	GC/MS	NIST 1941b
13	0.83	0.11	0.014	GC/MS	IAEA-459

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

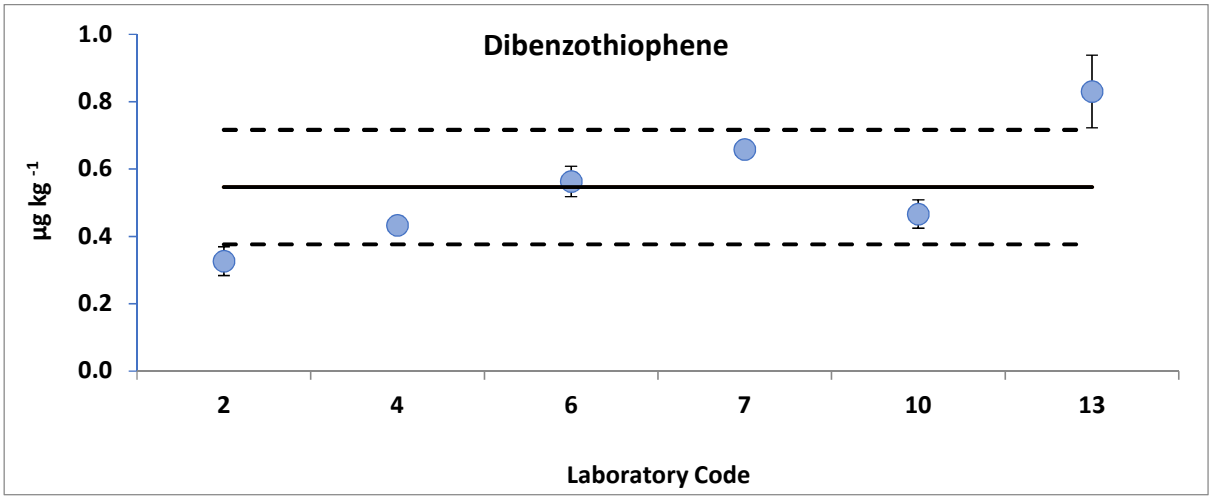

*FIG. 31. Laboratory results used to calculate the information mass fraction of Dibenzothiophene in IAEA-477 (*µg kg^{-1}).

TABLE 37. C1-DIBENZOTHIOPHENES RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
4	2.41	0.09	0.240	GC/MS	NIST 1941b
7	4.38	0.22	0.270	GC/HRMS	IAEA-459
10	1.47	0.13	0.070	GC/MS	NIST 1941b
13	1.69	0.14	0.014	GC/MS	IAEA-459

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

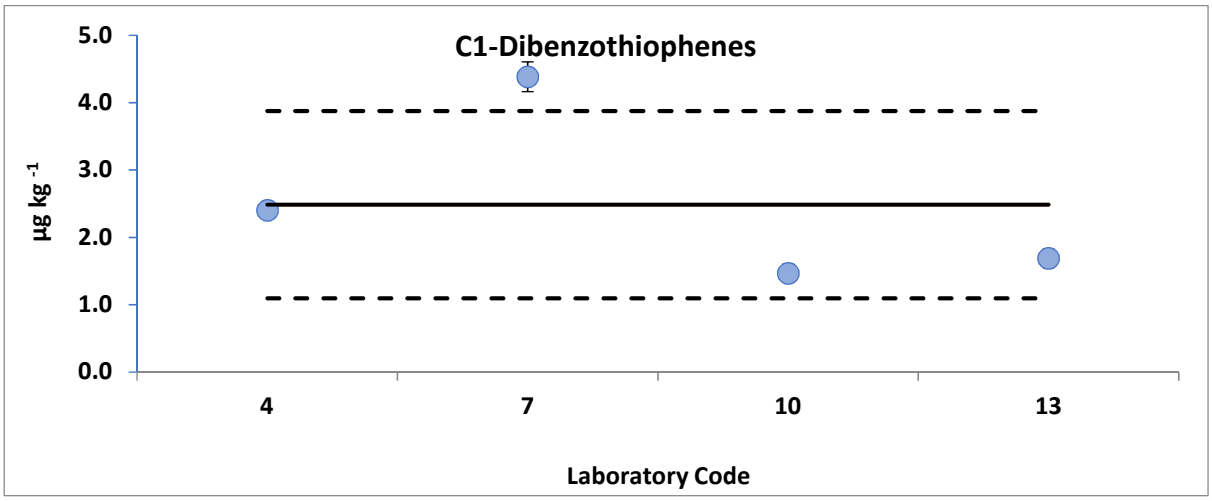

*FIG. 32. Laboratory results used to calculate the information mass fraction of C1-Dibenzothiophenes in IAEA-477 (*µg kg^{-1}*).*

TABLE 38. C2-DIBENZOTHIOPHENES RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
4	9.40	0.28	0.240	GC/MS	NIST 1941b
7	14.90	1.70	0.540	GC/HRMS	IAEA-459
10	2.95	0.37	0.070	GC/MS	NIST 1941b
13	3.21	0.19	0.014	GC/MS	IAEA-459

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

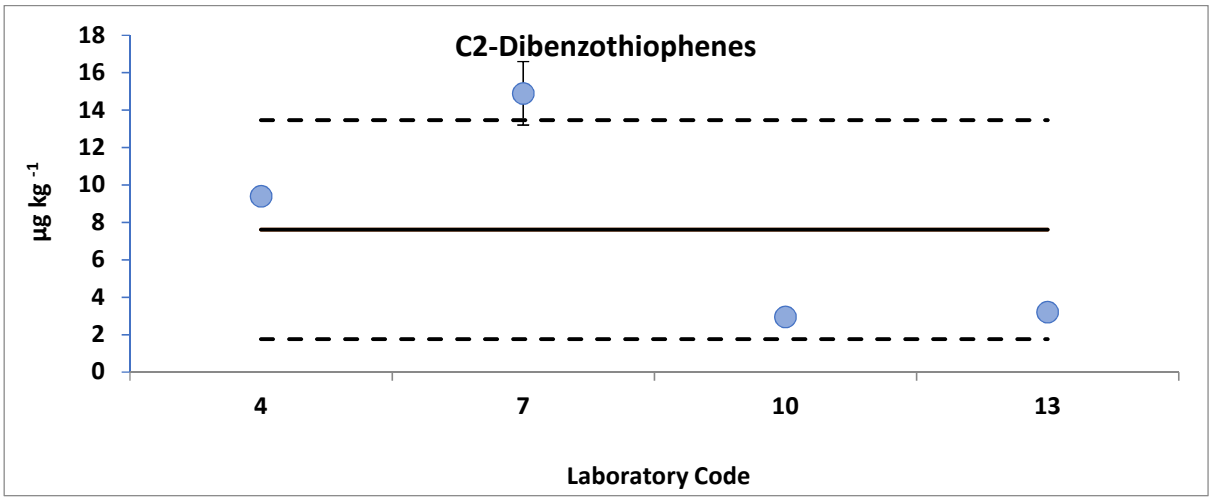

*FIG. 33. Laboratory results used to calculate the information mass fraction of C2-Dibenzothiophenes in IAEA-477 (*µg kg^{-1}*).*

TABLE 39. C3-DIBENZOTHIOPHENES RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
4	6.89	0.13	0.240	GC/MS	NIST 1941b
7	10.75	0.92	0.540	GC/HRMS	IAEA-459
13	4.94	0.21	0.014	GC/MS	IAEA-459

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

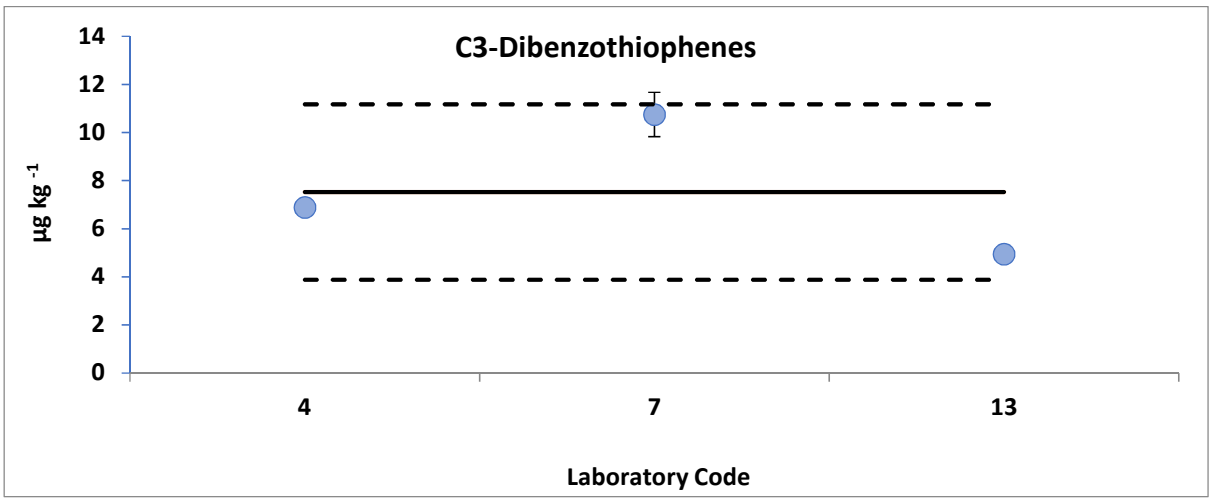

*FIG. 34. Laboratory results used to calculate the information mass fraction of C3-Dibenzothiophenes in IAEA-477 (*µg kg^{-1}).

TABLE 40. C2- PHEN/ANTH RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
1	7.05	0.35	0.700	GC/MS	NIST 1941b
2	7.93	0.33	0.290	GC/MSMS	IAEA-408
4	9.93	0.40	0.070	GC/MS	NIST 1941b
7	8.82	1.36	0.350	GC/HRMS	IAEA-459
10	14.08	1.33	0.110	GC/MS	NIST 1941b
13	3.70	0.19	0.014	GC/MS	IAEA-459

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

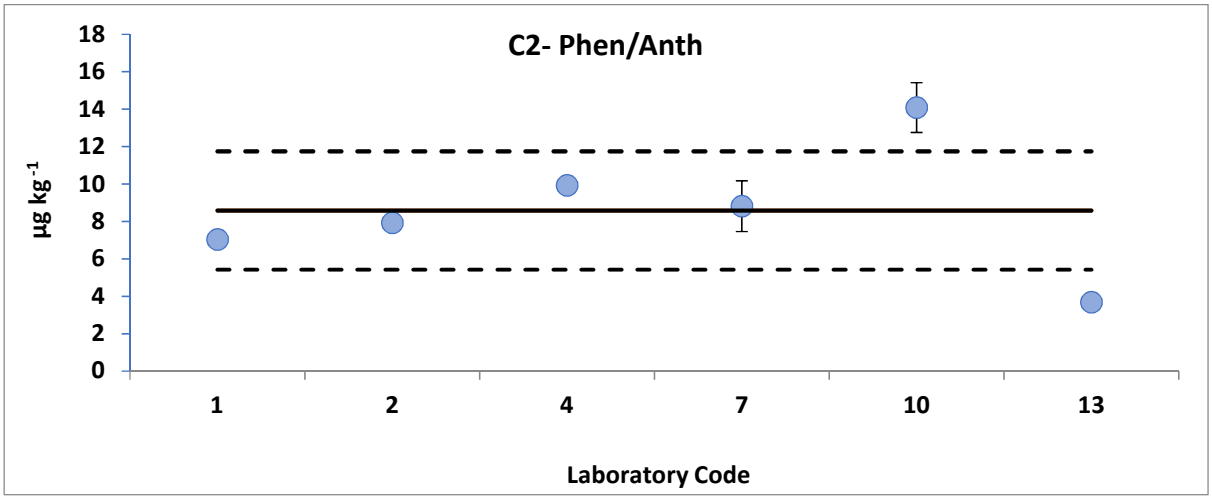

*FIG. 35. Laboratory results used to calculate the information mass fraction of C2- Phen/Anth in IAEA-477 (*µg kg^{-1}).

TABLE 41. C3- PHEN/ANTH RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
1	4.22	0.23	0.700	GC/MS	NIST 1941b
2	2.69	0.11	0.090	GC/MSMS	IAEA-408
4	8.14	0.15	0.080	GC/MS	NIST 1941b
7	5.02	0.14	0.310	GC/HRMS	IAEA-459
10	7.62	0.91	0.120	GC/MS	NIST 1941b
13	1.76	0.12	0.014	GC/MS	IAEA-459

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

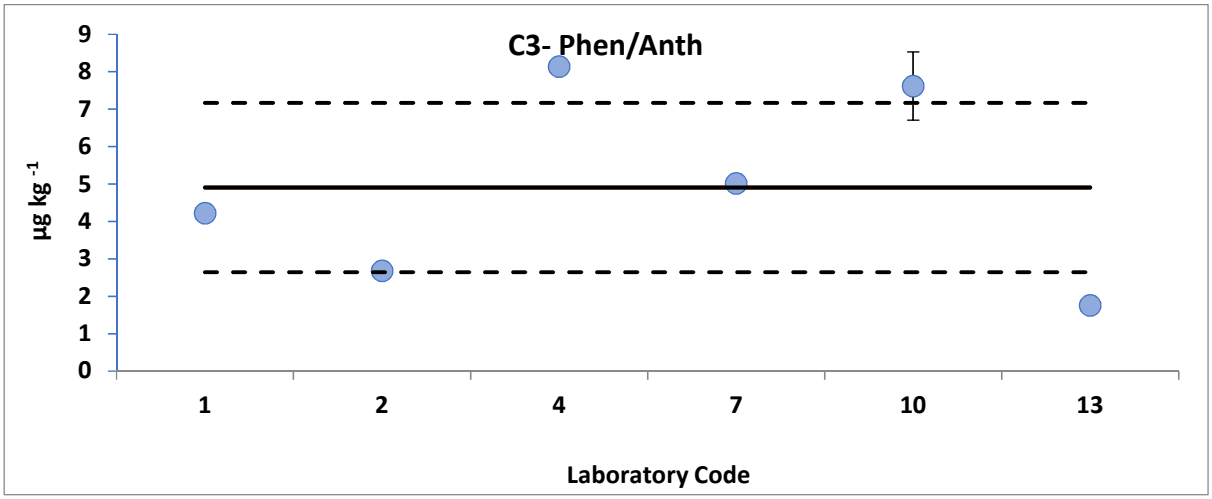

*FIG. 36. Laboratory results used to calculate the information mass fraction of C3- Phen/Anth in IAEA-477 (*µg kg^{-1}).

TABLE 42. C4- PHEN/ANTH RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
1	2.21	0.14	0.700	GC/MS	NIST 1941b
4	2.94	0.18	0.080	GC/MS	NIST 1941b
7	1.83	0.16	0.310	GC/HRMS	IAEA-459
13	2.68	0.08	0.014	GC/MS	IAEA-459

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

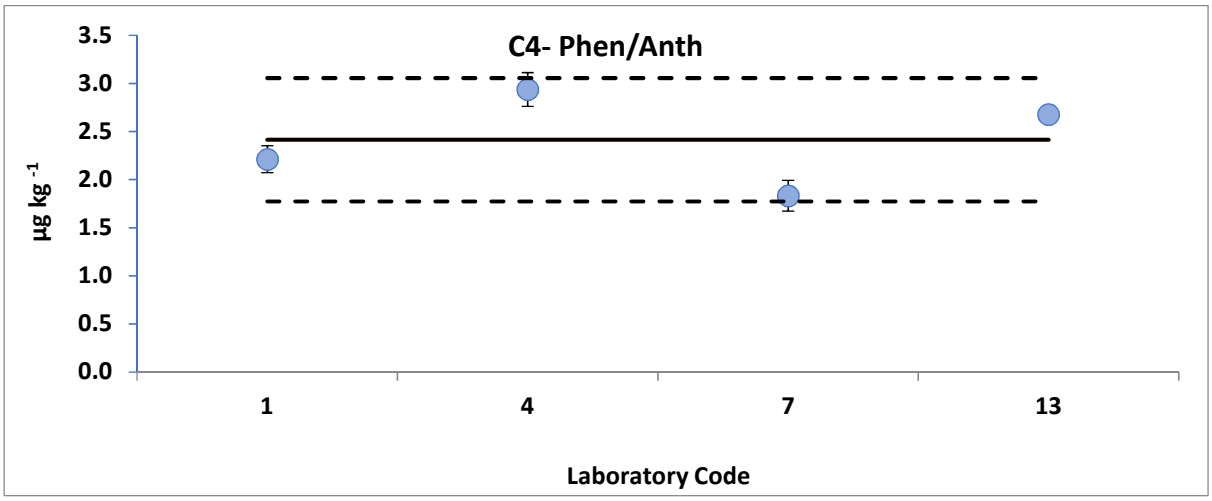

*FIG. 37. Laboratory results used to calculate the information mass fraction of C4- Phen/Anth in IAEA-477 (*µg kg^{-1}).

TABLE 43. 1-METYLPYRENE RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
7	0.48	0.03	0.020	GC/HRMS	IAEA-459
13	0.93	0.09	0.023	GC/MS	IAEA-459

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

TABLE 44. C1-FLUORANTHENES/PYRENES RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
1	3.52	0.16	0.300	GC/MS	NIST 1941b
4	1.17	0.05	0.640	GC/MS	NIST 1941b
7	4.40	0.15	0.240	GC/HRMS	IAEA-459
10	5.78	0.27	0.100	GC/MS	NIST 1941b
13	7.21	0.35	0.023	GC/MS	IAEA-459

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

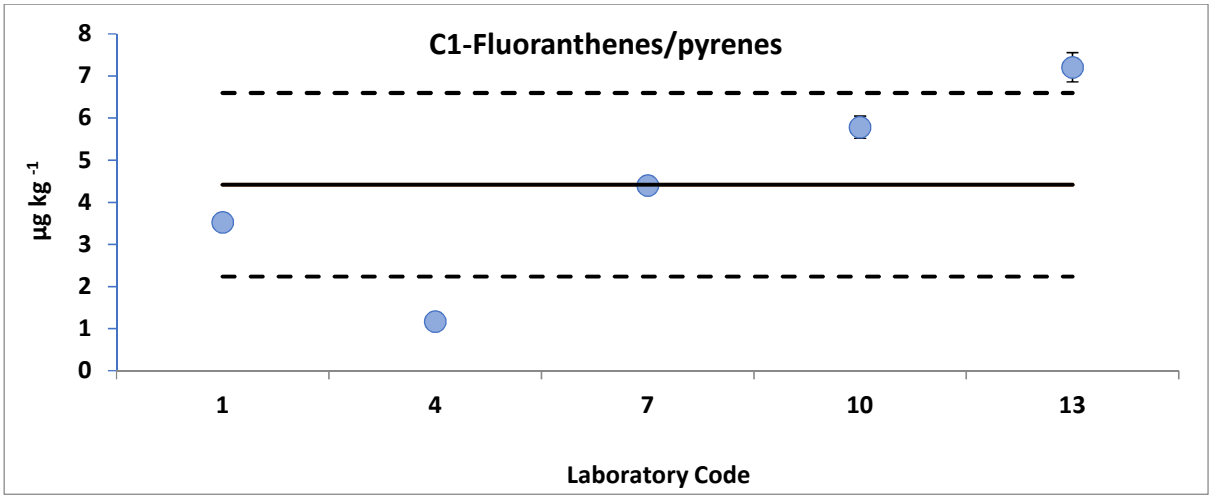

*FIG. 38. Laboratory results used to calculate the information mass fraction of C1-Fluoranthenes/pyrenes in IAEA-477 (*µg kg^{-1}*).*

TABLE 45. C2-FLUORANTHENES/PYRENES RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
1	3.72	0.18	0.300	GC/MS	NIST 1941b
4	1.11	0.03	0.640	GC/MS	NIST 1941b
7	2.53	0.27	0.240	GC/HRMS	IAEA-459
10	6.60	0.50	0.110	GC/MS	NIST 1941b

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

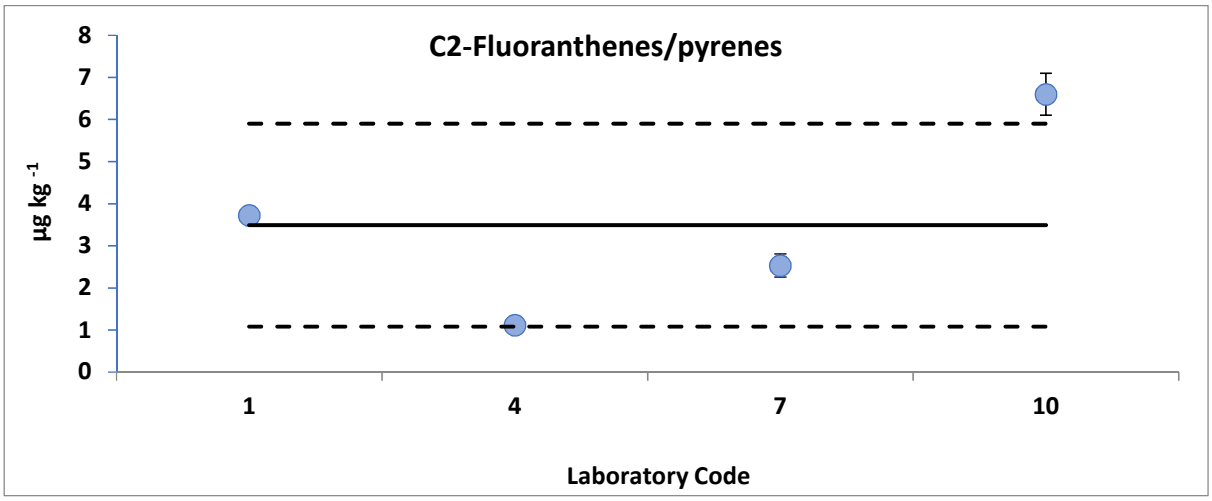

*FIG. 39. Laboratory results used to calculate the information mass fraction of C2-Fluoranthenes/pyrenes in IAEA-477 (*µg kg^{-1}*).*

TABLE 46. C3-FLUORANTHENES/PYRENES RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
1	2.67	0.08	0.300	GC/MS	NIST 1941b
7	1.53	0.17	0.240	GC/HRMS	IAEA-459

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

TABLE 47. CHRYSENE RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
1	4.23	0.15	0.300	GC/MS	NIST 1941b
3	1.71	0.16	0.500	HPLC-FLD	QPH094MS
6	7.55	0.53	0.500	GC/MS	NIST 1941b
7	2.52	0.10	0.020	GC/HRMS	IAEA-459

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

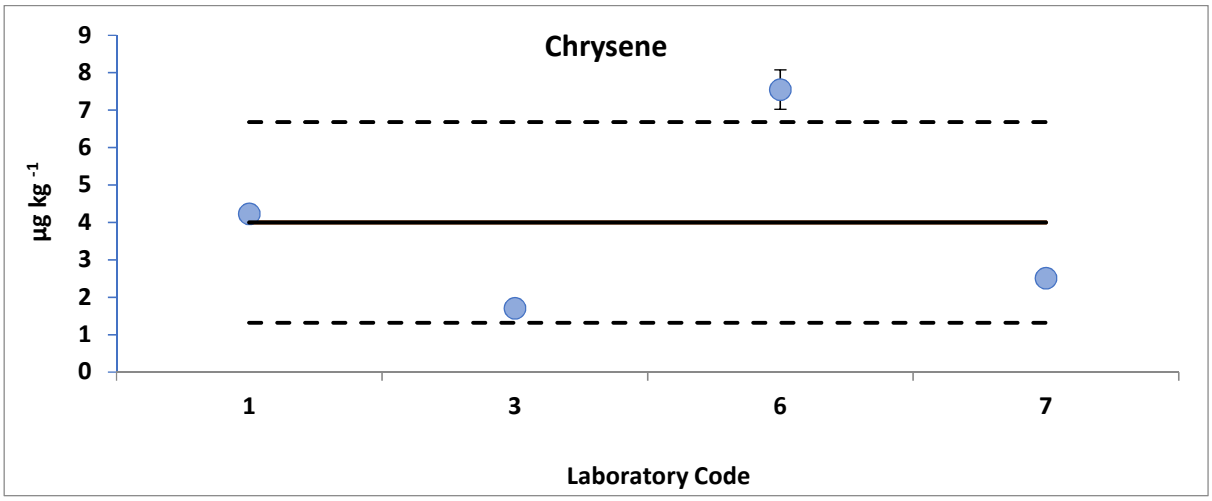

*FIG. 40. Laboratory results used to calculate the information mass fraction of Chrysene in IAEA-477 (*µg kg^{-1}).

TABLE 48. TRIPHENYLENE RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
1	0.84	0.04	0.300	GC/MS	NIST 1941b
7	0.78	0.04	0.020	GC/HRMS	IAEA-459

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

TABLE 49. C2-CHRYSENES RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
1	4.06	0.11	0.300	GC/MS	NIST 1941b
4	1.50	0.05	0.170	GC/MS	NIST 1941b
7	2.65	0.21	0.360	GC/HRMS	IAEA-459
10	3.90	0.25	0.120	GC/MS	NIST 1941b
13	5.50	1.78	0.011	GC/MS	IAEA-459

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

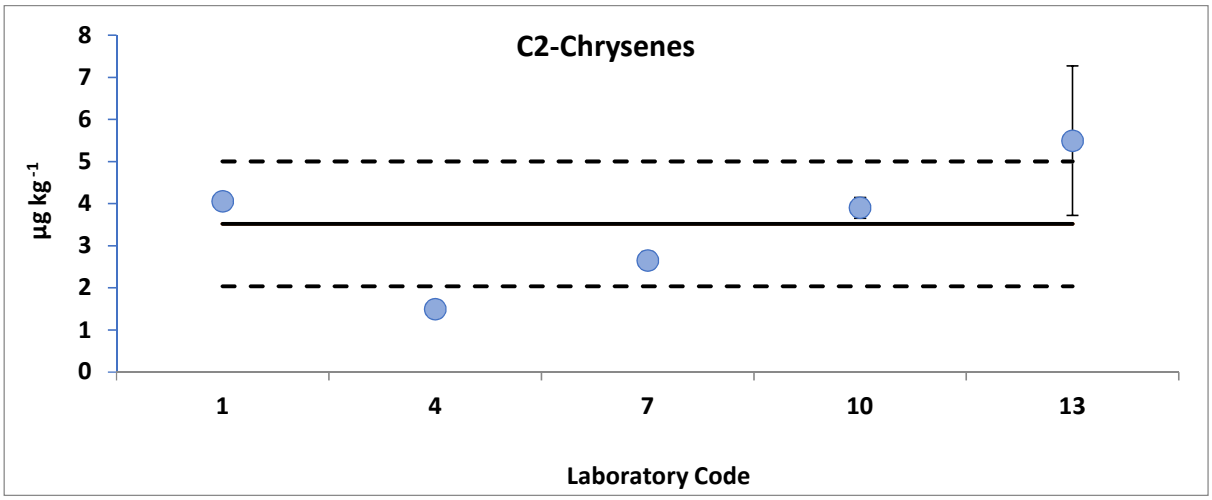

*FIG. 41. Laboratory results used to calculate the information mass fraction of C2-Chrysenes in IAEA-477 (*µg kg^{-1}).

TABLE 50. C3-CHRYSENES RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
1	1.53	0.07	0.300	GC/MS	NIST 1941b
7	1.55	0.11	0.360	GC/HRMS	IAEA-459
13	6.47	1.97	0.011	GC/MS	IAEA-459
Results not used for the assignment value					
4	<LOQ		0.170	GC/MS	NIST 1941b

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

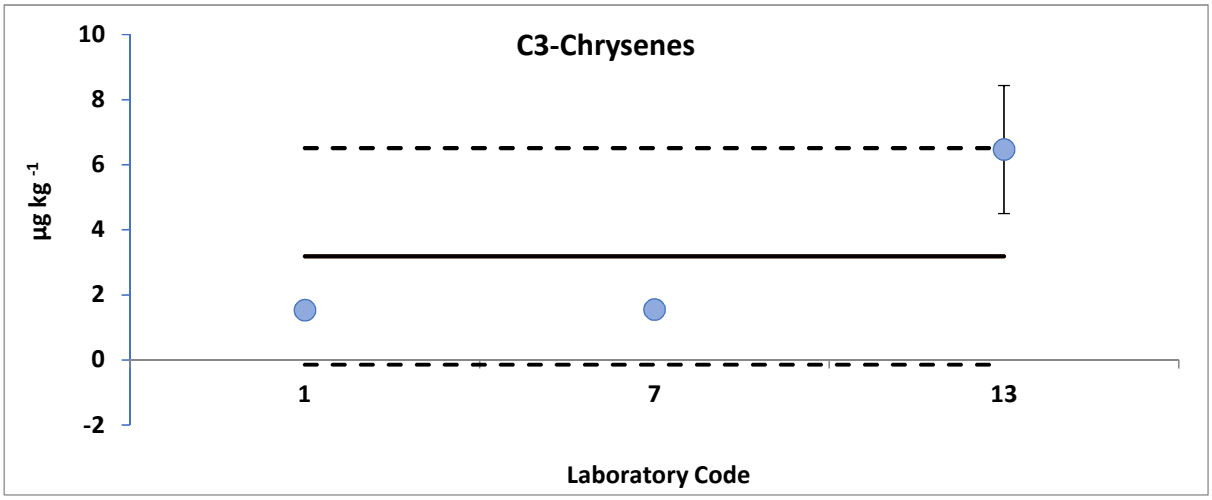

FIG. 42. Laboratory results used to calculate the information mass fraction of C3-Chrysenes *in IAEA-477 (*µg kg^{-1}).

TABLE 51. BENZO[B]FLUORANTHENE RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
1	3.09	0.11	0.700	GC/MS	NIST 1941b
3	5.45	0.26	0.500	HPLC-FLD	QPH094MS
6	11.10	1.46	0.500	GC/MS	NIST 1941b
7	3.42	0.27	0.010	GC/HRMS	IAEA-459

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

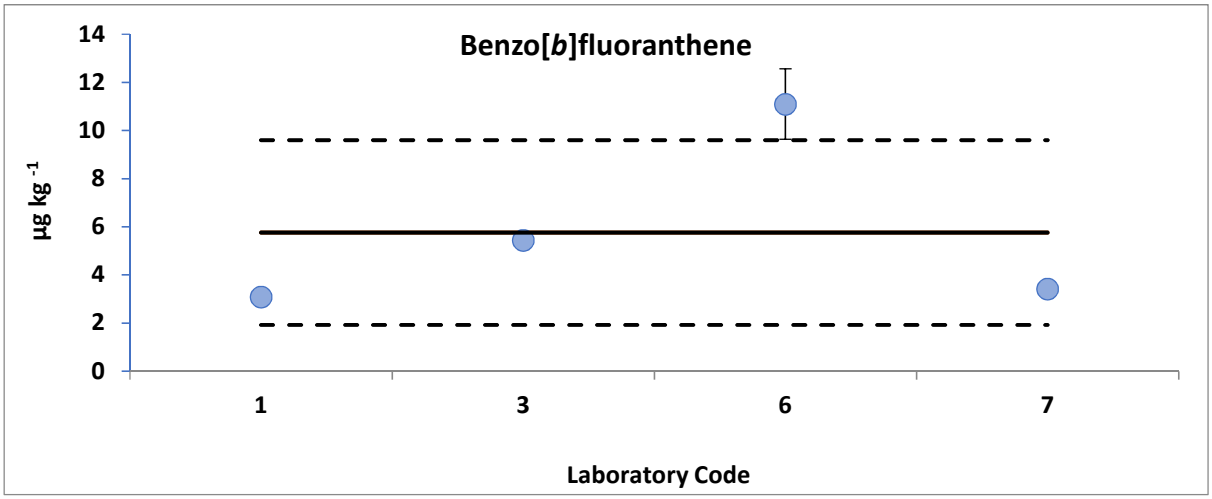

*FIG. 43. Laboratory results used to calculate the information mass fraction of Benzo(b)fluoranthene in IAEA-477 (*µg kg^{-1}).

TABLE 52. BENZO[J]FLUORANTHENE RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
1	1.59	0.05	0.700	GC/MS	NIST 1941b
6	4.00	0.27	0.500	GC/MS	NIST 1941b
7	0.90	0.16	0.010	GC-HRMS	IAEA-459

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

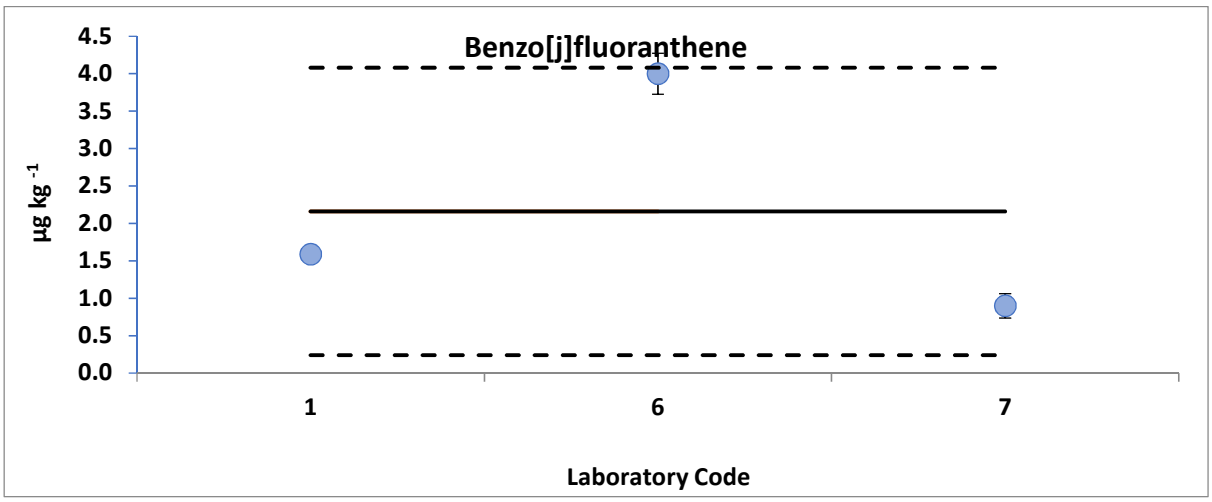

*FIG. 44. Laboratory results used to calculate the information mass fraction of Benzo(j)fluoranthene in IAEA-477 (*µg kg^{-1}).

TABLE 53. BENZO[A]FLUORANTHENE RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
1	0.61	0.04	0.700	GC/MS	NIST 1941b
13	0.80	0.19	0.024	GC/MS	IAEA-459

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

TABLE 54. DIBENZ(A,H)ANTHRACENE RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
1	0.45	0.02	0.700	GC/MS	NIST 1941b
2	0.45	0.04	0.290	GC/MSMS	IAEA-408
3	0.33	0.03	0.500	HPLC-FLD	QPH094MS
6	0.67	0.09	0.500	GC/MS	NIST 1941b
7	0.25	0.02	0.020	GC/HRMS	IAEA-459
9	0.43	0.04	0.009	GC/MSMS	IAEA-459
10	0.50	0.00	0.160	GC/MS	NIST 1941b
11	0.37	0.07	0.400	GC/MS	IAEA-383
13	0.40	0.05	0.131	GC/MS	IAEA-459
Results not used for the assignment value					
4	<LOQ		0.650	GC/MS	NIST 1941b
12	<2.69		1.620	GC/MS	IAEA-383

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

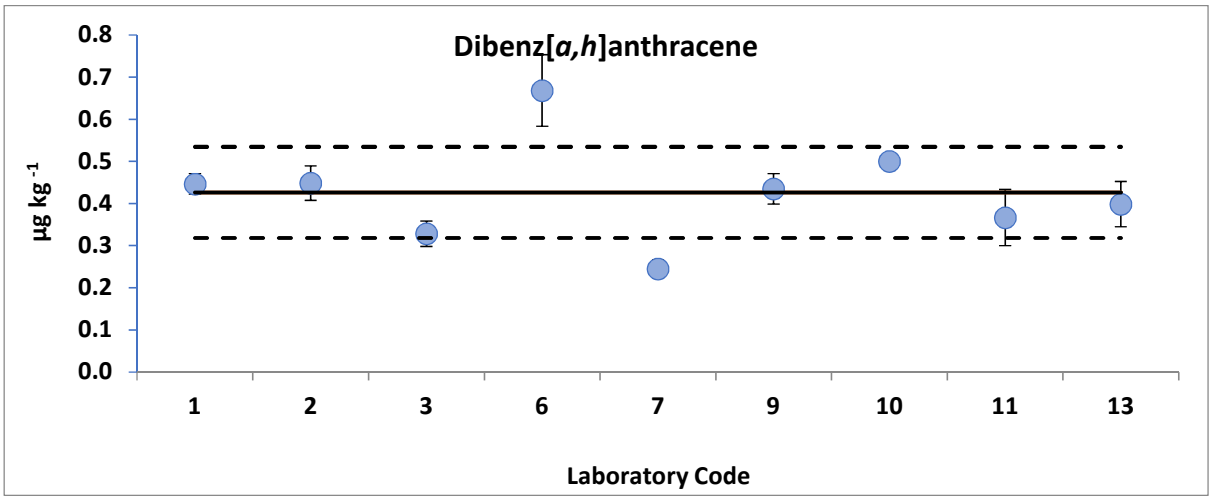

*FIG. 45. Laboratory results used to calculate the information mass fraction of Dibenz(a,h)anthracene in IAEA-477 (*µg kg^{-1}).

TABLE 55. PERYLENE RESULTS REPORTED BY PARTICIPANTS (µg kg^{-1})

LAB Code	Mean	Uncertainty*	Detection limit	Instrumentation	(C)RM use for QC
1	13.91	0.65	0.300	GC/MS	NIST 1941b
2	38.20	0.57	0.300	GC/MSMS	IAEA-408
4	8.82	0.26	1.100	GC/MS	NIST 1941b
6	74.83	7.33	0.500	GC/MS	NIST 1941b
7	16.62	0.58	0.010	GC/HRMS	IAEA-459
10	70.97	2.57	0.170	GC/MS	NIST 1941b
11	31.57	3.63	0.400	GC/MS	IAEA-383
12	39.07	3.44	1.630	GC/MS	IAEA-383
13	26.81	2.52	0.010	GC/MS	IAEA-459

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

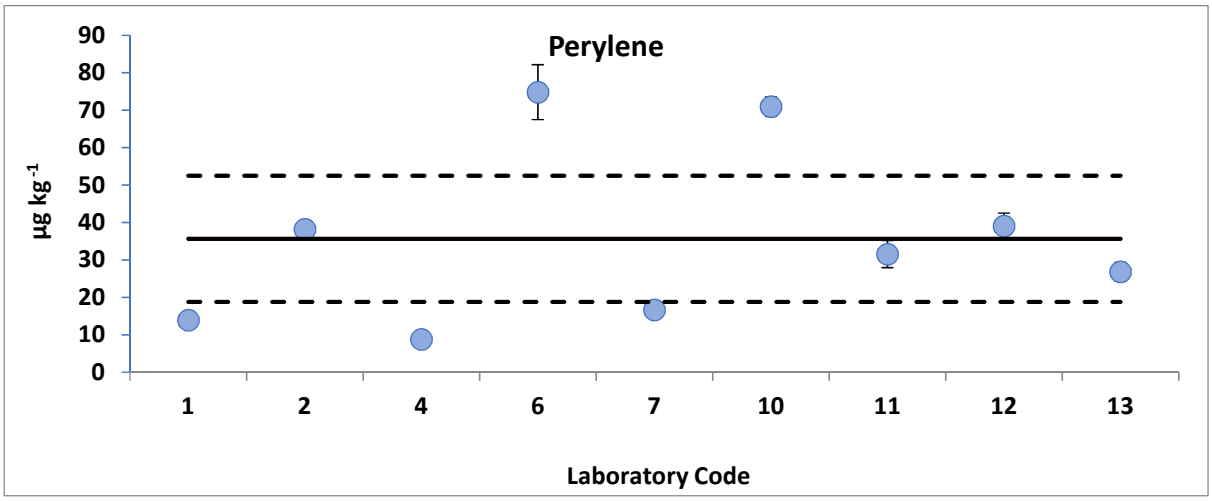

*FIG. 46. Laboratory results used to calculate the information mass fraction of Perylene in IAEA-477 (*µg kg^{-1}*).*

APPENDIX III: TRACEABILITY TABLE

Table 56. Calibrants, surrogates and CRMs used by Participants

Lab Code	Calibrants source	Calibrants used	Surrogates source	Surrogates used	CRMs source	Matrix CRMs Used
1	NIST	2260a	CAMBRIDGE Isotope Laboratories – 98%	d8-Naphthalene d8-Acenaphtylene d10-Acenaphtene d10-Fluorene d10-Phenanthrene d10-Anthracene d8-Dibenzothiophene d10-Fluoranthene d10-Pyrene d12-Benz[*a*]anthracene d12-Chrysene d12-Benzo[*b*]fluoranthene d12-Benzo[*k*]fluoranthene d12-Benzo[*e*]pyrene d12-Benzo[*a*]pyrene d12-Indeno[*1,2,3-c,d*]pyrene d14-Dibenz[*a,h*]anthracene d12-Benzo[*g,h,i*]perylene	NIST	SRM 1941b

Table 56. (cont.)

Lab Code	Calibrants source	Calibrants used	Surrogates source	Surrogates used	CRMs source	Matrix CRMs Used
2	Chiron AS, DR.EHRENSTRORFER GMBH	Acenaphthene Acenapthylene Anthracene Benz[*a*]anthracene Benzo[*a*]pyrene Benzo[*e*]pyrene Benzo[*b*]fluoranthene Benzo[*j*]fluoranthene Benzo[*k*]fluoranthene Benzo[*g,h,i*]perylene Chrysene Dibenz[*a,h*]anthracene Fluorene Fluoranthene Indeno[*1,2,3-c,d*] pyrene Naphthalene Perylene Phenanthrene Pyrene Triphenylene Naphthalene 1-Methylnaphthalene 2-Methylnaphthalene 2.6-Dimethylnaphthalene 2.3.5-Trimethylnaphthalene 2-Methylphenanthrene 3,6-Dimethylphenanthrene 1,2,8-Trimethylpibenzothiophene	Chiron AS, DR.EHRENSTRORFER GMBH	d8-Naphthalene d10-Acenaphthene d10-Anthracene d10-Fluoranthene d12-Benzo[*e*]pyrene d12-Benzo[*g,h,i*]perylene	IAEA	IAEA-408
3	ULTRASCIENTIFIC	PM-831A-1 (16 PAH compounds in acetonitrile-methanol 9:1)			QUASIMEME	Proficiency test QPH094MS

Table 56. (cont.)

Lab Code	Calibrants source	Calibrants used	Surrogates source	Surrogates used	CRMs source	Matrix CRMs Used
4	AccuStandard, Chiron AS	Naphthalene 1-Methylnaphthalene 2-Methylnaphthalene 2.6-Dimethylnaphthalene Acenaphthylene Acenaphthene 1-n-Propylnaphthalene 2-Butylnaphthalene Fluorene 1-Methylfluorene 9-Ethylfluorene 9-n-Propylfluorene 9-n-Butylfluorene Dibenzothiophene 4-Methyldibenzothiophene 1,2-Dimethyldibenzothiophene Phenanthrene Anthracene 3-Methylphenanthrene 2-Methylphenanthrene 4-Methylphenanthrene 1-Methylphenanthrene 1,5-/1,7-Dimethylphenanthrene 1,2,5-/1,2,7-Trimethylphenanthrene 1,2,6,9-Tetramethylphenanthrene Retene (1-Methyl-7-isopropylphenanthrene) Fluoranthene Pyrene	AccuStandard, Chiron AS	d8-Naphthalene d10- Acenaphthene d8-Dibenzothiophene d10-Phenanthrene d12-Chrysene d12-Perylene GC internal std: d14-p-terphenyl	NIST	1941b, 1944

Table 56. (cont.)

Lab Code	Calibrants source	Calibrants used	Surrogates source	Surrogates used	CRMs source	Matrix CRMs Used
4 (cont)		Benz[*a*]anthracene Chrysene 6-Methylchrysene 6-Ethylchrysene Benzo[*b*]Fluoranthene Benzo[*k*]Fluoranthene Benzo[*e*]pyrene Benzo[*a*]Pyrene Perylene Indeno[*1,2,3-c,d*]pyrene Dibenz[*a,h*]anthracene Benzo[*g,h,i*]perylene				

Table 56. (cont.)

Lab Code	Calibrants source	Calibrants used	Surrogates source	Surrogates used	CRMs source	Matrix CRMs Used
5	SUPELCO	Polynuclear Aromatic Hydrocarbons Mix 2000µg/mL each component in benzene: dichloromethane (50:50) Part Number: CRM47543 Naphthalene 1-Methylnaphthalene 2-Methylnaphthalene Acenaphthylene Acenaphthene Fluorene Phenanthrene Anthracene Fluoranthene Pyrene Benz[*a*]anthracene Chrysene Benzo[*b*]fluoranthene Benzo[*k*]fluoranthene Benzo[*a*]pyrene Indeno[*12,3-c,d*]pyrene Dibenz[*a,h*]anthracene Benzo[*g,h,i*]perylene	RESTEK, DR. EHRENSTORFER GMBH	Revised SV Internal Standard MIX Catalog No. 31885, Lot No. A0125855 d8-Naphthalene d10- Acenaphthene d10- Phenanthrene d12-Chrysene d12-Perylene GC internal std: d10-Fluoranthene	IAEA	IAEA-459

Table 56. (cont.)

Lab Code	Calibrants source	Calibrants used	Surrogates source	Surrogates used	CRMs source	Matrix CRMs Used
6	Chiron AS	Naphthalene 2-Methylnaphthalene 1-Methylnaphthalene Biphenyl 2.6-Dimethylnaphthalene 1.3-Dimethylnaphthalene 2.3-Dimethylnaphthalene 1.4-Dimethylnaphthalene Acenaphthylene Acenaphthene Dibenzofuran 1.3.7-Trimethylnaphthalene 2.3.5-Trimethylnaphthalene 1.2.3-Trimethylnaphthalene 1.4.6.7-Trimethylnaphthalene 1.2.5.6-Trimethylnaphthalene Fluorene 1-Methylfluorene 9-Ethylfluorene Dibenzothiophene Phenanthrene Anthracene 4-Methyldibenzothiophene 3-Methylphenanthrene 2-Methylphenanthrene 9-Methylphenanthrene 1-Methylphenanthrene 4-Ethyldibenzothiophene 3.6-Dimethylphenanthrene 4-Propyldibenzothiophene 1.7-Dimethylphenanthrene	Chiron AS	d8-Naphthalene d10-Biphenyl d8-Acenapththylene d10-Anthracene d10-Pyrene d12-Perylene d12-Indeno[*1,2,3-c,d*]pyrene GC internal std: d10-Phenanthrene	(LRM)	IMR Laboratory reference material (LRM)

Table 56. (cont.)

Lab Code	Calibrants source	Calibrants used	Surrogates source	Surrogates used	CRMs source	Matrix CRMs Used
6 (cont.)	Chiron AS	1.2-Dimethylphenanthrene 2.6.9-Trimethylphenanthrene 1.2.6-Trimethylphenanthrene 1.2.7-Trimethylphenanthrene 1.2.6.9-Tetramethylphenanthrene Fluoranthene Pyrene Benz[*a*]anthracene Chrysene Benzo[*b*]fluoranthene Benzo[*k*]fluoranthene Benzo[*a*]pyrene Benzo[*e*]pyrene Perylene Indeno[*1,2,3-c,d*]pyrene Dibenz[*a,h*]anthracene Benzo[*g,h,i*]perylene				

Table 56. (cont.)

Lab Code	Calibrants source	Calibrants used	Surrogates source	Surrogates used	CRMs source	Matrix CRMs Used
7	CIL	US EPA 16 PAH Cocktail (^{13}C, 99%): Acenaphthene Acenaphthylene Anthracene Benz[*a*]anthracene Benzo[*b*]fluoranthene Benzo[*k*]fluoranthene Benzo[*g,h,i*]perylene Benzo[*a*]pyrene Chrysene Dibenzo[*a,h*]anthracene Fluoranthene Fluorene Indeno[*1,2,3-c,d*] pyrene Naphthalene Phenanthrene Pyrene Native individual PAHs		Native individual PAHs	IAEA	IAEA-459

Table 56. (cont.)

Lab Code	Calibrants source	Calibrants used	Surrogates source	Surrogates used	CRMs source	Matrix CRMs Used
9	DR. EHRENSTORFER GMBH	PAH Mix9 Naphthalene Acenaphthene Acenaphthylene Fluorene Phenanthrene Anthracene Fluoranthene Pyrene Benz[*a*]anthracene Chrysene Benzo[*b*]fluoranthene Benzo[*k*]fluoranthene Benzo[*a*]pyrene Dibenz[*a,h*]anthracene Indeno[*1,2,3-c,d*] pyrene Benzo[*g,h,i*]perylene	DR. EHRENSTORFER GMBH	PAH Mix31 d10-Acenaphthene d12-Chrysene d8-Naphthalene d12-Perylene d10-Phenanthrene	IAEA	IAEA-459

Table 56. (cont.)

Lab Code	Calibrants source	Calibrants used	Surrogates source	Surrogates used	CRMs source	Matrix CRMs Used
10	Chemservice	1,2-Dimethylnaphthalene 1-Methylnaphthalene 1-Methylphenanthrene 2,3,5-Trimethylnaphthalene 2,6-Dimethylnaphthalene 2-Methylnaphthalene 2-Methylphenanthrene 3,6-Dimethylphenanthrene Acenaphthene Acenaphthylene Anthracene Benz[*a*]anthracene Benzo[*a*]pyrene Benzo[*b*]fluoranthene Benzo[*e*]pyrene Benzo[*ghi*]Perylene d12-Benzo[*ghi*]Perylene Benzo[*k*]fluoranthene Chrysene Dibenz[a,h]anthracene Dibenzothiophene Fluoranthene Fluorene Indeno[*1,2,3-c,d*]pyrene Naphthalene Perylene Phenanthrene Pyrene	Chemservice	d10-Acenaphthene d12-Benzo[*g,h,i*]Perylene d12-Chrysene d8-Naphthalene d12-Perylene d10-Phenanthrene d10-Pyrene	NIST	SRM 1941b

Table 56. (cont.)

Lab Code	Calibrants source	Calibrants used	Surrogates source	Surrogates used	CRMs source	Matrix CRMs Used
11	ULTRASCIENTIFIC	PAH MIX (22 analytes)	ULTRASCIENTIFIC	8270 Base/Neutral Surrogate Standard Mixture Semivolatiles internal std mix EPA 8270 (6 analytes)	NIST IAEA	1944 IAEA-383
12	ULTRASCIENTIFIC	PAH Mixture ITPM-023 Naphthalene Acenaphthylene Acenaphthene Fluorene Phenanthrene Anthracene Fluoranthene Pyrene Benz[*a*]anthracene Chrysene Benzo[*b*]fluoranthene Benzo[*k*]fluoranthene Benzo[*e*]pyrene Benzo[*a*]pyrene Perylene Indeno[*1,2,3-c,d*]pyrene Benzo[*g,h,y*]perylene Dibenz[*a,h*]anthracene Dibenz[*a,l*]pyrene Dibenz[*a,e*]pyrene Dibenz[*a,i*]pyrene Dibenz[*a,h*]pyrene Benzolfluoranthene	ULTRASCIENTIFIC	7 Deuterated PAH mix ISM-750 d10- Acenaphthene d10- Phenanthrene d10-Fluorantene d12-Benzo[*a*]antracene d12-Dibenzo[*a,*h]antracene GC internal std: 3 Deuterated PAH mix ISM-740A	IAEA	IAEA-383

Table 56. (cont.)

Lab Code	Calibrants source	Calibrants used	Surrogates source	Surrogates used	CRMs source	Matrix CRMs Used
13	ULTRASCIENTIFIC	Naphthalene 1-Methylnaphthalene 2-Methylnaphthalene 2.6-Dimethylnaphthalene 2.3.5-Trimethylnaphthalene Biphenyl Acenaphthene Acenaphthylene Fluorene 1-Metylfluorene Dibenzothiophene Anthracene Phenanthrene 1-Methylphenanthrene 2-Methylphenanthrene Fluoranthene Pyrene 1-Methylpyrene Benz[a]anthracene Chrysene 1-Metylchrysene Benzo[*b*]fluoranthene Benzo[*k*]fluoranthene Benzo[*a*]Fluoranthene Benzo[*a*]pyrene Benzo[*e*]pyrene Benzo[*g,h,i*]perylene Dibenz[*a,h*]anthracene Indeno[*1,2,3-c,d*]pyrene Perylene	ULTRASCIENTIFIC	d8-Naphthalene d10- Acenaphthene d8-Acenaphtylene d10- Phenanthrene d12-Chrysene d12-Perylene	IAEA	IAEA-459

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

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LIST OF PARTICIPATING LABORATORIES

FRANCE

Budzinski, H.	Molecular Science Institute (ISM) CNRS UMR 5255,Bat A12, LTPC Group 351 cours de la Liberation 33405 Talence Cedex

GERMANY

Brockmeyer, B.	Federal Maritime and Hydrographic Agency Bernhard-Nocht Strasse 78 20359 Hamburg

ITALY

Sesta, G.	ISPRA – Istituto Superiore per la Ricerca e la Protezione Ambientale Via di Castel Romano 100 00128 Roma
Anedda, R.	ARPA Sardegna Laboratorio dipartimentale di Cagliari Viale Ciusa, 6 9131 Cagliari

GREECE

Hatzianestis, I.	Hellenic Center for Marine Research 46,7 km Athens-Sounion Ave 19016 Anavyssos

KOREA, REPUBLIC OF

Yim, U. H.	South Sea Institute, KIOST Oil & POPs research group 391 Jangmok-ri, Jangmok-myom J-SHI 656-830, Gyeongsangnam-Do

MONACO

Tolosa, I.	International Atomic Energy Agency IAEA Environment Laboratories (NAEL-MESL) 4 Quai Antoine 1er 98000 Monaco

MONTENEGRO

Sukovic, D.
PI Center for ecotoxicological Research
Bulevar Sarla de Gola 2
81000 Podgorica

NORWAY

Klungsoyr, J.
Institute of Marine Research
(Havforskninginstituttet)
Marine Environmental Center
Nordnesgaten 50, 5005 Bergen
P.O. Box 1870 Nordnes
N-5817 Bergen

RUSSIAN FEDERATION

Shelepchikov, A.
A.N. Severtsov Inst. Ecological & Evolution
Russian Academy of Sciences
Leninsky Prospekt 33
119071 Moscow

TURKEY

Atabay, H.
TÜBİTAK-MRC Environment and Cleaner Production Inst.
Marine and Inland Waters Unit
Environment and Clean Production Institute
PO Box 21
41470 Gebze-Kocaeli

CONTRIBUTORS TO DRAFTING AND REVIEW

The following persons, all from the IAEA contributed to the draft and review of this publication:

I. Tolosa	International Atomic Energy Agency
R. Cassi	International Atomic Energy Agency
S. Azemard	International Atomic Energy Agency
M. Horsky	International Atomic Energy Agency
S. Sander	International Atomic Energy Agency
A. Mauring	International Atomic Energy Agency

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